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# DSC and TG Analysis of a Blended Binder Based on Waste Ceramic Powder and Portland Cement

Zbyšek Pavlík[1] · Anton Trník[1,2] · Tereza Kulovaná[1] ·
Lenka Scheinherrová[1] · Viviana Rahhal[3] ·
Edgardo Irassar[3] · Robert Černý[1]

**Abstract** Cement industry belongs to the business sectors characteristic by high energy consumption and high $CO_2$ generation. Therefore, any replacement of cement in concrete by waste materials can lead to immediate environmental benefits. In this paper, a possible use of waste ceramic powder in blended binders is studied. At first, the chemical composition of Portland cement and ceramic powder is analyzed using the X-ray fluorescence method. Then, thermal and mechanical characterization of hydrated blended binders containing up to 24 % ceramic is carried out within the time period of 2 days to 28 days. The differential scanning calorimetry and thermogravimetry measurements are performed in the temperature range of 25 °C to 1000 °C in an argon atmosphere. The measurement of compressive strength is done according to the European standards for cement mortars. The thermal analysis results in the identification of temperature and quantification of enthalpy and mass changes related to the liberation of physically bound water, calcium-silicate-hydrates dehydration and portlandite, vaterite and calcite decomposition. The portlandite content is found to decrease with time for all blends which provides the evidence of the pozzolanic activity of ceramic powder even within the limited monitoring time of 28 days. Taking into account the favorable results obtained in the measurement of compressive strength,

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✉ Zbyšek Pavlík
pavlikz@fsv.cvut.cz

[1] Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague, Thákurova 7, 16629 Prague, Czech Republic

[2] Department of Physics, Faculty of Natural Sciences, Constantine the Philosopher University in Nitra, A. Hlinku 1, 949 74 Nitra, Slovakia

[3] Departamento de Ingeniería Civil, Facultad de Ingeniería, Universidad Nacional del Centro de la Provincia de Buenos Aires, Av. del Valle 5737, B7400JWI Olavarría, Argentina

it can be concluded that the applied waste ceramic powder can be successfully used as a supplementary cementing material to Portland cement in an amount of up to 24 mass%.

# 1 Introduction

Concrete is the most often used construction material over the world which is largely due to the abundance of raw materials for cement manufacture, low relative cost, and the versatility in forming various structural shapes [1]. Concrete has excellent mechanical and durability properties but its production is accompanied with some harmful secondary effects to the human society and the environment. The first problem consists in the vast amount of natural resources needed to produce high concrete amounts each year. Cement manufacturing implies the extraction of 1.7 tons of rock to produce 1 ton of clinker. Then, the production of one ton of cement for concrete industry generates 0.55 tons of $CO_2$ and requires additional 0.39 tons of $CO_2$ in fuel emissions, accounting for a total of 0.94 tons of $CO_2$ [2]. In addition, cement production has a high energy impact, which is approximately $850 \, \text{kcal·kg}^{-1}$ of clinker [3]. Therefore, the replacement of cement in concrete by any type of industrial by-product or waste represents a tremendous saving of energy and has important environmental benefits. Besides, it can also have a substantial effect on decreasing the price of concrete, since cement represents more than 45 % of concrete costs.

For concrete which is designed, produced and used in an environmentally friendly manner, the term "Green Concrete" is commonly used [4]. Many industrial by-products have already found their application in concrete production, among them coal combustion fly ash, ground granulated blast furnace slag, and silica fume as typical examples, but other alternatives are being sought continuously. Waste ceramics belong to very prospective options in that respect. In Europe, the amount of waste in the different production stages of the ceramic industry reaches some 3 % to 7 % of its global production. This represents millions of tons of calcined clays per year that are mostly landfilled [5]. Although the process of reutilization of ceramic wastes already began, the total amounts are still very low. The pozzolanic properties of calcined clays attracted the attention of various research groups working on the production of new hydraulic materials for masonry and concrete industry within the last decade [6]. A portion of ceramic products formed after burning the illite-group minerals can become active pozzolan if ground to a suitable fineness [7]. Therefore, waste ceramic materials have a great potential to become a cheaper but almost equivalent alternative to metakaolin as a supplementary binder in mortar and concrete.

