

# Fine demolition wastes as Supplementary cementitious materials for CO<sub>2</sub> reduced cement production

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## ARTICLE INFO

### Keywords:

Demolition waste  
Mixed waste  
Supplementary Cementitious Materials  
Thermal treatment  
Hydraulic activity  
Pozzolanic activity  
Inert filler

## ABSTRACT

Construction and demolition waste accounts for a significant amount of the total solid waste produced worldwide, and its recycling is challenging. Although some demolition waste is processed into recycled sand and rubble, the finer fractions resulting from screening and washing of recycled aggregates are not used. This research investigates the potential of use of real demolition wastes, namely concrete screening fines (CS), mixed concrete-ceramic screening fines (MS), and mud from recycled aggregates washing (WM), as supplementary cementitious materials (SCMs) in eco-efficient blended cement. The study employed various experimental methods, such as isothermal calorimetry, thermogravimetric analysis (TGA), and setting time tests, to evaluate the hydraulic activity of waste materials and the Chapelle test and TGA to assess their pozzolanic activity. The mechanical properties and microstructure of mortars containing 20% of waste powders were evaluated using compressive strength tests and scanning electron microscopy (SEM). The results showed that thermal treatment of waste materials at 500 °C improved the mechanical properties of mortars, increasing Strength Activity Index (SAI) by 10% for CS and MS and by 6% for WM after 90 days of curing. All three waste types achieved similar mechanical properties, with compressive strengths of at least 37.93 MPa, 46.25 MPa, and 51.33 MPa after 7, 28, and 90 days of curing, respectively. The contribution of waste powders to mortar strength was due to filler effect and partially dehydrated C-S-H products. However, pozzolanic ceramic inclusions in waste powders did not affect mortar strength at a 20% substitution rate. Therefore, the research findings indicate that waste materials derived from demolition can potentially be used as environmentally friendly materials in construction. Their use as SCMs with a substitution rate of 20% can reduce the CO<sub>2</sub> emissions of cement production by at least 10.7%.

## 1. Introduction

Worldwide, construction and demolition waste (CDW) accounts for 30–40% of the total solid waste produced [1,2]. A large part of these wastes are inert materials such as concrete and ceramics, and although they are not hazardous, their recycling is quite challenging. Currently, part of the demolition waste is successfully processed into recycled sand and recycled rubble, which are used as aggregates in road works [2]. In addition, there is a growing interest in exploring the possibility of using these materials to create high-strength and long-lasting concrete and mortar [3–5]. The use of recycled aggregates for concrete production faces a major challenge of adherent hydrated cement, resulting in a double interfacial zone between the aggregate and the new cement matrix [6–10]. This causes a decline in the strength of concrete made with recycled aggregates. Hence, to enhance aggregates quality, it is necessary to clean them of the old cement matrix. Numerous studies

have explored methods to improve the quality of recycled aggregates [11–13] including mechanical [14], thermal [5,15] and chemical [7,8,16–19] approaches. Nevertheless, in practice, processing plants primarily utilize screening and washing to remove small, non-conforming fractions from the aggregates. However, even this method generates a large amount of secondary wastes such as screening fines and washing sludge, which are still not used. Thus, more than two thirds of CDW are disposed in the landfills [20], which not only disfigure the landscape, but can also change the composition of the soil and groundwater and thereby lead to the disruption of ecosystems [1,21]. In addition, dusty waste from screening and washing in dry weather conditions forms aerosols, that adversely affects air quality.

Recently, due to the threat of global climate change, studies on the possibility of using CDW as Supplementary Cementitious Materials (SCMs) [22–30] and as an alternative raw material for the production of cement clinker [25,31–35] have been increasingly conducted since the

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<https://doi.org/10.1016/j.conbuildmat.2023.131991>

Received 21 March 2023; Received in revised form 17 May 2023; Accepted 29 May 2023

Available online 6 June 2023

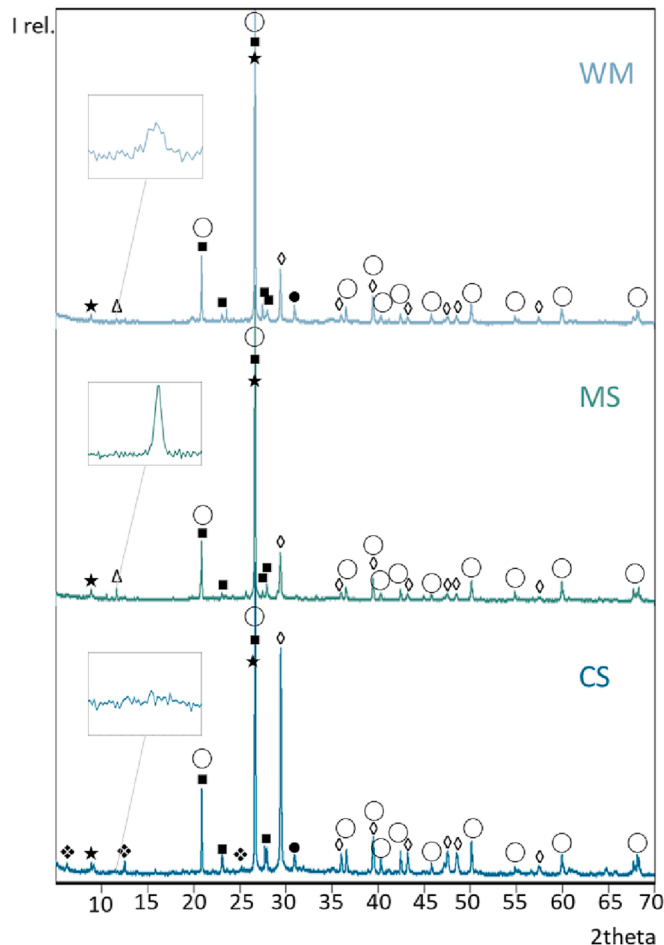
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**Table 1**

Oxide composition of the investigated materials, mass-%.

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	TiO <sub>2</sub>	MnO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	LOI*
CS	28.42	5.90	4.12	33.29	1.63	0.45	0.12	0.35	1.25	0.17	0.80	23.50
MS	41.94	8.10	5.51	21.24	1.11	0.77	0.11	0.47	2.15	0.44	2.56	15.60
WM	44.60	9.42	5.83	17.76	1.37	0.81	0.11	0.30	2.30	0.34	1.27	15.90
CEM I	16.07	3.91	3.58	66.72	1.45	0.37	0.08	0.26	1.18	0.39	3.88	2.10

\* Loss on ignition at 950 °C.

**Fig. 1.** XRD patterns of the investigated materials: ○ – quartz, ◇ – calcite, ■ – feldspars, ★ – muscovite, ● – dolomite, ◆ – chlorites, Δ – gypsum.

production of Ordinary Portland cement is associated with high CO<sub>2</sub> emissions. For the countries of the European Union, the use of CDW as a cement substitution is a promising area of research, since traditional SCMs such as natural pozzolans, fly ash and granulated blast furnace slag have limited availability [36,37]. On the other hand, according to the analysis performed on the example of Luxembourg by Bogoviku and Waldmann [38], construction and demolition waste should cover the demand for materials to produce new cement for at least the next 80 years.

In addition, studies of the characteristics of concrete after a fire have shown that, after contact with water, concrete that has lost its strength under the influence of high temperatures is capable of rehydration and partial restoration of mechanical properties [39–42]. This gave impetus to research on the possibility of reactivating cement-based waste for use as a hydraulic binder or hydraulic SCM. Thus, according to Splittgerber and Mueller [40], the hydration of pure clinker is a completely reversible process. However, during the dehydration of cement containing gypsum additional intermediate phases are formed, such as ye'elimite.

Zhang et al. [43] and Shi et al. [44] also state that when hydrated cement paste is calcined at temperatures above 500 °C, the resulting product includes β-C2S, which is a component of Portland cement, and lime. However, other studies [45–50] have shown that β-C2S is only formed at temperatures above 800 °C. At lower temperatures, depolymerization of C-S-H resulted in the formation of α-C2S, which is a more reactive polymorph of C2S than β-C2S. This explains the higher initial strength of heat-treated cement pastes compared to Portland cement, as well as the non-linear increase in the strength of samples from processed cement depending on the processing temperature. Thus, 600–750 °C was named as the optimal range of activation temperatures [45,46,48,50]. On the other hand, Wang et al. [51] state that the optimum treatment temperature for hydrated cement is 450 °C, at which partial dehydration of C-S-H has already taken place, but decarbonization of calcite particles has not yet begun, that provided filler and nucleation effect. It also should be noted that C3S is not formed during the thermal treatment of hydrated cement [47,52,53].

In general, according to the literature, the processes occurring during the heat treatment of hydrated cement can be considered as follows [23,46,47,52,54,55,56]:

- 20 – 130 °C: elimination of evaporable water and a part of the bound water.
- 110 – 200 °C: dehydration of ettringite and gypsum.
- 140 – 450 °C: dehydration of calcium silicate hydrates and carboaluminate hydrates.
- 450 – 650 °C: formation of lime due to portlandite dehydroxilation, and formation of α-C2S due to calcium silicates depolymerization.
- 600 – 900 °C: formation of lime due to calcite decarbonization, and polymorphic transformation of α-C2S to β-C2S.
- Above 1000 °C: melting.

