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# Sustainable Materials and Technologies



## Effectiveness of ceramic tile polishing residues as supplementary cementitious materials for cement mortars

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### ARTICLE INFO

#### Article history:

Received 8 February 2015

Received in revised form 23 April 2015

Accepted 8 May 2015

Available online 19 May 2015

#### Keywords:

Residue

Supplementary cementitious materials

Autogenous shrinkage

### ABSTRACT

The sludge coming from the polishing process of ceramic tiles, particularly 'porcellanato' and 'monoporosa', results in a large amount of waste that requires disposal in controlled landfills. Consequently, the financial and environmental costs of landfilling are very high. However, the 'porcellanato' and 'monoporosa' polishing residues could be used as supplementary cementitious material (SCM) instead of being disposed or landfilled. Therefore, in this study, the synergistic effect of 'porcellanato' and 'monoporosa' polishing residues (MixPR) as supplementary cementitious materials was reported. The physical and chemical characteristics of MixPR were determined by laser diffraction (particle size), X ray fluorescence (chemical composition) and X ray diffraction (mineral composition). The variability of the characteristics of the studied MixPR was evaluated over a period of three months at the source. Mortar compositions were studied replacing the cement content by MixPR (0, 10, 20, 25, 30 and 40% mass fractions). The mortars were characterized by their consistency index (flow-table), compressive strength, pozzolanic activity index, thermal behavior (calorimetry) and autogenous shrinkage. As a result, the mortar compositions using MixPR maintain their plasticity and show a high rate of pozzolanic activity index, reaching 111%. The compressive strength at 120 days of curing was 41.5 MPa when using 25% MixPR, as compared to 40.0 MPa when no MixPR was used (0% addition). At early ages (28 days) the use of 25% MixPR reduces the compressive strength by 10–15% due to the slow nature of the pozzolanic activity caused by the residue. The results show an improvement of the efficiency index (given in  $\text{kg m}^{-3} \text{MPa}^{-1}$ ) when using MixPR due to the reduction on cement consumption by 30%, reducing therefore the  $\text{CO}_2$  emissions.

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### 1. Introduction

Ceramic polishing residues (PR) can be used for the manufacturing of several products, including ceramic tiles [1–3]. However, studies on the reuse of ceramic polishing residues in cement-based materials are scarce [4–6]. The polishing residue, called PPR if coming from 'porcellanato' tiles or MPR if coming from 'monoporosa' tiles, is a common waste from the ceramic industry, being, usually, landfilled. In the polishing process almost 1 mm of the tile surface is removed by the action of SiC and/or diamond tools that are fixed on water-cooled machines. As a result, a water suspension formed by a mixture of the abraded tool with the abraded ceramic surface is formed, that is called polishing residue. This sludge is processed in effluent treatment plants. There are large amounts of this sludge with relatively similar characteristics piled up in ceramic tile industries [1–3].

According to the NBR 10,004/2004 Brazilian Standard, both sludge ('monoporosa' and 'porcellanato' polishing residues) are classified as 'non-inert Class IIA residue' due to their pH after solubilization tests (pH = 13.8 for the mix). Therefore, the sludge must be adequately disposed on controlled landfills or adequately used as filler for cementitious materials [4–6].

Polishing residues, along with their filler effect [6–8], can maximize the hydration process in Portland cement due to the high amount of amorphous silica and alumina, that promotes the pozzolanic reaction. During hydration, the residue can act as nucleation centers owing to its small particle size after polishing.

Tests performed by Rambaldi et al. on cement mortars using 10% and 20% polishing residue (mass fractions) showed that the compressive strength at 56 days age increased by 50% [1]. At the same study, thermogravimetric (TG) results showed that portlandite was consumed by silica, which is part of the waste composition, in order to form C-S-H, presenting, therefore, pozzolanic action. The pozzolanic effect presented by polishing residues enables its use as supplementary cementitious materials (SCM) because the polishing residue (PR) improves cement performance due to the reduction in

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cement consumption, reducing, therefore, the environmental impacts caused by carbon dioxide emissions caused by Portland cement production [1,4].