Characterization of the hydration products of blended cements containing waste ceramics as pozzolanic additions can be done to advantage by differential scanning calorimetry and thermogravimetry. Heating during the experiment is performed under controlled conditions, making possible to observe the changes in composition of the studied material [8]. The hydration of cement starts immediately after its contact with

water and it is almost completed within about 28 days. The monitoring of hydration reactions is the key to a proper design of high-quality concrete. The information on $Ca(OH)_2$ (portlandite) formation is very important because this component affects significantly the properties of concrete. It was found out that the $Ca(OH)_2$ content decreases with the hydration time and water/cement ratio [9]. Thermogravimetry has been used to identify the various substances present in cement pastes in many research studies before [10–14]. It was also successfully used to study the hydration process of blended cements containing different pozzolanic materials, such as metakaolin [15–17], clays [18,19], bricks [20], or porcelain [21].

In this paper, a fine ceramic powder originating in brick production is studied as a possible partial Portland cement substitute. The course of the hydration process in blended binders containing ceramic waste in a range of 0 to 24 mass% of Portland cement is analyzed using differential scanning calorimetry (DSC) and thermogravimetry (TG). The DSC and TG measurements are done for 2, 7, and 28 days cured samples. In addition, the compressive strength is measured to link the changes in composition to the mechanical properties.

## 2 Samples and Experimental Procedures

The studied samples of cement pastes were prepared using Portland cement CEM I 42.5 R and ceramic powder. The chemical composition of both materials was measured by X-ray fluorescence (XRF); it is given in Table 1. The density of Portland cement and ceramic powder was $3.10\,g\cdot cm^{-3}$ and $2.72\,g\cdot cm^{-3}$, respectively. The ceramic powder was used as a partial replacement of cement in the amount of 8 mass%, 16 mass%, and 24 mass%. Thus, three types of samples—denoted here as C8, C16, and C24—with different cement substitution levels were prepared to study the effect of the ceramic addition on the hydration process. The reference samples without a cement replacement (denoted here as REF) were also prepared. The water/cement ratio was 0.5 in all cases.

To study the course of hydration and pozzolanic reaction in the cement pastes containing ceramic powder, a simultaneous thermal analysis (STA) was used. The STA was done using a Labsys Evo (Setaram) device that allows simultaneous DSC–TG measurements [22]. Ground samples were prepared for the experiment which were placed into an alumina crucible with a volume of $100\,mm^3$. The mass of the samples was around 50 mg. The STA was realized in the temperature range from $25\,°C$ to $1000\,°C$ with a heating rate of $5\,°C\cdot min^{-1}$ in an argon atmosphere (flow rate $40\,mL\cdot min^{-1}$). The compressive strength was tested according to the technical standard ČSN EN 196-

**Table 1** Chemical composition of Portland cement and ceramic waste (in mass%)

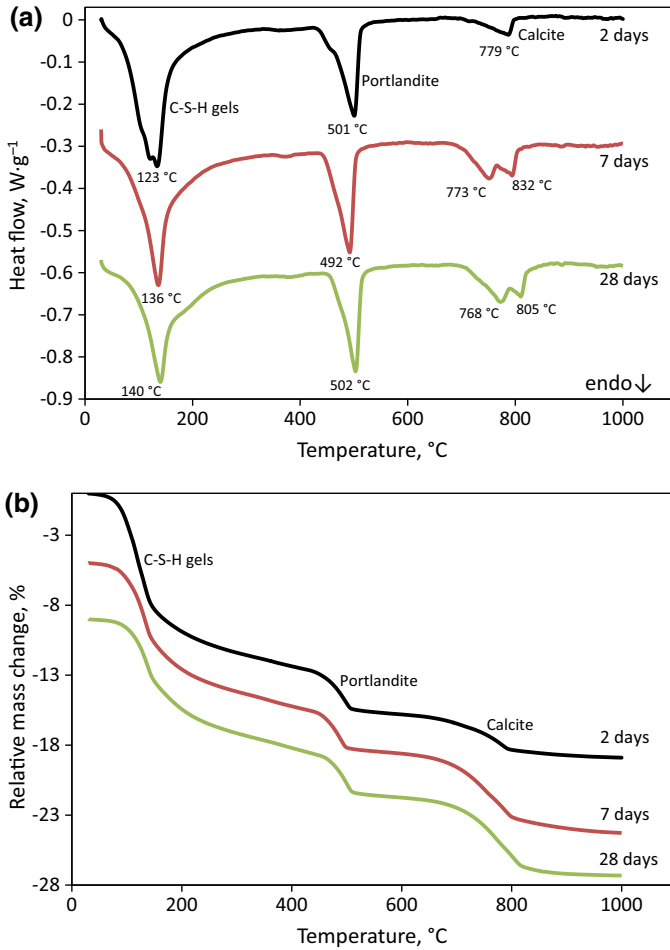|  | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $K_2O$ | $Na_2O$ | $TiO_2$ | $SO_3$ | LOI |
|---|---|---|---|---|---|---|---|---|---|---|
| Portland cement | 19.9 | 5.7 | 3.8 | 63.3 | 1.0 | 1.1 | 0.1 | 0.3 | 3.3 | 1.5 |
| Ceramic waste | 51.3 | 19.3 | 6.0 | 11.5 | 4.5 | 3.2 | 1.3 | 0.8 | 1.0 | 1.1 |