The study of the rehydration of heat-treated cement carried out by Bogas et al. [47,49], Real et al. [48] and Carriço et al. [50] showed the formation of C-S-H, portlandite and ettringite, similar to those found in Portland cement. In addition, calcium carboaluminate hydrates and AFm phases were detected already after 8 h of rehydration. Moreover, the recycled cement showed higher degree of hydration in the early stages due to the high reactivity of α-C2S and CaO, but by the 28th day, the hydration was significantly reduced compared to Portland cement, leading to weaker strength gain.

It is important to note that all the above studies on thermal activation of demolition waste focus on research conducted on laboratory-made hydrated paste and concrete as the precursor material. This approach eliminates the influence of impurities and allows for more controlled and accurate results. However, it is crucial to investigate the thermal activation of actual demolition waste as it may differ significantly from laboratory-prepared samples. The presence of impurities and contaminants, such as calcite and quartz derived from aggregates, as well as high levels of carbonization resulting from prolonged service life, can greatly impact the thermal activation process. Understanding the thermal activation of real demolition waste is crucial for the development of effective and sustainable waste management strategies and for reducing the environmental impact of construction and demolition activities.

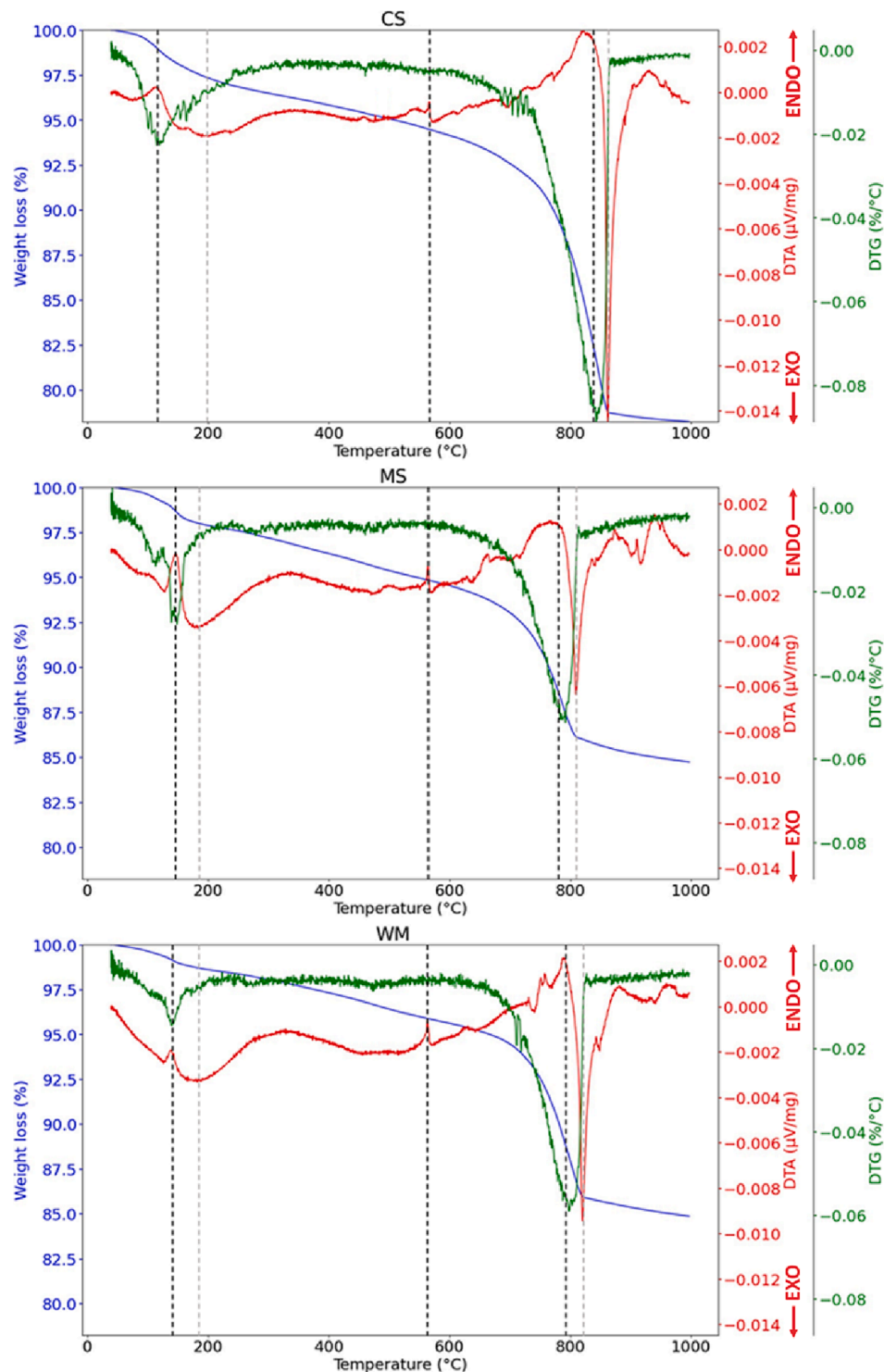


Fig. 2. TGA/DTG/DTA curves of investigated materials.

## 2. Goals and significance of the research

This paper presents the results of a study investigating the recycling potential of fine waste generated during the production of recycled aggregates. The study focuses on the thermal activation of real demolition waste, which has not been extensively studied in previous comparable works. A comparative table of available works on the use of dehydrated cement in the production of binders is provided in the appendix to this article.

The materials studied in this paper are waste from the processing of

recycled aggregates, which have already been exposed to an entire life cycle in a structure and thus have a high degree of carbonation and high impurities. Screening fines and wash mud, that are the final wastes from recycled aggregates processing, have not yet found a practical application due to their substandard granulometry and high concentration of organic impurities and gypsum.

The main objective of the research is to understand whether the investigated materials possess hydraulic activity, or they can be used as inert fillers, which are important properties for the use of waste materials in the production of blended cement. The study aims to identify the

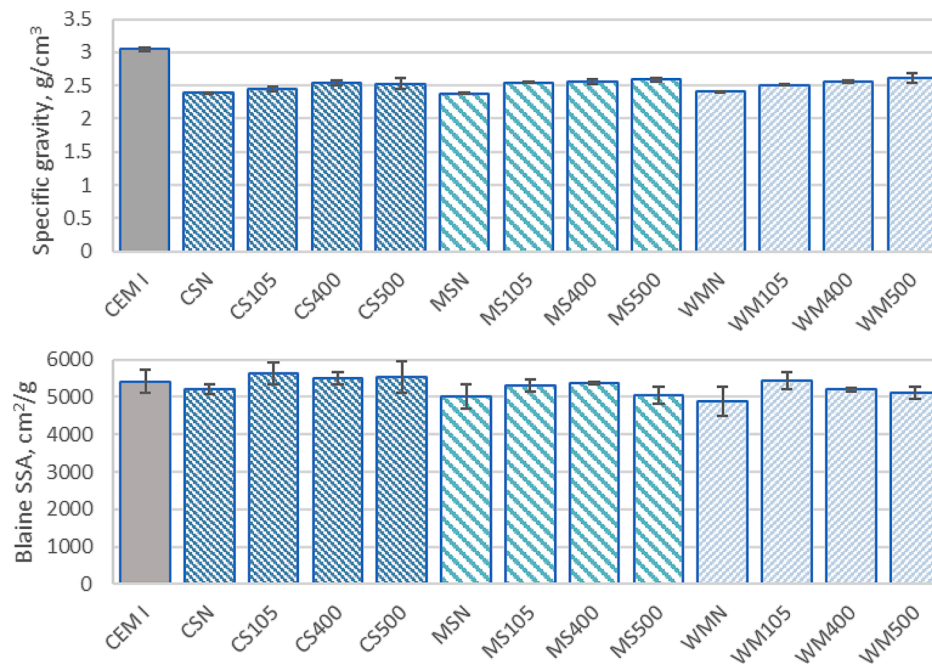


Fig. 3. Physical properties of powders treated at different temperatures.

possibility of using these fines generated from the processing of demolition waste in the production of cement with reduced CO<sub>2</sub> emissions. As discussed in section 1, the process of decarbonization in concrete-containing waste typically starts at around 600 °C, while the breakdown of hydration products and the activation of hydrated cement can occur as low as 450 °C. To avoid excessive CO<sub>2</sub> emissions from the decarbonization of raw materials and fuel combustion, this study has chosen to utilize treatment temperatures no higher than 500 °C to minimize the environmental impact of the treatment process while still achieving the desired outcomes.

The novelty of the study lies in the fact that it considers the mud from the washing of recycled aggregates, which has not yet been studied, and the effect of thermal activation on mixed demolition waste, including the pozzolanic activity of ceramic inclusions. This can be an important factor, since according to Bogas et al. [47], the high amount of CaO found in dehydrated cement quickly reacts with water, providing ample CH for pozzolanic reactions. The combination of pozzolanic admixtures with thermally activated cement may address some of the main issues reported in the literature, such as short setting time and high water demand.

The present research aims to contribute to the growing body of knowledge on the use of real demolition waste in the production of cement. The study will provide valuable information on the feasibility of using wash mud and mixed demolition waste in the production of cement with reduced CO<sub>2</sub> emissions and will fill the gap in the existing literature on the topic.

### 3. Materials

#### 3.1. Raw materials description

The materials investigated in this study were three types of fine demolition waste provided by the Belgian company Tradecowall, which processes inert waste into recycled aggregates and sand. During processing, the aggregates go through several screening stages, as a result of which they are separated into different fractions. Substandard fractions sifted out in the screening process cannot be used as aggregates and are considered as final waste which is stored on site. After screening, the recycled aggregates are washed forming a wash mud which is also stored

on site.