Another important issue when using Portland cement for the production of more efficient materials, such as high-performance concrete, is the phenomenon of retraction, caused by the autogenous shrinkage. Retraction (or shrinkage) can lead to cracking of hydrated cement matrix and the early deterioration of concrete structures. The effect of PR addition on the autogenous shrinkage of cement pastes can be caused by four phenomena [9]: (i) cement dilution by PR, with less cement generating less shrinkage; (ii) heterogeneous nucleation of hydrates on the surface of PR particles, accelerating cement hydration and, consequently, increasing shrinkage; (iii) pozzolanic reaction of PR with CH produced by cement and; (iv) increase of capillary tension, due to the refinement of pore size distribution, leading to an increase in autogenous shrinkage.

Andreola et al. [4] showed in their study on polishing residues as supplementary cementitious material that the use of PR has resulted in an increase in compressive strength and a reduction in porosity. For them, there was chemical activity between the residue and portlandite (calcium hydroxide). The chemical composition of the sludge studied by Andreola et al. [4] is similar to the sludge studied in this work. In their study, the compressive strengths were 63.2, 56.8 and 53.3 MPa for the compositions using no residue (0% PPR), 25% 'porcellanato' residue and 25% glazed residue, respectively. For 'porcellanato' residue and glazed residues, respectively, the pozzolanic activity indices were 89.9 and 84.3 at 28 days, and 101.4 and 88.0 at 90 days. The replacement of 25% cement by PR outperformed the pure cement, showing the synergistic effect of mixing cement with PR, and leading to the improvement of permeability and durability of the products. The water/cement ratio was 0.50.

The results of Pelisser et al. [6] corroborate this behavior. The use of PPR as addition to cement resulted in an increase in compressive strength, from 27.2 MPa for 0% PPR to 41.2 MPa for 20% PPR addition after 56 days. Considering that, for the reference mortar, 17 kg of cement are needed to obtain 1 MPa of strength with a cement consumption of 456 kg/m<sup>3</sup>, the increase in compressive strength would save, in theory, about 238 kg of cement per cubic meter of mortar. In this case, the performance index of cement consumption would decrease from 16.7 kg/MPa for 0% PPR addition to 10.4 kg/MPa for 20% PPR addition. For both examples, the cement yield is higher than 40%.

Wattanasiriwech et al. [5] studied the use of polishing residues for the production of paving blocks. The results showed that the use of 20% cement in the composition could result in a compressive resistance of 35 MPa after 28 days age.

'Monoporosa' is another common polishing residue, which has not been extensively studied for application as SCM in Portland cement products. For some ceramic tile industries, the amount of 'monoporosa' polishing residue is higher than that of 'porcellanato' polishing residue. As our previous study [6] showed the effect of 'porcellanato' sludge as supplementary cementitious materials (SCM), studying the effect of 'monoporosa' as SCM is important in order to compare the feasibility of using both residues in cement products. Furthermore, to date there are no studies on the variability of the properties of both residues over time for use as SCM.

Therefore, the aim of this work was to study the effect and variability over time of production of PPR ('porcellanato' polishing residue) and MPR ('monoporosa' polishing residue) as supplementary cementitious materials (SCM). In order to evaluate the variability in their physical-chemical characteristics in batch production, both residues were collected in a period of three months from a company specialized in polishing ceramic tiles located at Criciúma city, Santa Catarina state, Brazil. The residues were characterized by XRF, XRD and PSD and a mixture of them was added to mortar compositions to determine their effectiveness as SCM.

## 2. Materials and methods

CP V ARI Portland cement (equivalent to CEM I 52.5 cement according to EN 197-1 [10] standard) and Brazilian standard sand (NBR 7215 standard, mixing the four particle sizes [11]) were used for the composition of all cement pastes and mortars developed in this study.

The 'porcellanato' and 'monoporosa' polishing residues were collected during eight weeks in order to evaluate the variability of the residue. The samples were collected from a company specialized in polishing and lapping ceramic tiles (wall and floor tiles), located at Criciúma, southern of Santa Catarina state, Brazil. The water used in the processing (polishing and lapping) lines, cleaning and cooling equipment is drained by gravity into channels to the wastewater treatment system. Any residue coming from 'porcellanato' or 'monoporosa' tiles share the same circuit pipes to the treatment system, preventing the separation of both before the filter press system.

Also, there is a great daily or even weekly variation in the production of both residues; sometimes the production lines produce more 'monoporosa' residue, sometimes more 'porcellanato' tile residue. Due to the change in volume coming from both residues, a schedule of collections (eight weeks) was drawn up on the same day of the week, in order to well represent the variation in output and to determine if there were significant differences on both residues. The samples were identified as 'porcellanato' polishing residue (PPR) and 'monoporosa' polishing residue (MPR) due to the company having processed more 'porcellanato' tiles or 'monoporosa' tiles.