**Fig. 1** Heat flow (a) and relative mass change (b) of the reference cement paste

1 [23] (increasing loading force was at the rate of $(2400 \pm 200)$ N·s$^{-1}$ until a fracture occurred). The DSC, TG, and compressive strength measurements were done for 2, 7, and 28 days cured samples.

## 3 Experimental Results and Discussion

### 3.1 Heat Flow and Relative Mass Change Data

The heat flow (in W·g$^{-1}$) and relative mass change (in %) obtained for the reference cement paste samples REF at a given temperature and hydration time are shown in Fig. 1a, b, respectively. In Fig. 1a, three significant endothermic heat flow peaks can be observed. The first peak in the temperature interval from 50 °C to 250 °C corresponds

**Table 2** Relative mass change of the samples containing 0 mass% to 24 mass% of ceramic waste during C–S–H gels decomposition in dependence on hydration time (in %)

| Time (days) | REF | C8 | C16 | C24 |
|---|---|---|---|---|
| 2 | −12.00 | −9.71 | −11.28 | −9.98 |
| 7 | −8.78 | −7.85 | −7.72 | −7.66 |
| 28 | −8.37 | −7.75 | −7.23 | −6.57 |

**Table 3** Relative mass change of the samples containing 0 mass% to 24 mass% of ceramic waste during portlandite decomposition in dependence on hydration time (in %)

| Time (days) | REF | C8 | C16 | C24 |
|---|---|---|---|---|
| 2 | −3.04 | −2.60 | −2.46 | −2.34 |
| 7 | −2.94 | −2.62 | −2.31 | −2.00 |
| 28 | −2.91 | −2.46 | −2.15 | −1.85 |

to the release of physically bound water from pores, together with the dehydration reactions due to the loss of water from calcium silicate hydrates (C–S–H gel) [24–26]. The peak temperature of the C–S–H gel decomposition increases with hydration time from 123 °C (for 2 days) to 140 °C (for 28 days). The reactions in this temperature interval are also associated with a significant mass loss (see Fig. 1b for a detail). For the 2 days sample the mass loss is about 12.0 %. Then the mass loss decreases with the curing time of the sample, and reaches ∼8.4 % for 28 days sample (see Table 2).

The second peak in the temperature interval from 420 °C to 520 °C (see Fig. 1a) represents the decomposition of portlandite [25,27,28]. The peak temperature position is between 492 °C (for 7 days) and 502 °C (for 28 days). The portlandite decomposition is also accompanied by a mass loss (see Fig. 1b). This effect is visible for all studied samples but its magnitude is different. It slightly decreases from 3.0 % (2 days sample) to 2.9 % (28 days sample), see Table 3.

The peaks in the temperature interval 650 °C to 800 °C correspond to the decomposition of calcite ($CaCO_3$) [24,25] and carbonated C–S–H gels [29] (see Fig. 1a). The carbonation of C–S–H leads to the formation of vaterite, which seems to be an unstable type of calcium carbonate which decomposes typically between 500 °C and 700 °C, at a lower temperature than calcite [30]. The temperature range of the corresponding decompositions is difficult to determine because the peaks overlap. The peak temperature of the vaterite decomposition is visible for 7 and 28 days sample and it is located at temperature 773 °C and 768 °C, respectively. The peak temperature of the calcite decomposition is between 779 °C (for 2 days) and 832 °C (for 7 days). These reactions are accompanied by a significant mass loss which is ∼2.5 % for 2 days sample, then increases with the hydration time of the sample and reaches ∼5.1 % for 28 days sample (Table 4).