Approximately 30 kg of three types of demolition waste fines were sampled:

- Screening waste from concrete (CS) aggregates processing consisted of concrete fines mixed with a minor proportion of impurities, including metal reinforcement debris.
- Screening waste from mixed (MS) aggregates processing consisted of about 60% of concrete, 30% of ceramics, glass and stones with minor impurities of organic materials such as wood, bitumen, polymers and fabrics.
- Mud from recycled aggregates washing (WM), which was a dark gray mass with a light petroleum-like odor.

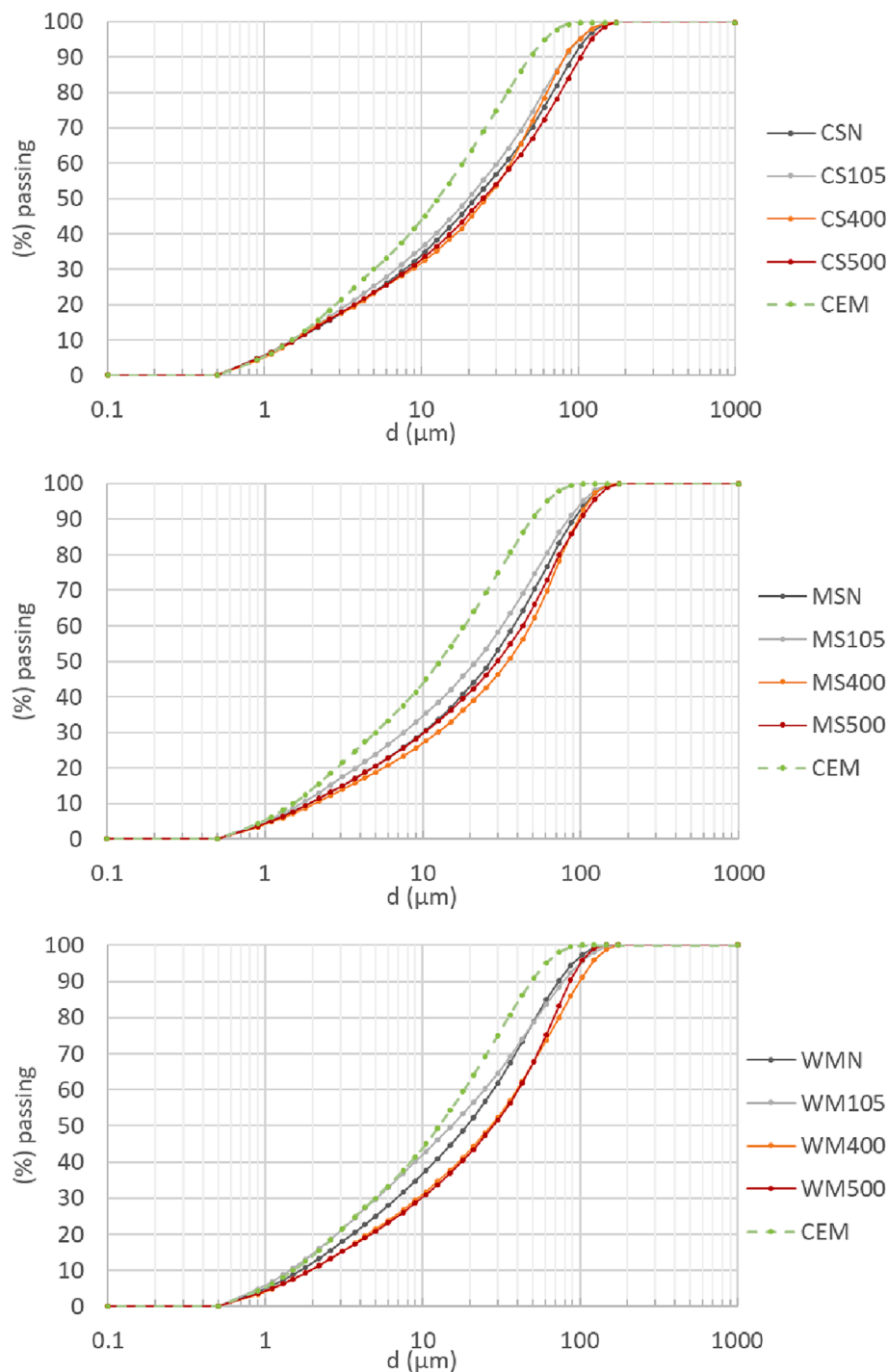
According to the manufacturer, the size of the screening waste and wash mud used in this study was 0/8 mm and 0/63 µm respectively. Moisture content in the screening wastes was about 13 mass-%, while in the wash mud was 22 mass-%.

Besides the investigated wastes in the research, a Portland cement CEM I 52.5 R with specific gravity of 3.05 g/cm<sup>3</sup> and Blaine specific surface area of 5418 cm<sup>2</sup>/g provided by the French manufacturer Vicat, CEN Standard Sand according to EN 196-1 [57], and tap water were used for the mortar specimens mixing.

#### 3.2. Materials preparation

After sampling in the field, the materials were separated into two groups and dried in two different modes: at room temperature and at 105 °C to constant mass. Dried materials were subjected twice to crushing. Preliminary crushing was carried out in a jaw crusher, and the final crushing was performed in an impact mill with a bottom sieve of 2 mm mesh size. After crushing, the wastes were sieved on a 125 µm mesh sieve for their separation into two fractions: 0.125/2 mm and 0/125 µm. Regardless of the drying method, the finer fraction of CS and MS accounted for one third of the total material, while in the case of WM, three quarters of the material consisted of particles smaller than 125 µm. The discrepancy of the WM fineness declared by the manufacturer can be explained by the partial cementation of the material during storage and drying, as well as by its contamination with larger debris. Fraction





**Fig. 4.** Particle size distribution of powders treated at different temperatures (CS – concrete fines, MS – mixed fines, WM – wash mud, CEM – Portland cement CEM I 52.5 R).

of 0.125/2 mm can potentially be used as recycled sand. In this study, only the 0/125  $\mu\text{m}$  fraction was used.

Thus, after drying, grinding and sieving, 6 investigated powders were obtained: concrete fines, mixed fines and wash mud dried in natural conditions (CSN, MSN and WMN), and dried at 105 °C (CS105, MS105 and WM105). Furthermore, the materials dried at 105 °C were calcined in a muffle furnace at 400 °C and 500 °C for 2 h with heating mode 10 °C/min without ventilation and subsequent natural cooling in a closed furnace. As a result, 6 more materials were obtained: CS400, MS400, WM400, CS500, MS500 and WM500.

## 4. Experimental methods

### 4.1. Characterization of investigated wastes

Dried in ambient conditions and crushed materials underwent thermal DTA/TGA analysis with a STA 449 F5 Jupiter thermal analyzer at air atmosphere and with a heating rate of 20 °C/min. After materials preparation, the changes in their mineralogical composition, density and fineness depending on the treatment temperature were investigated. Mineralogical composition was studied using D2 PHASER X-ray diffractometer with following parameters: Cu-K $\alpha$  radiation, 10 mA, 30 kV,