The residues were characterized by X-ray diffraction (XRD, CuK $\alpha$ ,  $\lambda = 1.5418 \text{ \AA}$ , 10° to 80° (2 $\theta$ ) in 2°/min reading time), laser diffraction and X-ray fluorescence (by WDS) techniques in order to determine their mineralogical, particle size distribution and chemical characteristics. No significant differences could be observed between the PPR and MPR samples, as shown in Table 1. Therefore, both residues were mixed together to study their effect on cementitious materials, and the residue was called only PR (polishing residue) and assigned as MixPR for analysis. After mixing, the average particle size was determined to be 13.7  $\mu\text{m}$ .

The mortars were produced in 1:3:0.6 ratio (cement: sand: water/binder ratio) with cement replacement by MixPR at 0, 10, 20, 25, 30 and 40 wt.% concentrations. The replacement was performed in relation to the cement mass, because the bulk density of both materials is close. The plasticity (determined by the flow-table method, according to EN 1015 [12] standard) was kept constant at  $27 \pm 2 \text{ cm}$ .

To characterize the cement/residue compositions, compressive strength, pozzolanic activity, differential thermal analysis and isothermal calorimetry tests were performed:

i) Compressive strength tests according to ASTM 1231 standard [13] at 2, 7, 28, 100 and 120 days of age. Additionally, strength tests (at 28 days) using limestone filler ( $D_{50} = 29.47 \mu\text{m}$ ) in place of the MixPR were performed;

ii) Pozzolanic activity index (PAI) according to ASTM C 311 standard [14], considering PAI = 75%. The pozzolanic activity index was determined using Eq. (1) [14]:

$$BI = \frac{\text{cement cons.}}{\text{Strength}} \quad 1$$

where: BI is the efficiency index or binder index; cement cons. is the cement consumption (kg/m<sup>3</sup>); and strength is the performance indicator, in this case, the strength of the samples (MPa).

iii) Thermal analysis in samples containing 0, 20, 25 and 40 wt.% MixPR at 28 days age (Q600 SDT, TA Instruments, 10 °C/min heating rate, 20–800 °C, 100 ml/min N<sub>2</sub> flow, platinum crucible, 15 mg sample). The amount of calcium hydroxide was calculated considering Eq. (2) (Silva et al. [15])

$$CH = pmCH \times \frac{mmCa(CH)_2}{mmH_2O} \quad 2$$

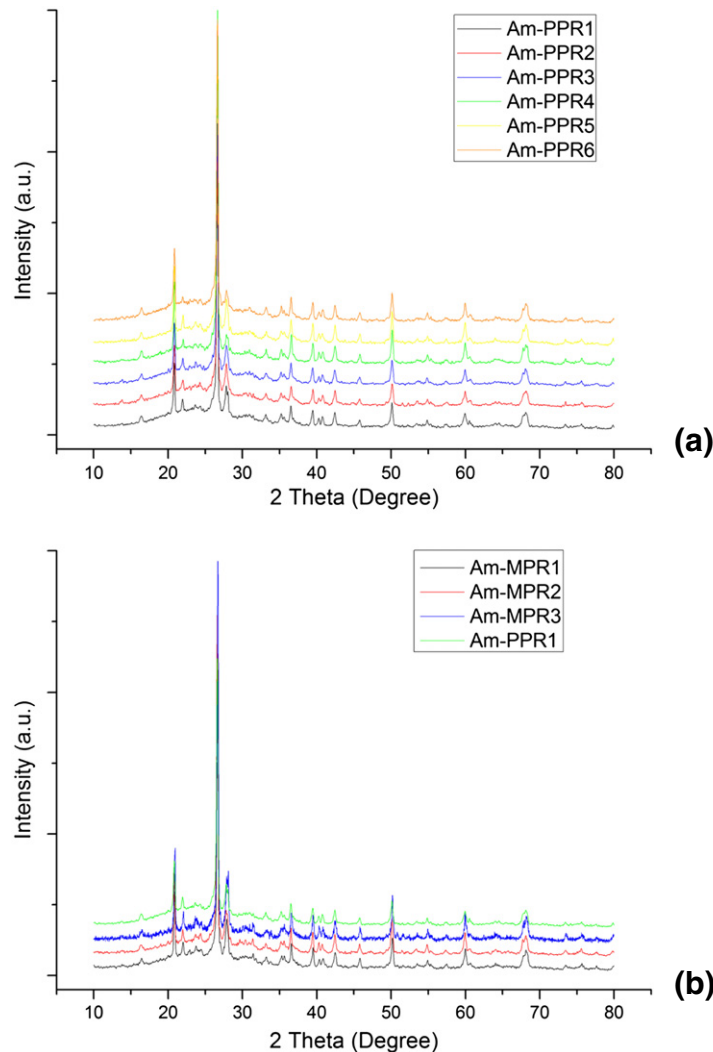
**Table 1**  
Chemical analyzes (XRF) and particle size distribution of the polishing residue and cement.