The influence of ceramic powder in the studied samples on the three significant endothermic reactions which were discussed before is presented in Figs. 2, 3 and 4. The first reaction is in the temperature interval from 50 °C to 250 °C and corresponds to the C–S–H gels decomposition. The peak temperature of the C–S–H gel decomposition increases with the hydration time for all studied samples with ceramic powder (C8, C16, C24) and reaches 136 °C for 28 days (Figs. 2a, 3a, 4a). Thus, the differences in

**Table 4** Relative mass change of the samples containing 0 mass% to 24 mass% of ceramic waste during calcite and vaterite decomposition in dependence on hydration time (in %)

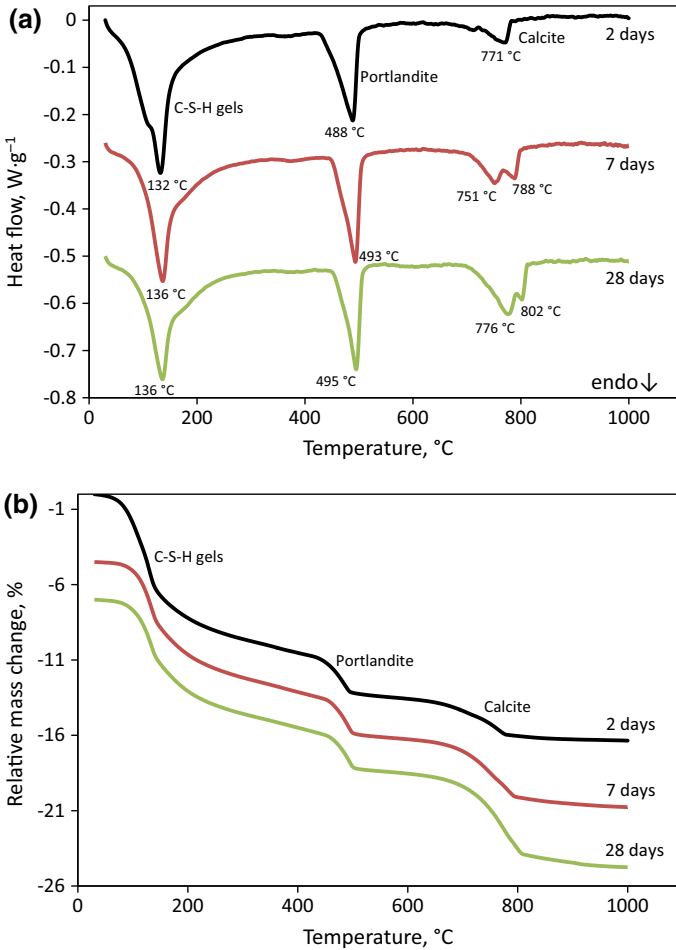| Time (days) | REF | C8 | C16 | C24 |
|---|---|---|---|---|
| 2 | −2.47 | −2.49 | −1.48 | −2.25 |
| 7 | −4.57 | −3.87 | −4.08 | −4.02 |
| 28 | −5.14 | −5.52 | −4.50 | −4.36 |