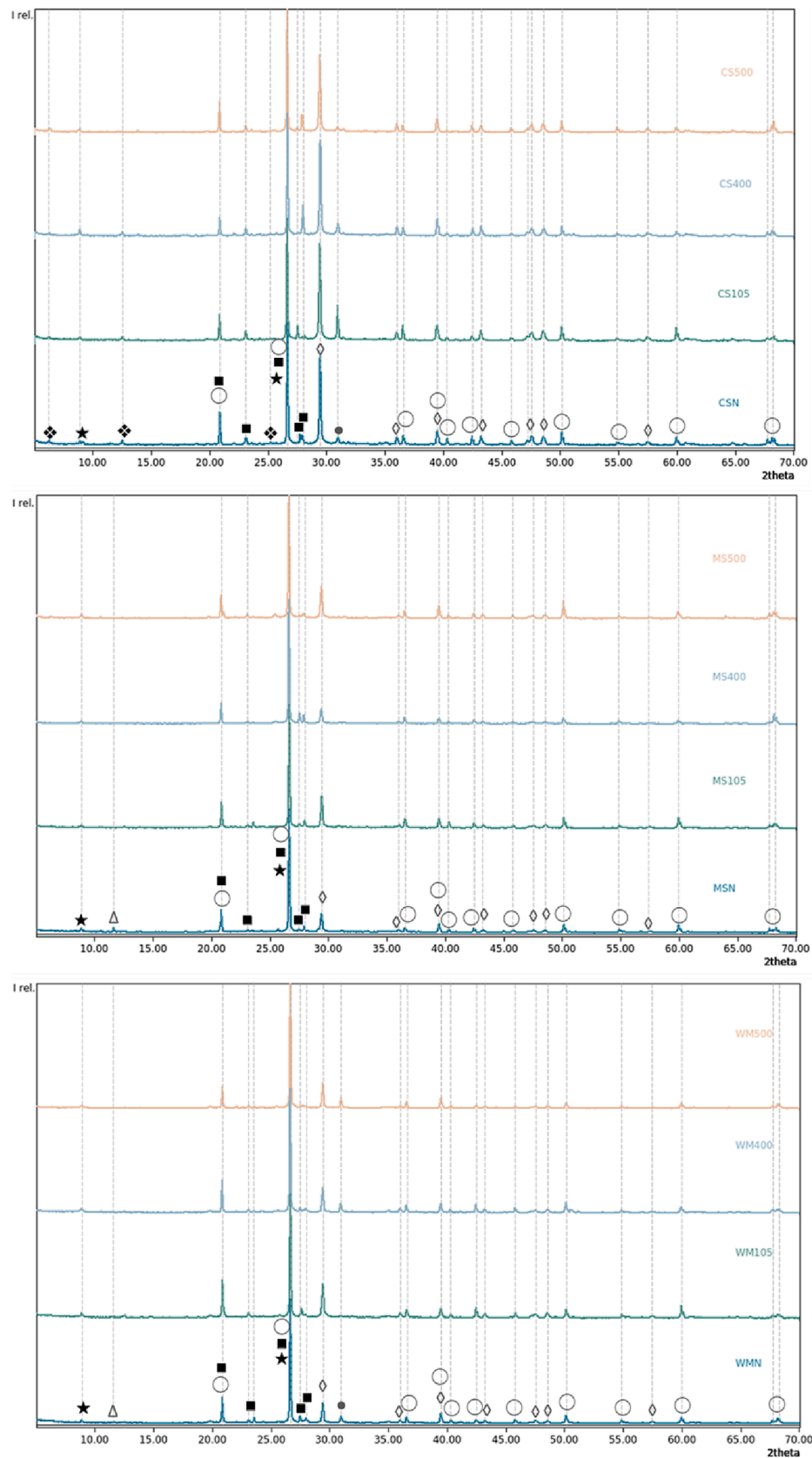
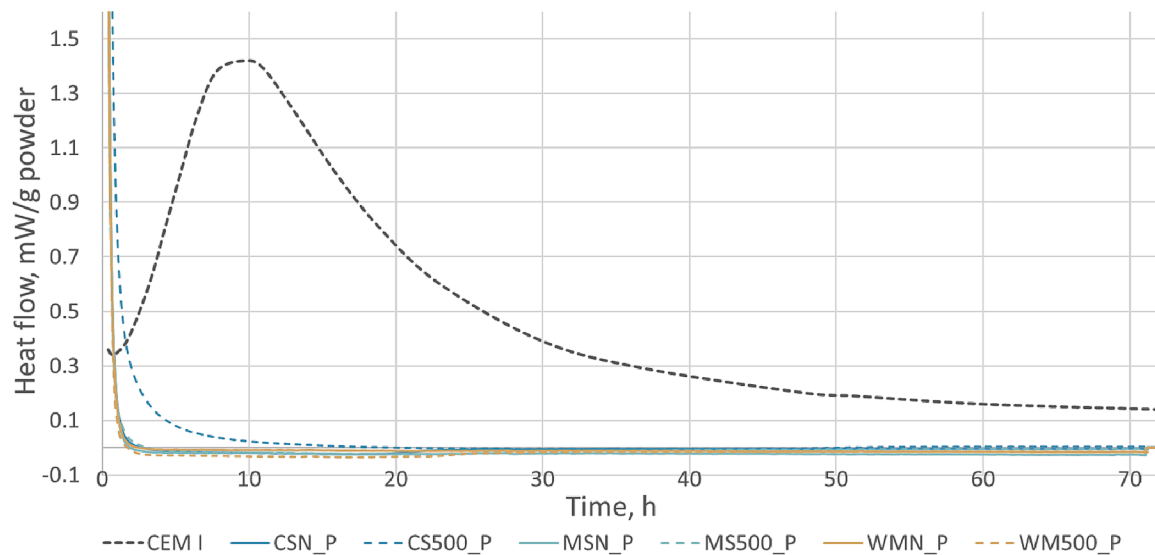
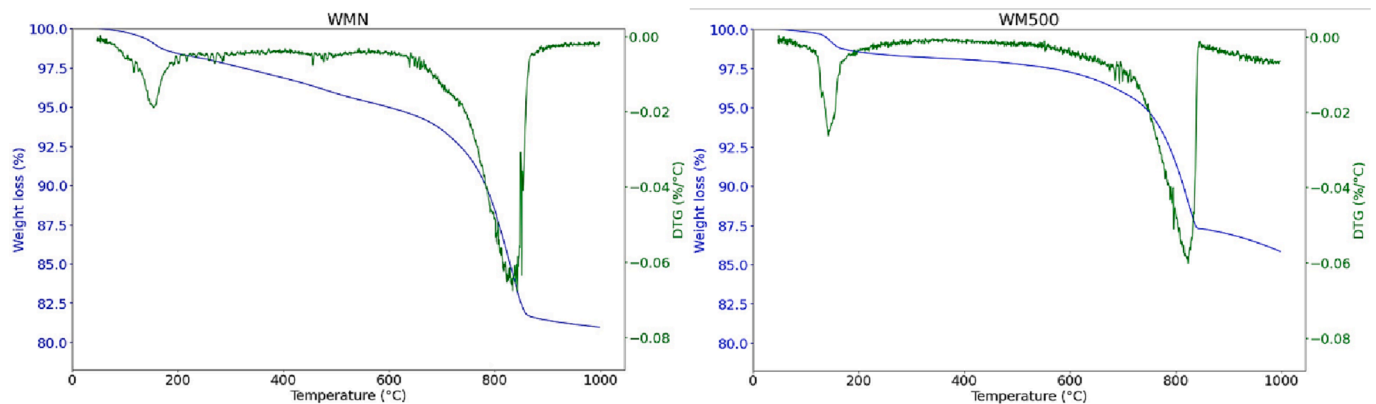


Fig. 5. XRD patterns of investigated materials before and after thermal treatment: ○ – quartz, ◇ – calcite, ■ – feldspars, ★ – muscovite, ● – dolomite, ◆ – chlorites, Δ – gypsum.

**Table 2**

Mineralogical composition of the investigated materials before and after thermal treatment, mass-%.

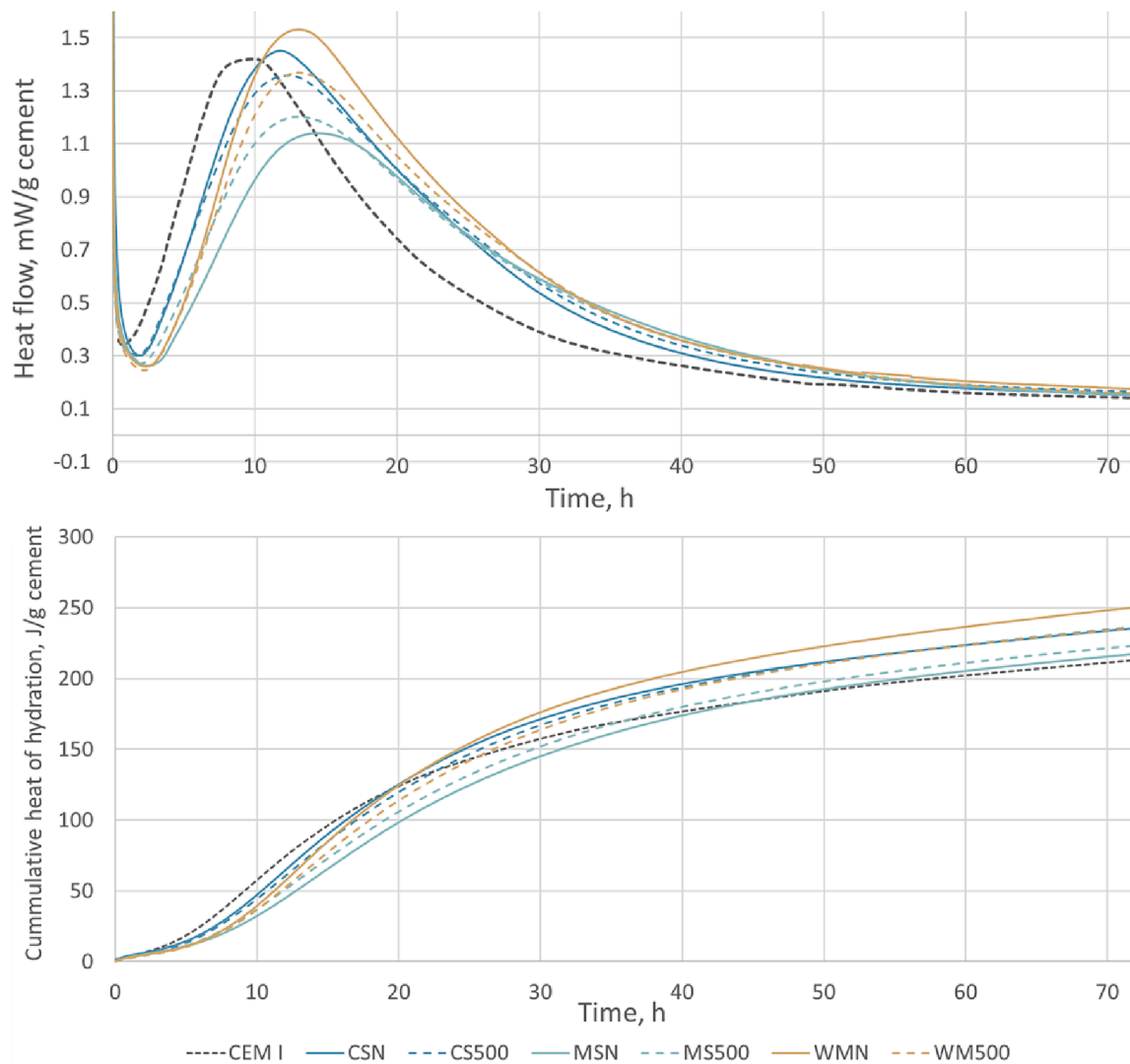
	Quartz	Calcite	Feldspars	Muscovite	Dolomite	Chlorites	Gypsum
CSN	35.2	33.6	13.4	5.7	6.5	5.6	–
CS105	30.4	38.7	13.1	5.2	7.7	4.9	–
CS400	26.9	35.2	18.4	8.2	7.5	3.8	–
CS500	38.0	35.9	13.3	5.8	5.5	1.5	–
MSN	52.7	20.1	13.0	10.0	–	–	4.2
MS105	54.6	23.6	14.7	7.1	–	–	–
MS400	56.7	20.1	15.0	8.2	–	–	–
MS500	58.4	20.9	12.4	8.3	–	–	–
WMN	46.4	17.1	15.5	12.7	6.2	–	2.1
WM105	47.6	25.4	13.1	12.2	1.7	–	–
WM400	45.2	18.2	17.3	12.4	6.9	–	–
WM500	47.7	19.0	10.6	12.4	10.3	–	–

**Fig. 6.** Heat flows of pure pastes: CEM I is paste consisted of 100% Portland cement; CSN\_P, MSN\_P, WMN\_P, CS500\_P, MS500\_P, WM500\_P are pastes consisted of 100% waste powder.**Fig. 7.** TGA/DTG curves of pastes consisted of 100% WMN and WM500 after 90 days curing.