Oxides	Materials									
	CEM	PPR	MPR	MPR	PPR	PPR	PPR	PPR	MPR	PPR
Al <sub>2</sub> O <sub>3</sub>	5.2	21	20.9	20.9	20.9	20.7	19.4	20	20.1	20.8
CaO	58.5	1.8	3.1	3.7	1.6	2.1	1.7	2.6	4.6	1.2
Fe <sub>2</sub> O <sub>3</sub>	2.5	1.7	1.7	1.67	1.7	1.7	1.9	1.9	1.9	1.5
K <sub>2</sub> O	0.53	2.5	2.3	2.3	2.3	2.5	2.3	2.3	2	2.4
MgO	1.6	1.2	1.1	1.1	1.2	1.2	1.9	1.5	1.3	1.1
Na <sub>2</sub> O	0.11	3.3	2.6	2.3	3.6	3.3	2.1	2.4	1.5	3.2
P <sub>2</sub> O <sub>5</sub>	0.06	0.11	0.1	0.1	0.11	0.08	0.06	0.07	0.08	0.08
SiO <sub>2</sub>	20.3	66.4	66	65.7	66.2	66.1	68.9	67.1	66.2	68.1
TiO <sub>2</sub>	0.25	0.77	0.76	0.7	0.79	0.74	0.57	0.65	0.68	0.59
BaO	0.23	0.12	0.21	0.2	0.21	0.21	0.24	0.3	0.25	0.07
ZrO <sub>2</sub>	< 0.1	0.46	0.49	0.61	0.5	0.45	0.37	0.48	0.57	0.43
LOI	3.5	0.5	0.5	0.57	0.49	0.62	0.5	0.59	0.52	0.47
Particle size, D <sub>50</sub> (μm)	10.8	12.2	10.4	6.7	11.6	10	5.7	7.5	10.6	7.4

where: pmCH is the loss of weight of Ca(OH)<sub>2</sub> (wt.%); mmCa(CH)<sub>2</sub> is the molar mass of calcium hydroxide (= 74); mmH<sub>2</sub>O is the molar mass of water (= 18);

iv) Isothermal calorimetry analysis, performed to evaluate the replacement of cement by residue in amounts of 0, 10, 20 and 25 wt.% and considering additions of 10, 20 and 25 wt.% of residue over cement. Samples of 11 ± 0.1 g were used in tests conducted at 22 °C (TAM Air, TA Instruments);

v) Autogenous shrinkage of pastes: According to NBR 12,650 standard [16], 2.5 × 2.5 × 30 cm<sup>3</sup> sample molds and 25 cm reading length. The deformations were measured in the center of the span using two LVDTs, positioned on opposite sides of the center span (2 mm ± 2 μm readability). The test was performed in a climatic chamber with a constant temperature of 22 ± 2 °C. The molds were sealed on the surface with resin and a glass plate. The cement paste with 0, 20 and 40 wt.% residue addition was placed 5 min after mixing. The test was performed



**Fig. 1.** (a) Diffractograms for the 'porcellanato' polishing residue (PPR) samples and (b) for the 'monoporosa' polishing residue (MPR) samples.

**Table 2**  
Chemical analyzes (XRF) and particle size distribution of the polishing residue mixture (MixPR).

Oxides	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	Na <sub>2</sub> O	SiO <sub>2</sub>	TiO <sub>2</sub>	ZrO <sub>2</sub>	LOI	Particle size, D <sub>50</sub> (μm)
MixPR	21.0	2.6	1.7	2.3	1.2	2.7	66.4	0.7	0.4	0.4	13.7

between 23 and 30 days with 50 s reading intervals, but the age of 23 days was used for comparison of the results. The test was performed just to separately assess the effect of waste and cement.