**Fig. 2** Heat flow (a) and relative mass change (b) of the cement paste containing 8 mass% of ceramic powder

the peak temperature between the reference sample and the samples containing the ceramic powder are negligible. The mass loss (Figs. 2b, 3b, 4b) decreases with the hydration time of the samples. This is valid for all studied samples. Also, the mass loss decreases with the amount of ceramic powder in the sample. For 28 days the mass loss is 8.4 % for the reference sample and 6.6 % for the sample C24 (Table 2).
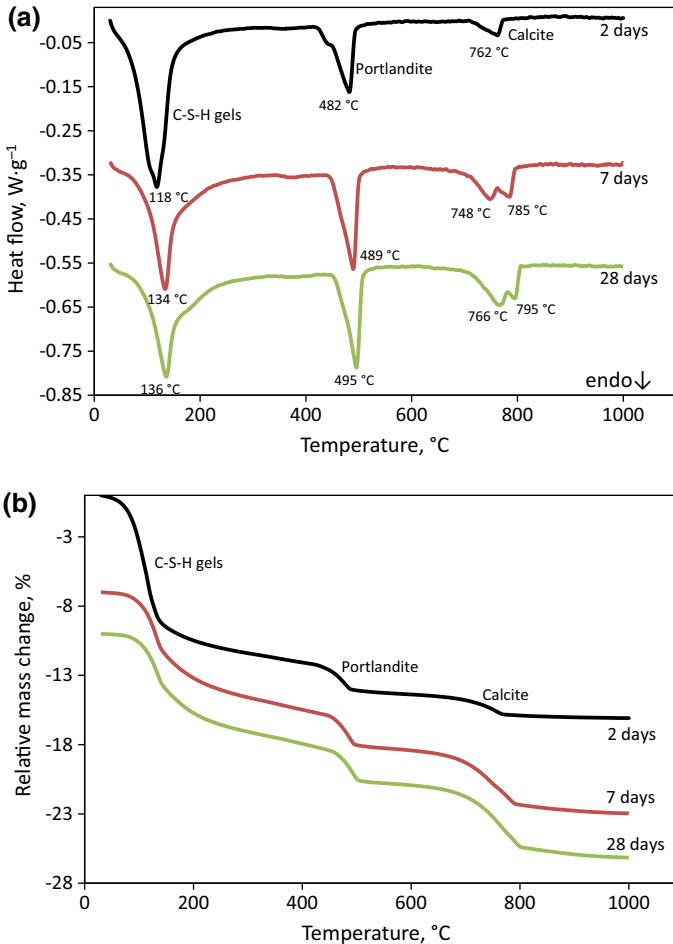
**Fig. 3** Heat flow (a) and relative mass change (b) of the cement paste containing 16 mass% of ceramic powder

The second endothermic peak corresponds to the portlandite decomposition and its peak temperature increases with the hydration time for the samples C8 and C16, and reaches 495 °C for 28 days samples. For the sample C24 the peak temperature is lower (486 °C for 28 days curing). The temperature of the peak decreases with the amount of the ceramic powder from 502 °C to 486 °C (Figs. 2a, 3a, 4a). The mass loss decreases with the hydration time of the samples, except for the sample C16 for which the mass loss increases. The increasing amount of ceramic powder in the samples causes a decrease of the mass loss. For 28 days samples it is 2.9 % for the reference sample and 1.9 % for the sample C24 (Table 3).

Finally, the last reactions in Figs. 2, 3, and 4 correspond to the calcite and vaterite decomposition. The peak of the vaterite decomposition is not visible for 2 days samples. It can be observed only for 7 and 28 days curing, and the peak temperature of this
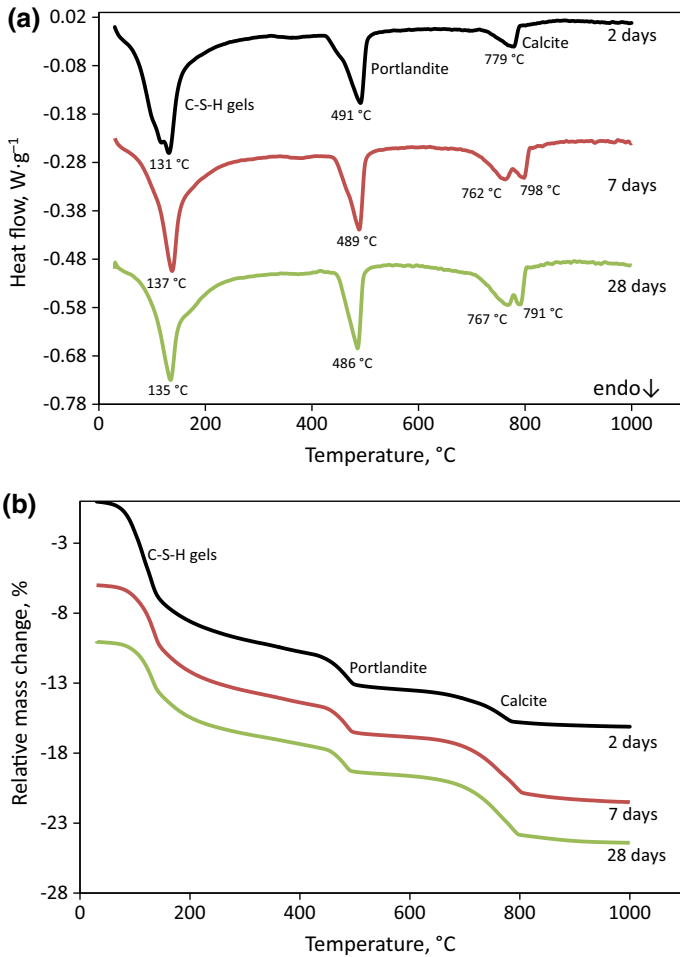
**Fig. 4** Heat flow (a) and relative mass change (b) of the cement paste containing 24 mass% of ceramic powder