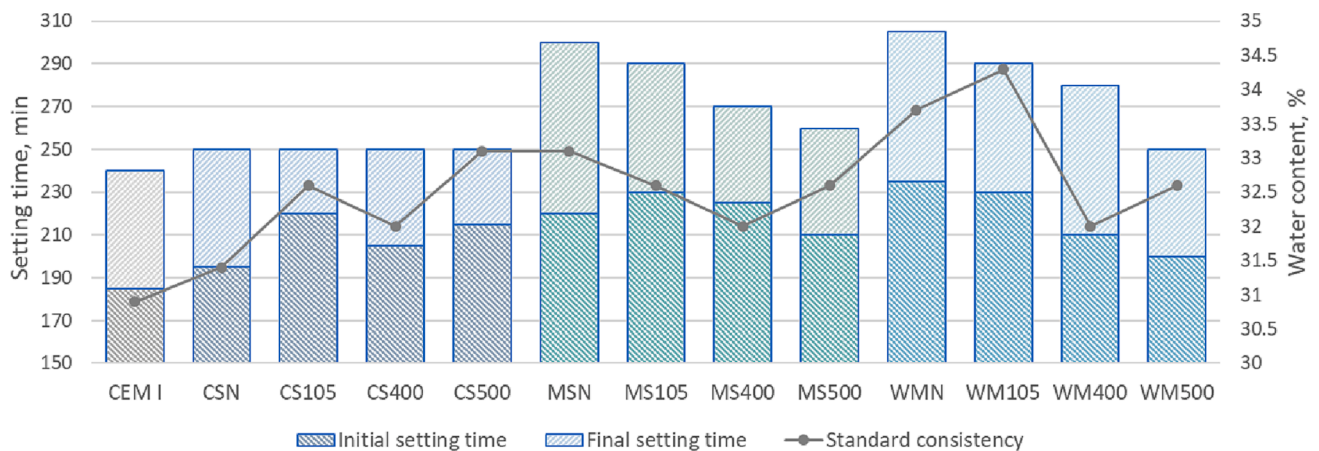
rotation between 5° and 70° with a 0.02° 2θ step and a step time of 0.6 s, and measured by Rietveld method using Profex software. The pycnometer method with petrol as liquid was used to determine the density. Fineness of the materials was determined by Blaine method for air permeability specific surface area measurement as well as by particle size distribution analysis obtained with HELOS & RODOS laser granulometer.

#### 4.2. Evaluation of hydraulic activity

The change in the hydraulic activity depending on the treatment temperature of the investigated powders was estimated by the measurement of the hydration heat using micro DSC 7 evo isothermal calorimeter. TGA of pastes made of 100% WMN and WM500 and w/b



**Fig. 8.** Heat flows and cumulative heat of hydration of pastes: CEM I is paste consisted of 100% Portland cement; CSN, MSN, WMN, CS500, MS500, WM500 are pastes containing 80% of Portland cement and 20% of waste powder.



**Fig. 9.** Standard consistency, initial and final setting times of pastes with 20% of SCMs.

ratio of 0.5 after 90 days curing and drying at 50 °C until weight constancy was carried out to check if the hydration products formed at later stages. In addition, the measurement of the setting time of binder pastes using a Vicat apparatus in accordance with the European standard EN

196–3 [58] was used, except that the pastes were not immersed in water. The dry part of paste mixtures consisted of 0.8 parts Portland cement and 0.2 parts SCM, the water content in the pastes was determined by standard consistency test in accordance with EN 196–3 standard [58].



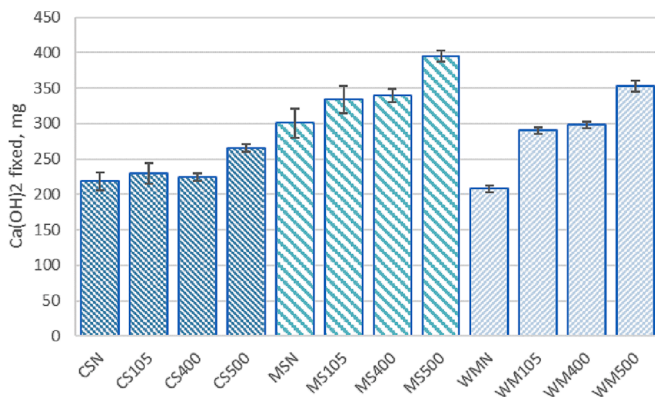


Fig. 10. Pozzolanic activity of investigated powders measured by modified Chapelle test.

#### 4.3. Evaluation of pozzolanic activity

The Chapelle test combined with the TGA were used to estimate the pozzolanic activity of the investigated materials.

For the Chapelle test, 1 g of investigated powder, 2 g of calcium oxide and 250 ml of distilled water were placed in a 500 ml Erlenmeyer flask. The flask with magnetic stir bar and laboratory condenser was placed on a magnetic-stirrer hot plate heated to a temperature of 85 °C. The mixture was stirred for 16 h. After cooling to a room temperature, the solution was mixed with 250 ml of saccharose syrup (0.7 M) and stirred for 30 min to extract unreacted lime. Then, the mixture was filtered on a filter paper and 25 ml of the filtrate were titrated with 0.1 N solution of hydrochloric acid using phenolphthalein as an indicator. To calculate the amount of fixed lime a blank test without pozzolan was carried out. The pozzolanic activity was calculated according to the formula:

$$\text{mg of Ca(OH)}_2 \text{ fixed} = 2 * ((V_1 - V_2) / V_1) * (M_{\text{Ca(OH)}_2} / M_{\text{CaO}}) * 100, \quad (1)$$

where  $V_1$  is the volume of HCl solution consumed in the blank test,  $V_2$  is the volume of HCl solution consumed in the pozzolan test,  $M_{\text{Ca(OH)}_2}$  is the molar mass of calcium hydroxide (74 g/mol),  $M_{\text{CaO}}$  is the molar mass of calcium oxide (56 g/mol).

TGA was carried out only on the paste samples with 20% of MSN and MS500 and w/b ratio of 0.4 after 90 days curing and drying at 50 °C until weight constancy to check if there are differences in portlandite consumption by ceramic particles.

#### 4.4. Evaluation of compressive strength

To assess the suitability of the materials for use as SCMs, their effect on the compressive strength of 40x40x160 mm mortar bars in accor-

dance with the ISO 679:2009 [59] international standard was studied. The water-to-binder (w/b) and sand-to-binder (s/b) ratios of all mixtures were fixed at 0.5 and 3 respectively. Portland cement substitution rate was set at 20%. This substitution rate was selected for the possibility of assessing Strength Activity Index (SAI) according to the ASTM C311/C311M standard [60]. The use of SAI is favorable for evaluating the effect of investigated materials on mortar strength and their suitability for use as SCMs. The specimens were cured in the moisture chamber at 20 °C during 7, 28 and 90 days before testing on a hydraulic press. Compressive strength test results of reference specimens and specimens containing 20% SCMs were used to calculate SAI using the following formula:

$$\text{SAI} = (R/R_{\text{ref}}) * 100\%, \quad (2)$$

where  $R$  is the average strength of the mortar bars with 20% of investigated SCMs,  $R_{\text{ref}}$  is the average strength of the referenced mortar.

#### 4.5. Microstructure of mortar specimens

After undergoing strength tests for 90 days of curing, the fragments remaining from the reference mortar specimens, as well as the mortar specimens CS500, MS500 and WM500, were stored in plastic bags for a year. To analyze the microstructure, they were coated with a 4 nm layer of gold and examined with a field emission scanning electron microscope JEOL JSM-6010LA.

### 5. Results and discussion

#### 5.1. Characterization of investigated wastes

The chemical composition of the investigated materials and cement obtained by X-Ray Fluorescence analysis is given in the Table 1. The main oxides of all wastes are  $\text{SiO}_2$  and  $\text{CaO}$ , followed by  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{K}_2\text{O}$  and  $\text{MgO}$ . As expected, MS and WM contain more silica, alumina, and iron oxides than CS due to ceramics and glass presence. However, the content of pozzolanic oxides in all the studied wastes is less than 70% that is not high enough to consider them as materials with significant pozzolanic activity. Nevertheless, the higher content of these oxides in MS and WM suggests some difference in the rehydration of these materials compared to pure concrete. The loss on ignition of concrete fines is noticeably higher than that of the other two materials, which indicates a higher content of cement hydration and carbonation products in CS.