### 3. Results and discussion

#### 3.1. PPR and MPR characterization

Chemical (by XRF) analysis for all samples of PPR and MPR showed similar results, Table 1. Also, the mean diameters (by laser diffraction) for all samples are quite similar particle (Table 1). From the chemical analysis, the polishing residues ('porcellanato' and 'monoporosa') are mainly formed by silica (~66 wt.%) and alumina (~21 wt.%) with smaller amount of alkaline (~5 wt.%) and earth-alkaline (~4 wt.%) oxides, typical composition for vitrified ceramic tiles. The average particle size for all samples, 9 μm, is typical for residue coming from a polishing operation of ceramic tiles.

Fig. 1a shows the diffractograms for the 'porcellanato' polishing residue (PPR) samples and Fig. 1b for the 'monoporosa' polishing residue (MPR) samples. The analysis for all samples shows that there are virtually no differences between the 2θ values of the main peaks for all samples. PPR and MPR samples present the same crystalline phase, quartz (PDF-file 46-1045). However, besides the presence of quartz, the residue can be considered a reactive material due to the amorphous halo between 18° and 36° (2 theta).

Considering the results, the samples of PPR and MPR were mixed together, resulting in a sample called MixPR that was used in the preparation of mortar and paste compositions. Its chemical analysis and particle size is shown in Table 2.

After mixing, the amount of SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> oxides for the MixPR composition was 89.1 wt.% and its particles showed a mean diameter of 13.7 μm and, therefore, MixPR can be classified as a pozzolanic material in accordance to ASTM C618 standard [17]. That standard recommends that the sum of SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> should be at least 70 wt.% for a particle size with an equivalent diameter less than 45 μm.

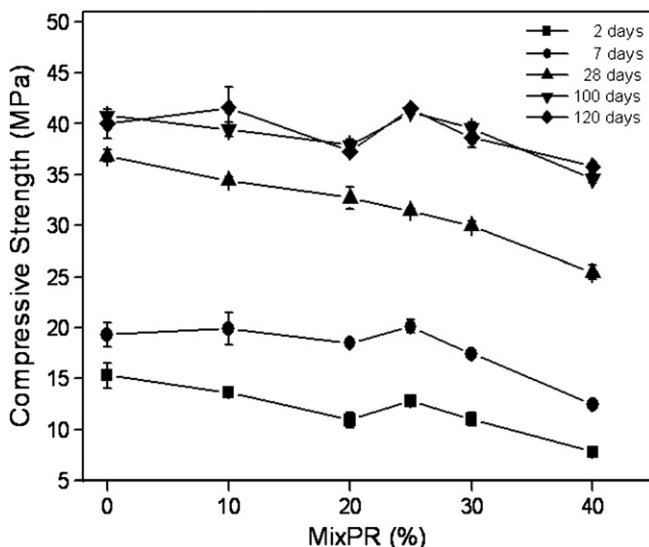


Fig. 2. Compressive strength for mortars with MixPR addition.

#### 3.2. Mechanical properties of mortars

During mixing, the mortar plasticity was quite constant. The flow-table test results were 29.4, 28.4, 30.6, 30.3, 30.9 and 30.5 cm for mortars with 0, 10, 20, 25, 30 and 40 wt.% MixPR contents, respectively. From 2 to 28 days of age the compressive strength remained constant until the addition of 25 wt.% MixPR in the mortar composition, Fig. 2. For higher ages, the increasing in resistance is higher for compositions with more amount of MixPR. The composition with 30 wt.% MixPR achieved equivalent performance to the reference sample (without MixPR addition). As a comparison, the use of 30 wt.% limestone as filler in the mortar compositions resulted in a reduction of 25% on the compressive strength of the samples regarding the mortar composition with 30 wt.% MixPR (Fig. 3). The compressive strength reduction was 40% for the mortar composition with 40 wt.% of limestone. The strength results show the effect of the polishing residue (MixPR) as a pozzolanic material.