reaction increases for all studied samples (from 751 °C to 776 °C for C8, from 748 °C to 766 °C for C16, and from 762 °C to 767 °C for C24), see Figs. 2a, 3a, and 4a. The peak of the calcite decomposition increases for the samples C8 (from 771 °C to 802 °C) and C16 (from 762 °C to 795 °C). For 28 days curing the peak temperature decreases with the amount of ceramic powder (from 802 °C to 791 °C). The mass loss of the studied samples increases with the hydration time and for the 28 days sample it decreases with the amount of cement replacement (from 5.5 % for C8 to 4.4 % for C24), see Table 4.

## 3.2 Portlandite Content

The portlandite content was estimated from the thermogravimetry results using a known value of the mass change during the decomposition of pure Ca(OH)$_2$ (which

**Table 5** Portlandite content in the samples containing 0 mass% to 24 mass% of ceramic waste in dependence on hydration time (in %)

| Time (days) | REF | C8 | C16 | C24 |
|---|---|---|---|---|
| 2 | 12.47 | 10.68 | 10.11 | 9.61 |
| 7 | 12.06 | 10.76 | 9.86 | 8.22 |
| 28 | 11.96 | 10.11 | 8.22 | 7.60 |

is 24.34 % [31]) and the mass change in the same temperature interval of the studied samples.

The obtained results show (Table 5) that the amount of portlandite in the studied samples slightly decreases with the hydration time from 12.5 % to 12 % for reference sample REF, from 10.7 % to 10.1 % for sample C8, and from 9.6 % to 7.6 % for sample C24. For the sample C16 the portlandite content increases from 8.2 % to 10.1 %.

### 3.3 Enthalpy

The enthalpy and temperature of reactions occurring in the studied samples after heating were determined from the DSC peaks (Figs 1a, 2a, 3a, 4a). The first reaction was associated with the liberation of physically bound water and C–S–H gel decomposition. The results summarized in Table 6 show that the temperature and enthalpy decrease with the increasing amount of ceramic powder in the 28 days samples. The enthalpy ranges from 194.03 J·g$^{-1}$ for REF to 151.70 J·g$^{-1}$ for C24. It decreases with the hydration time for all studied samples.

The second studied reaction represented the portlandite decomposition. The results are presented in Table 7. The temperature and enthalpy decrease with the increasing amount of ceramic powder. The enthalpy of the sample REF is for 28 days samples 86.70 J·g$^{-1}$, while for the sample C24 it is only 51.92 J·g$^{-1}$. These findings provide an information on the magnitude of the pozzolanic reaction in the studied concretes which consumes a part or the available Ca(OH)$_2$.

The results obtained for the vaterite and calcite decomposition are summarized in Table 8. The enthalpy of the corresponding reactions is difficult to determine, because the peaks overlap, so the calculated enthalpy is an effective value that includes both processes. It was observed that the temperature and the corresponding enthalpy of the reactions decreased with the amount of the ceramic powder (it was 81.00 J·g$^{-1}$ for REF and 70.83 J·g$^{-1}$ for C24). The enthalpy and temperature increased with the hydration time of the sample.

### 3.4 Compressive Strength

The measurements of the compressive strength of the studied samples are summarized in Table 9. The sample REF has the highest values of the compressive strength for all studied hydration times (it is ∼53.2 MPa for 28 days cured sample), whereas the lowest values were observed for the samples containing 24 mass% of ceramic powder (∼42.2 MPa for 28 days cured sample). The compressive strength increases with the

**Table 6** Enthalpy and temperature of reaction at C–S–H gels decomposition in dependence on hydration time for the samples containing 0 mass% to 24 mass% of ceramic waste

| Time (days) | REF | | C8 | | C16 | | C24 | |
|---|---|---|---|---|---|---|---|---|
| | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) |
| 2 | 122.58 | 244.92 | 132.17 | 211.39 | 118.15 | 221.68 | 131.34 | 196.06 |
| 7 | 136.02 | 200.13 | 136.39 | 185.97 | 133.90 | 173.89 | 137.38 | 165.22 |
| 28 | 139.79 | 194.03 | 135.94 | 177.08 | 136.14 | 167.51 | 134.54 | 151.70 |