The X-ray diffraction patterns of the investigated materials shown in Fig. 1, indicate the presence of high levels of quartz and calcite in all three samples. However, compared to CS, which contains approximately equal amounts of both minerals, MS and WM have reduced levels of calcite and elevated levels of muscovite, which can be attributed to the presence of ceramic particles and lower concrete content. The presence

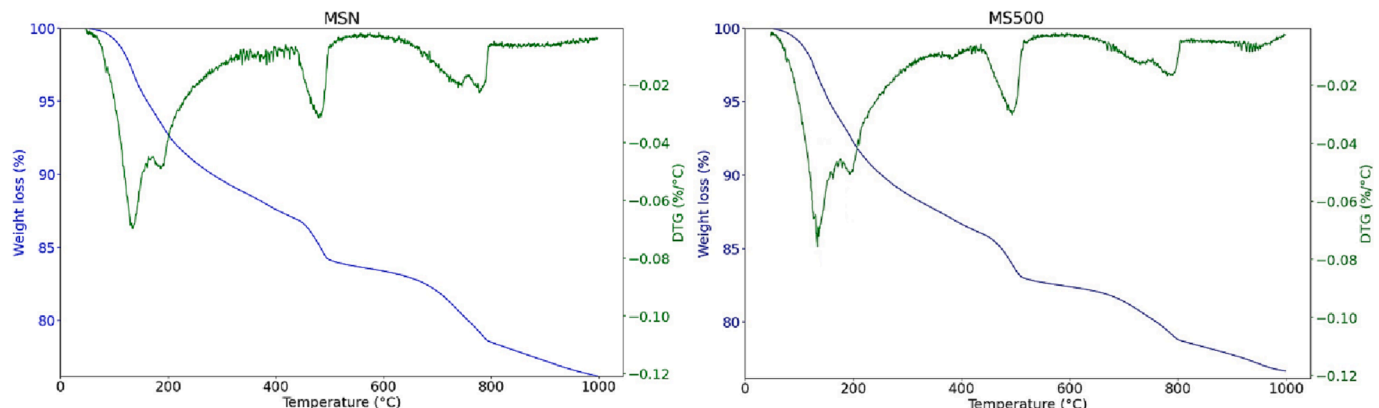


Fig. 11. TGA/DTG curves of pastes containing 20% of MSN and MS500 after 90 days curing.

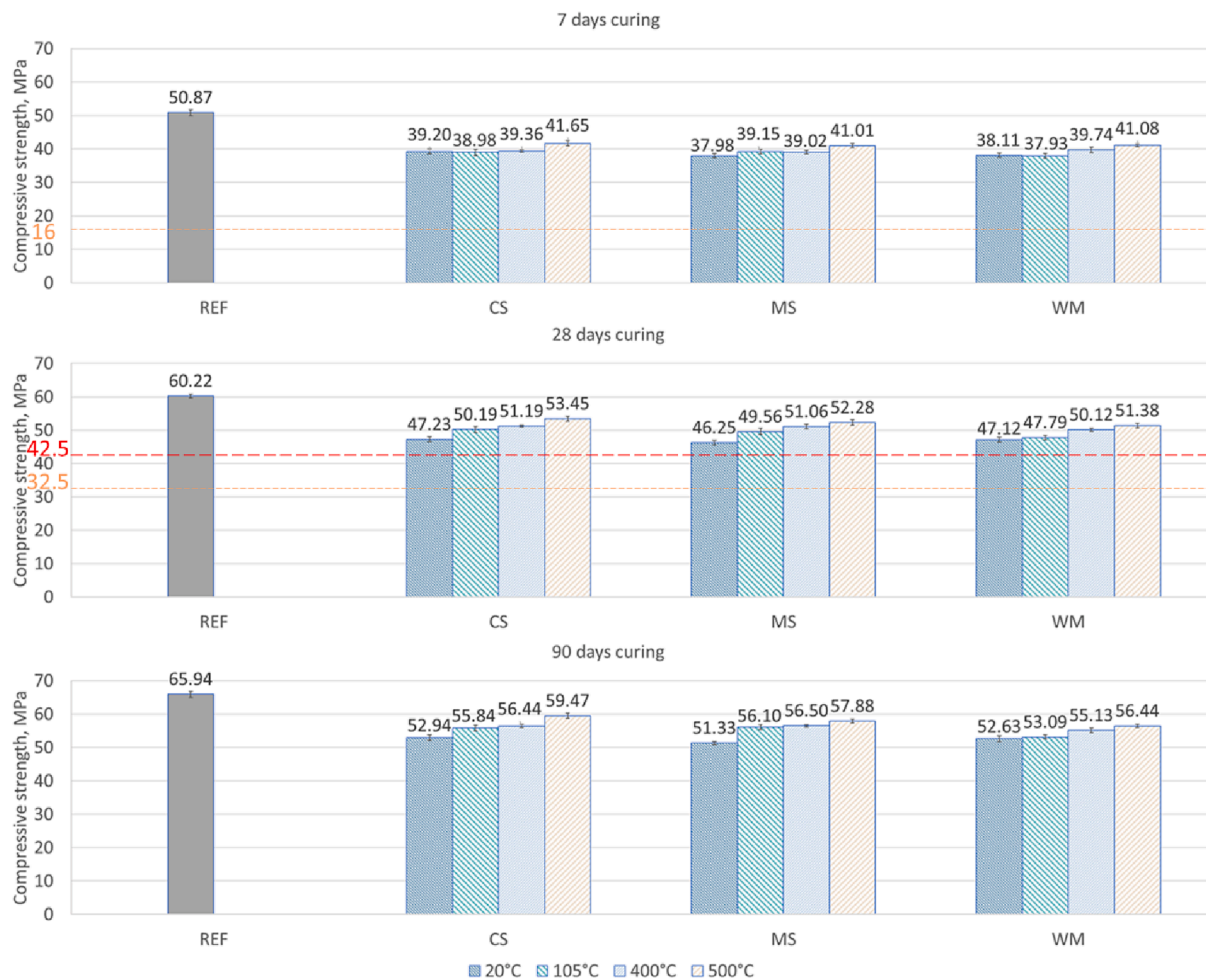


Fig. 12. Compressive strength of mortars containing 20% of SCMs treated at different temperatures.

of dolomite was detected in CS and WM, but not in MS. Furthermore, small amounts of gypsum were observed in MS and WM, with MS exhibiting a higher concentration. Clay minerals, chlorites, were exclusively detected in CS. The presence of a faint amorphous halo between 20 and 40°, especially in sample CS, and broad peaks indicates the presence of some moderate amount of amorphous phase in the materials.

Unlike the data of Gastaldi et al. [32], Florea et al. [23] and Shui et al. [54], mineralogical analysis of all investigated materials did not show the presence of portlandite and ettringite that have the main peaks at  $18.0^\circ 2\theta$  and  $9.1^\circ 2\theta$  respectively. The absence of these phases in the investigated materials is explained by their transformation into calcite over time as a result of carbonation processes. It should be noted that portlandite and ettringite are the phases that are important for reactivation of recycled cement by thermal treatment due to their dehydration and dehydroxylation, and the non-availability of these phases in the investigated wastes may indicate the ineffectiveness of heat treatment for their activation.

The results of the mineralogical analysis were confirmed by DTA/TGA analysis. Three main endothermic and two exothermic peaks are observed on the DTA curves of all materials shown in the Fig. 2. The first endothermic between 100 °C and 200 °C, accompanied by a weight loss of about 5% for CS and 2.5% for MS and WM, refers to the removal of free water and dehydration of calcium silicate hydrates. The second endothermic peak with a maximum at 570 °C, which is not accompanied by a weight change, is related to the polymorphic transformation of  $\alpha$ -quartz into  $\beta$ -quartz. The third endothermic peak around 800 °C related to the decarbonization processes is accompanied by a loss in

mass of 13%, 8.5% and 7.5% for CS, WM and MS respectively. The wide exothermic peak between 150 °C and 350 °C refers to the organic impurities burning processes, and the exothermic peak at 820 – 860 °C refers to the crystallization of wollastonite as a result of sintering of silicon oxide with calcium oxide released during calcite decarbonization. Portlandite dehydroxylation process was not detected for any material.

Since the thermal decomposition of cement hydration products, which should facilitate waste grinding, takes place already at a temperature of 100 – 200 °C, it can be expected that materials dried at increased temperature followed by grinding will have a higher fineness than materials dried in natural conditions. On the other hand, Carriço et al. [53] noticed that fine thermoactivated cement particles tend to agglomerate, which decreases their fineness. Also, considering that when materials are heated to a temperature of 500 °C, further decomposition of hydration products occurs as well as oxidation processes and organic matter burnout. Thus, it can be assumed that the thermal treatment of powders can affect their density, granulometric and mineralogical composition.