Regarding the efficiency index for cement consumption ( $\text{kg m}^{-3} \text{MPa}^{-1}$ ), the reference composition (at 120 days of age) shows an index of  $11.41 \text{ kg m}^{-3} \text{MPa}^{-1}$ . The mortar with the addition of 40 wt.% MixPR shows an index of  $7.65 \text{ kg m}^{-3} \text{MPa}^{-1}$ , a saving in cement consumption of 33%. This result fits in benchmarks of cement use efficiency cited by Daminele et al. [18].

The effect is attributed to the pozzolanic activity showed by MixPR. Fig. 4 shows that the pozzolanic activity rates for mortars with 25 wt.% MixPR are 85, 101 and 104% at 28, 100 and 120 days of age, respectively.

As the concentration of MixPR is increased in the mortar samples, the concentration of calcium hydroxide is lowered. The calcium hydroxide amount is reduced from 19 wt.% (for 0 wt.% MixPR) to 9 wt.% (for 40 wt.% MixPR addition), as can be observed by the thermal analysis results, Fig. 5. The reduction in the calcium hydroxide content is improved at the age of 120 days. Obviously, the reduction in cement consumption in mortars using MixPR results in less amount of calcium hydroxide, but the reduction observed, 47 wt.%, was greater than the reduction in Ca(OH)<sub>2</sub> that should be expected for a smaller cement consumption.

Another possible contribution to the increasing in resistance was the filler effect and the heterogeneous nucleation caused by the fine particles of MixPR during cement hydration, as can be concluded from the calorimetric analysis, Fig. 6. The normalized heat (W/g) increases during the acceleration period (up to 0.0045 W/g) for mortar compositions that

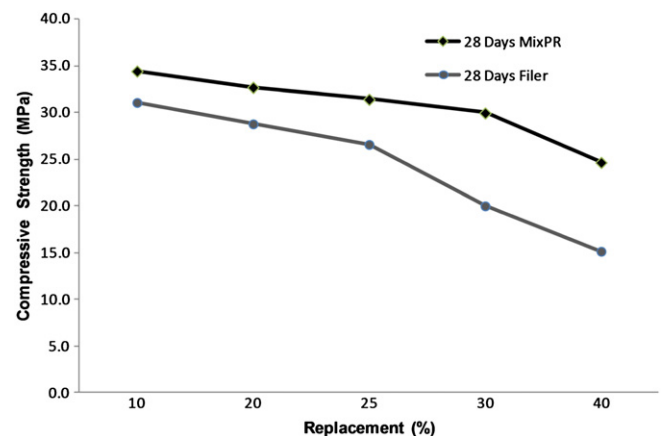


Fig. 3. Compressive strength for mortars + MixPR and mortars + filler.

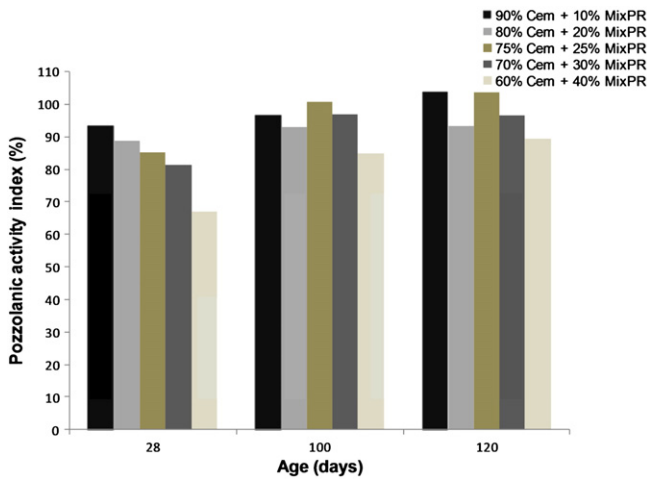


Fig. 4. Pozzolanic activity index of the compositions.

exchanged cement by MixPR residue and even for those that used MixPR as an addition over cement.

### 3.3. Autogenous shrinkage of pastes

The autogenous shrinkage was also significantly reduced with the use of MixPR, Fig. 7. The reference samples show an overall shrinkage of 603  $\mu\text{m}/\text{m}$  (average of three samples). The samples with 25 wt.% of MixPR show an overall shrinkage of 382  $\mu\text{m}/\text{m}$ , a reduction of 37% in shrinkage. The samples with 40 wt.% of MixPR show an overall shrinkage of 108  $\mu\text{m}/\text{m}$ , a reduction of 82% in shrinkage. Although the test was performed between 23 and 30 days with 50 s reading intervals, the age of 23 days was used for comparison of the results for all samples.