**Table 7** Enthalpy and temperature of reaction at portladite decomposition in dependence on hydration time for the samples containing 0 mass% to 24 mass% of ceramic waste

| Time (days) | REF | | C8 | | C16 | | C24 | |
|---|---|---|---|---|---|---|---|---|
| | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J·g$^{-1}$) |
| 2 | 500.64 | 89.54 | 488.53 | 79.49 | 482.18 | 73.80 | 491.15 | 62.62 |
| 7 | 491.85 | 67.62 | 493.43 | 76.84 | 489.46 | 70.48 | 488.60 | 56.40 |
| 28 | 502.21 | 86.70 | 494.94 | 68.45 | 495.48 | 60.55 | 485.70 | 51.92 |

**Table 8** Enthalpy and temperature of reaction at vaterite and calcite decomposition in dependence on hydration time for the samples containing 0 mass% to 24 mass% of ceramic waste

| Time (days) | REF | | C8 | | C16 | | C24 | |
|---|---|---|---|---|---|---|---|---|
| | Temperature of peak (°C) | Enthalpy (J.g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J.g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J.g$^{-1}$) | Temperature of peak (°C) | Enthalpy (J.g$^{-1}$) |
| 2 | 779.28 | 31.89 | 771.33 | 32.13 | 761.91 | 14.68 | 778.86 | 25.38 |
| 7 | 832.47 | 78.52 | 788.28 | 61.92 | 784.88 | 64.64 | 797.92 | 62.48 |
| 28 | 804.97 | 81.00 | 802.03 | 78.69 | 794.95 | 73.98 | 791.21 | 70.83 |

**Table 9** Compressive strength of the samples containing 0 mass% to 24 mass% of ceramic waste in dependence on hydration time (in MPa)

| Time (days) | REF | C8 | C16 | C24 |
|---|---|---|---|---|
| 2 | 26.62 | 19.61 | 17.49 | 16.13 |
| 7 | 41.56 | 35.38 | 31.79 | 31.75 |
| 28 | 53.23 | 47.12 | 43.02 | 42.16 |

**Table 10** Strength activity index of the samples containing 0 mass% to 24 mass% of ceramic waste in dependence on hydration time (in %)

| Time (days) | C8 | C16 | C24 |
|---|---|---|---|
| 2 | 73.7 | 65.7 | 60.1 |
| 7 | 85.1 | 76.5 | 76.4 |
| 28 | 88.5 | 80.8 | 79.2 |

hydration time of the sample for all studied materials. On the other hand, it decreases with the amount of ceramic powder in the sample.

The strength activity index (SAI) was used to determine whether the ceramic powder incorporation into the blended cement results in an acceptable level of strength. The strength activity index is in ASTM C311 defined as SAI = (A/B) × 100, where A is the average compressive strength of blended cement mortar, and B the average compressive strength of control mortar samples. According to ASTM C618, the mixture with supplementary cementing material should provide 75 % of the strength of the reference mixture at 7 or 28 days. In our case, the SAI for 28 days cured samples varied between ∼88.5 % for the material with 8 mass% ceramic powder and ∼79.2 % for the 24 mass% ceramic powder dosage (see Table 10). Taking into account the limit of SAI, the studied ceramic powder is applicable for all studied substitutions of Portland cement. Nevertheless, one can assume that for higher amounts of ceramic powder in the blended binder its substantial part will not participate in the hydration process and act as a fine aggregate only, similarly to findings reported in [32].

## 4 Conclusions

In this paper, the possibility of using waste ceramic powder as a pozzolanic addition to Portland cement was studied. The thermal and mechanical characterization methods applied for the blended cement pastes included DSC and TG in the temperature range of 25 °C to 1000 °C with a heating rate of 5 °C·min$^{-1}$ in an argon atmosphere and the compressive strength measurements according to the European standards for cement mortars.

The thermal analysis showed changes in the composition of the studied samples at high temperatures. The temperature, enthalpy, and mass changes of the liberation of physically bound water and C–S–H gel dehydration, portlandite decomposition, and the vaterite and calcite decompositions were identified and quantified. The content of portlandite in the samples was found to have a decreasing course during the analyzed hydration time for all blended binders. This data provided the evidence of the pozzolanic activity of ceramic waste powder even within the limited monitoring time of

28 days. Taking into account also the favorable results obtained in the measurement of compressive strength, it can be concluded that the applied waste ceramic powder can be successfully used as a supplementary cementing material to Portland cement in an amount of up to 24 mass%.

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