Fig. 3 shows the results of measuring the specific gravity and specific surface area (SSA) of the investigated materials. As expected, samples crushed after drying at elevated temperatures have a higher SSA than samples dried at room temperature. For CS, MS and WM this increase is from 5216, 5009 and 4888 to 5620, 5316 and 5436 respectively. However, after calcination, the fineness of the powders decreases from 5620, 5316 and 5436  $\text{cm}^2/\text{g}$  to 5539, 5049 and 5105  $\text{cm}^2/\text{g}$  for CS, MS and WM respectively. These data are confirmed by particle size distribution plots obtained using laser granulometry (Fig. 4). This can be



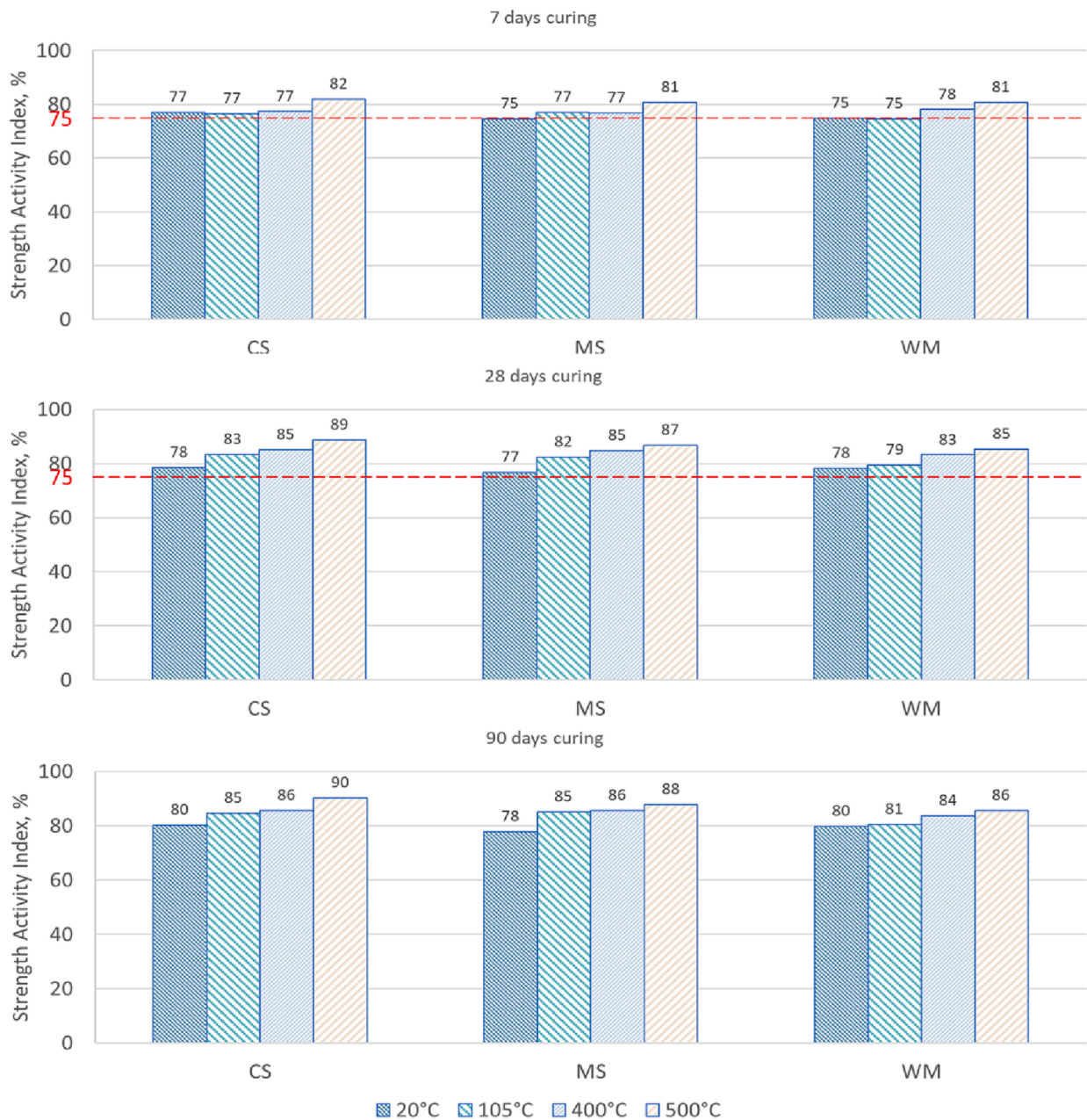


Fig. 13. Strength Activity Indices of mortars containing 20% of SCMs treated at different temperatures.

explained by the agglomeration of particles due to melting of low-melting inclusions. However, the difference in SSA and particle size distribution of all powders seems insignificant, and these values are quite close to those of Portland cement.

Regarding the change in density, from the Fig. 4, it can be seen that with an increase of treatment temperature, the density of materials slightly increases. Thus, after heating CS from room temperature to 500 °C, its density increased from 2.40 g/cm<sup>3</sup> to 2.53 g/cm<sup>3</sup> (5.4%), while the density of MS and WM increased from 2.38 g/cm<sup>3</sup> to 2.59 g/cm<sup>3</sup> (8.8%) and from 2.42 g/cm<sup>3</sup> to 2.61 g/cm<sup>3</sup> (7.9%) respectively. This increase in specific gravity is due to elimination of chemically bonded water and combustion of organic impurities present in the demolition wastes that have low density.

The mineral composition of the waste samples remained largely unchanged with increasing treatment temperatures, as indicated in Fig. 5 and Table 2. The only notable change was the disappearance of gypsum peaks in MS and WM and a decrease in chlorites peaks in CS. As

a result of its partial dihydroxylation, the content of chlorites presented dropped from 5.6 % in the unheated sample to 1.5% in the sample treated at 500 °C. Since no portlandite was detected in the starting material, and the heating temperatures were below the decarbonization point of calcite and dolomite, no peaks of free lime were observed on the X-ray diffraction patterns of the calcined materials. Similarly, no patterns showed C2S polymorphs, a product of C-S-H dehydration that gives a broad peak between 30 and 35° 2theta, as this phase seems to form at higher heating temperatures, according to various studies [23,45,47–50,54]. However, it is worth noting that some dehydration products may still be present in the amorphous phase, which cannot be indicated by XRD.

## 5.2. Hydraulic activity

The microcalorimetric analysis of the investigated pure waste materials revealed a heat flux only within the first 2 h of hydration for all

**Table 3**

SAI values for cement substitution rate of 20% by different materials: RC is recycled cement, LF is limestone filler, QF is quartz filler.

Additive material	Treatment temperature, °C	7 days SAI, %	28 days SAI, %	90 days SAI, %	Source
RC	500	–	77	–	[56]
RC	700	108	92	88	[50]
RC	–	81	74	–	[50]
RC	–	70	77	–	[23]
RC	500	66	68	–	[23]
RC	800	84	83	–	[23]
RC	–	74	69	75	[52]
RC	350	83	73	77	[52]
RC	650	88	78	78	[52]
RC	350	79	74	65	[52]
RC	650	85	79	82	[52]
RC	650	90	77	85	[52]
RC	650	92	88	95	[52]
Average SAI		77 ± 11	72 ± 6	72 ± 8	
LF	–	95	87	–	[74]
LF	–	91	83	–	[74]
LF	–	88	82	–	[74]
LF	–	77	76	–	[74]
LF	–	90	87	–	[74]
LF	–	88	88	–	[74]
LF	–	80	78	–	[74]
LF	–	77	77	–	[74]
LF	–	80	81	–	[75]
LF	–	83	81	84	[76]
LF	–	85	66	84	[77]
LF	–	80	84	82	[78]
Average SAI		85 ± 6	81 ± 6	83 ± 1	
QF	–	91	78	81	[79]
QF	–	74	69	76	[79]
Average SAI		83 ± 12	74 ± 6	79 ± 4	

samples, irrespective of the treatment temperature (see Fig. 6). These findings are consistent with the calorimetry results reported by Zhang et al. [61] for real demolition waste. However, they differ from the results of the analyses conducted on laboratory-made samples dehydrated at 450 °C, as reported by Wang et al. [51], as well as from those of laboratory-made samples dehydrated at higher temperatures, as reported by Splittgerber and Mueller [40], Bogas et al. [47,49] and Carriço et al. [50], who observed a heat flux peak between 10 and 20 h of hydration.

Xu et al. [46] attributed the initial heat release during the hydration of thermally activated recycled cement to the hydration of free lime. However, the materials investigated in this study do not contain free lime. Therefore, the initial heat release during hydration may have been caused by other factors, such as the dissolution of soluble compounds and the high reactivity of C-S-H dehydration products [47,49,50].

Fig. 7 shows TGA/DTG curves for pastes consisting of 100% WMN and WM500. These curves confirm that the sample made from thermally treated powder contains a higher amount of C-S-H rehydration products than the sample made from unheated powder. Thus, according to DTG curves, during heating, the WM500 sample lost up to 0.026% of its mass per 1 °C at temperatures between 100 °C and 400 °C due to dehydration of ettringite and calcium silicate hydrates. In contrast, the WMN sample only lost up to 0.019%/°C. This indicates that the thermally treated powder has slightly greater hydraulic activity.

To evaluate the effect of waste powders on cement hydration, microcalorimetry was performed on pastes containing 80% Portland cement and 20% waste powders. The resulting heat flow curves, normalized per gram of cement, are shown in Fig. 8 and indicate that the addition of waste powders enhances the cement hydration process, that can be attributed to their filler effect.

The incorporation of waste powders can influence the water demand

and setting time of the binder. Xu et al. [46] and Carriço et al. [53] identified specific factors of dehydrated cement that increase water demand, including specific surface area, porous morphology of dehydrated compounds, and high free lime content. The comparison of standard consistency of the pure Portland cement pastes with pastes containing 20% of investigated wastes showed an increase in water demand with the addition of waste powders, particularly in the case of MS and WM, where this increase is 7% and 11% for MSN and WM105 respectively. Additionally, all pastes containing waste treated at 400 °C showed a decrease in water demand, with a subsequent increase for waste treated at 500 °C. Since the materials do not contain free CaO and the fineness of waste powders is comparable to the used cement, this can be explained only by particles morphology and high reactivity of the C-S-H dehydration products.