Fig. 7 shows the three stages of shrinkage, particularly for reference samples and samples with 25 wt.% MixPR. Initially there is a small shrinkage in the first day, followed by an expansion, and finally an increasing in shrinkage. According Bathar [19] the behavior can be explained as follows: i) first shrinkage or early retraction occurs after the onset of cement setting (2 to 3 h after mixing) and is associated with the onset of hydration reactions and with the Le Chatelier contraction (when the volume of hydrates formed is less than the volume of product plus the non-hydrated water); ii) period of expansion, between 4 and 10 h after mixing, and may extend up to 10–20 h. This expansion occurs due to the formation and precipitation of large crystals of ettringite during setting; and iii) second shrinkage or ‘self-drying’ shrinkage, that begins after setting and when all the cement hydration is complete. It is

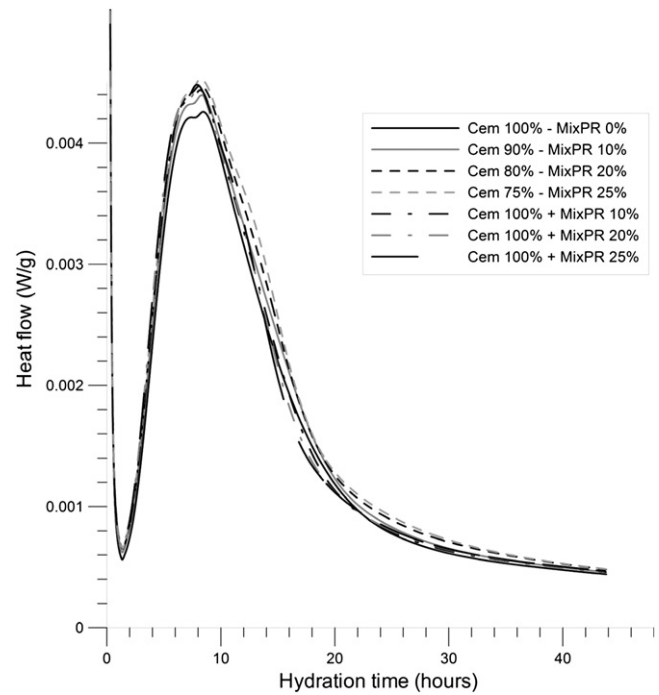


Fig. 6. Kinetic curves of cement hydration for mortars with MixPR (substitution and addition).

responsible for much of the autogenous shrinkage. The expansion period can also be related with the thermal expansion of the cement paste, since the reactions of cement hydration are exothermic [20].

Considering the above, the reduction of the autogenous shrinkage is explained by two situations: i) the pozzolanic action of MixPR, which promotes slower reactions; and the phenomenon known as ‘inner healing’, in which pozzolanic materials adsorb significant amounts of water in their surfaces, which in sequenced is released to the environment during cement hydration, and therefore reducing the capillary pressure between the grains; and ii) the effect of cement dilution in the mixtures (mortars), beginning from 25 wt.% substitution of cement by MixPR. Therefore, as the compositions show the same plasticity and strength, the reduction in shrinkage can be related to the use of MixPR in the mortar compositions.

Finally, an analysis of variance, ANOVA, was performed in order to determine, statistically, if the polishing residue mix (MixPR) could influence the autogenous shrinkage of the samples. Table 3 shows the

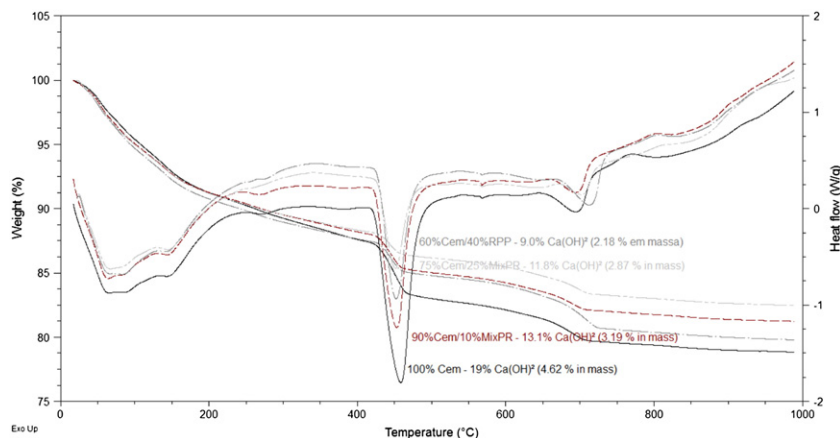


Fig. 5. Thermal analysis.

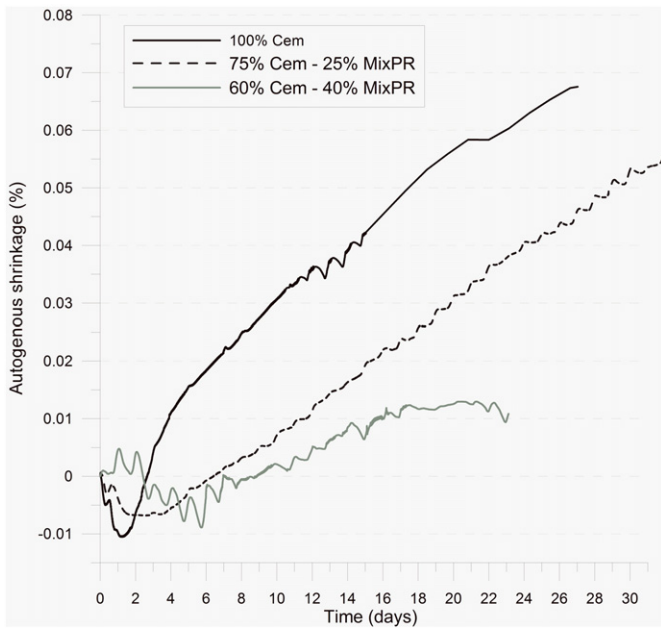


Fig. 7. Autogenous shrinkage.

significant effect of MixPR in reducing the autogenous shrinkage (ANOVA, 95% reliability). In ANOVA, Table 3, the amount of the polishing residue mix (MixPR) was the independent variable (the factor under study) and the autogenous shrinkage was the dependent variable (the response when the factor was changed). For a 95% confidence interval, the result shows that the use of MixPR in the mortar compositions significantly affects the autogenous shrinkage, with a reliability of the results ( $p$ -value) of 99.99% regarding the error of the experiment.

The results obtained in this study show the outstanding performance of MixPR as a supplementary cementitious material. The use of this residue contributes to the sustainability of the construction industry because its use promotes the recycling of solid waste and the use of more efficient and durable materials, resulting in savings with expenditures for maintenance and rehabilitation of structures.

#### 4. Conclusions

The study was carried out with several samplings coming from two kinds of polishing residues, 'porcellanato' tiles and 'monoporosa' tiles. The surfaces of both products are quite similar: the 'monoposa' surface presents a vitreous layer produced by glazing and the 'porcellanato' surface (and body) is partially vitrified due to its composition after firing. Both residues present silica and alumina as main components, with smaller amount of alkaline and earth-alkaline oxides, with similar composition. Also, their particle sizes are quite similar. The XRD analysis showed that both residues are mainly composed by quartz and an amorphous phase. Therefore, they can be used as supplementary cementitious materials.

Table 3

ANOVA for the effect of MixPR on the autogenous shrinkage.

Variable	$SS_M$	dF	$SS_e$	F	p
MixPR (mass %)	0.003684	2	0.001842	74.3147	0.000058
Error	0.000149	6	0.000025	–	–

$SS_M$ : Sum of squares of the main effect; dF: Degrees of freedom;  $SS_e$ : Sum of squares of the error.

Regarding the efficiency ratio of cement consumption ( $\text{kg m}^{-3} \text{MPa}^{-1}$ ), the results showed that the use of MixPR in mortar compositions has improved the efficiency ratio, reducing the cement consumption by 30%. The effect was caused by the pozzolanic and filler effects of MixPR. Also, the use of MixPR, the mixture of 'porcellanato' and 'monoporosa' polishing residues in mortar compositions, besides increasing the onset time of shrinkage, has reduced the total autogenous shrinkage, that nowadays is a serious problem for durability of structural concrete. A higher shrinkage causes premature cracking during concrete use.

This effect contributed to the study and to the applications of supplementary cementitious materials, while optimizing the use of Portland cement and reducing the environmental impact of carbon dioxide emissions originating from its production.

#### Acknowledgments

The authors gratefully acknowledge Poligress Porcelanato for providing the polishing residue for this research.

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