The previous studies have reported that thermally activated recycled cement generally exhibits shorter setting times than ordinary Portland cement [46,53], which can have a negative effect on the fresh properties of the materials and their durability. However, Xu et al. [46] caution that this may be due to a false setting phenomenon resulting from the rapid reaction between free lime and water, leading to the formation of portlandite in the absence of gypsum.

However, the results of measuring the setting time of pastes with 20% thermally activated materials (Fig. 9) showed no acceleration of setting since the materials do not contain free lime and contain a small amount of gypsum. This also can be explained by the lower activation temperature as it was noted by Vysvaril et al. [61], who observed an increase in the setting time of pastes made of recycled cement dehydrated at 200–400 °C.

Although, as shown in Table 1, MS and WM contain more alkali, which act as set accelerators [62,64], than CS, results of setting time measurement shown on Fig. 9 indicates that pastes with untreated MS and WM powders have a significantly longer setting time compared to those with untreated CS. This difference diminishes as waste treatment temperature increases, and at 500 °C, the initial and final setting times of pastes containing different wastes differ by no more than 10 min. Therefore, since in the investigated wastes, alkalis are in an insoluble state in the composition of muscovite and glass particles, they do not affect the setting time.

In contrast, gypsum, present in MS and WM but not in CS, acts as a retarder by reacting with C3A to form ettringite [65–67]. This likely explains the longer setting time of pastes with untreated MS and WM powders. When heated, gypsum dehydrates, and at temperatures above 300 °C, insoluble  $\beta$ -anhydrite (natural anhydrite, anhydrite II) forms [68,69]. This compound, as evidenced by numerous studies [65–67], accelerates cement setting and explains the observed decrease in setting time of the pastes with MS and WM subjected to heat treatment.

### 5.3. Pozzolanic activity

In general terms, it is known that after mixing cement with water, in the first instance C-S-H gel and portlandite are formed as a result of hydration of C3S and C2S, and ettringite as a result of the reaction of C3A with gypsum [70]. Afterwards, portlandite reacting with amorphous silica and alumina, transforms into strong hydrated calcium silicates and aluminates, while ettringite, reacting with alumina, forms monosulfoaluminate of hydrated calcium [71]. Reactions occurring between SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and cement hydration products with the formation of strong compounds are called pozzolanic. The phases formed as a result of pozzolanic reactions reduce the average pore size in the binder matrix, thereby improving the mechanical properties and durability of concrete [71]. In addition, Fe<sub>2</sub>O<sub>3</sub> also reacts with portlandite to form hydrated C-F-H and C-F-S-H gel, as well as enhances the pozzolanic reactions [72].

Section 5.1 already pointed out that the materials under study cannot be classified as pozzolanic, as their SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> contents are insufficient. Nonetheless, MS and WM contain ceramic inclusions that



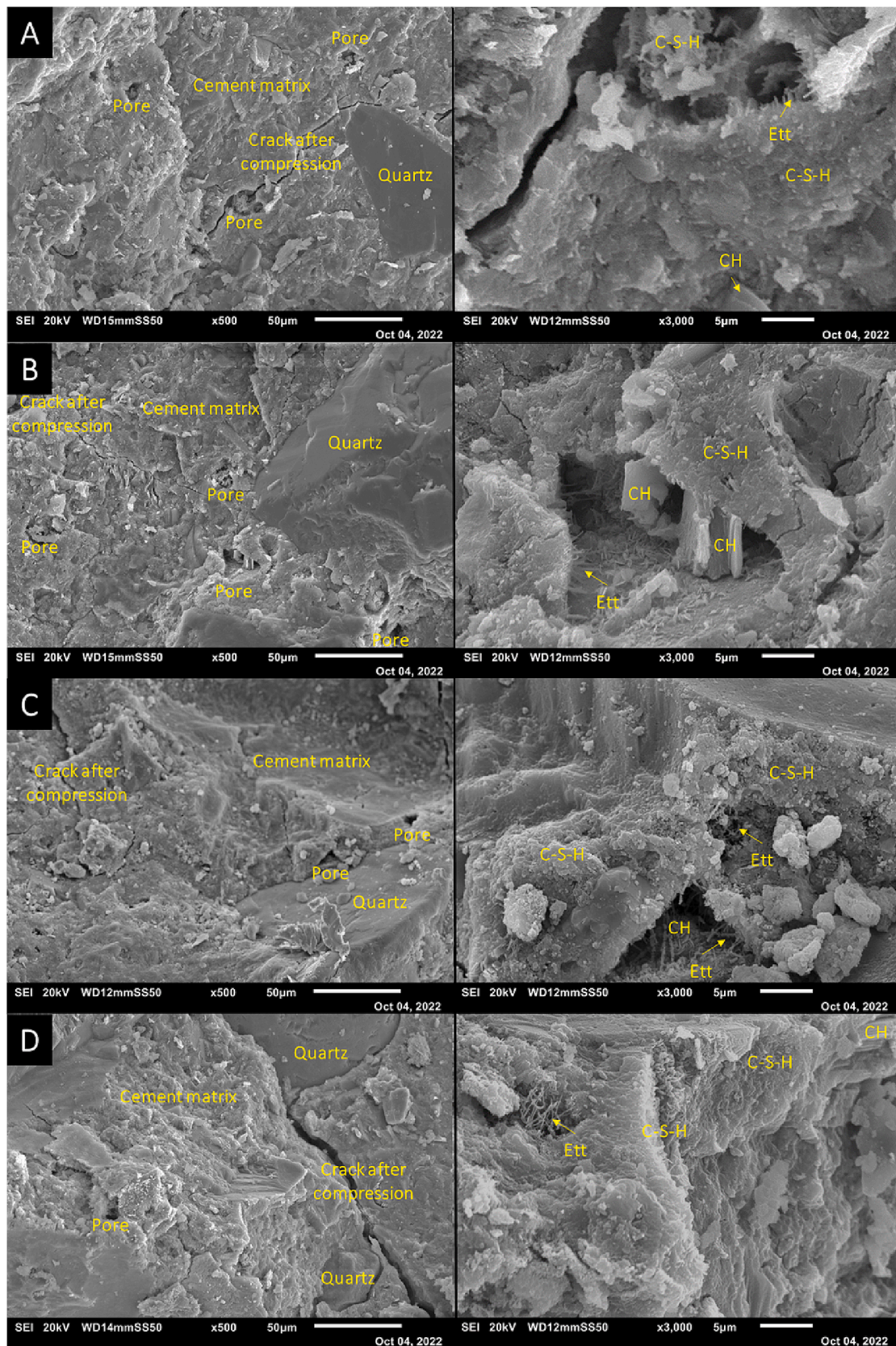


Fig. 14. SEM pictures of mortars with 20% of waste powder after 90 days curing and 1 year of storage: A – reference OPC mortar, B – CS500, C – MS500, D – WM500.

**Table 4**

Comparison of energy consumption and CO<sub>2</sub> emissions related to production of 1 kg ordinary Portland cement and 1 kg of blended binder with cement substitution rate of 20%.

Waste	Drying conditions	Calcination temperature, °C	Energy consumption, kJ/kg <sub>binder</sub>	Energy consumption related to reference, %	CO <sub>2</sub> emissions, kgCO <sub>2</sub> /kg <sub>binder</sub>	CO <sub>2</sub> emissions related to reference, %
CS; MS	Ambient	–	3224	–20.0	0.624	–20.0
		400	3251	–17.6	0.631	–19.1
		500	3272	–17.0	0.632	–19.0
	105 °C	–	3614	–8.3	0.656	–15.9
		400	3714	–5.8	0.663	–15.0
		500	3735	–5.3	0.664	–14.9
WM	Ambient	–	3224	–20.0	0.624	–20.0
		400	3251	–17.6	0.631	–19.1
		500	3272	–17.0	0.632	–19.0
	105 °C	–	4081	+3.5	0.688	–11.8
		400	4177	+5.9	0.695	–10.9
		500	4198	+6.4	0.696	–10.7
Ordinary Portland cement			3944		0.780	

**Table 5**

Comparison of changes in strength, energy consumption and CO<sub>2</sub> emissions depending on the SCMs treatment temperature. Percentages are relative to their respective uncalcined SCMs dried at natural condition.

Waste	Drying conditions	Calcination temperature, °C	Strength gain with thermal treatment of SCMs, %	Energy consumption increase with thermal treatment of SCMs, %	CO <sub>2</sub> emissions increase with thermal treatment of SCMs, %	Energy effectiveness index	CO <sub>2</sub> reduction effectiveness index
CS	Ambient	400	8.4	0.8	1.1	10.5	7.6
		500	13.2	1.5	1.3	8.8	10.2
	105 °C	–	6.3	12.1	5.1	0.5	1.2
		400	8.4	15.2	6.3	0.6	1.3
		500	13.2	15.8	6.4	0.8	2.1
MS	Ambient	400	10.4	0.8	1.1	13.0	9.5
		500	13.0	1.5	1.3	8.7	10
	105 °C	–	7.2	12.1	5.1	0.6	1.4
		400	10.4	15.2	6.3	0.7	1.7
		500	13.0	15.8	6.4	0.8	2.0
WM	Ambient	400	6.4	0.8	1.1	8.0	5.8
		500	9.0	1.5	1.3	6.0	6.9
	105 °C	–	1.4	26.6	10.3	0.1	0.1
		400	6.4	29.6	11.4	0.2	0.6
		500	9.0	30.2	11.5	0.3	0.8

may possess weak pozzolanic properties. Thus, by subjecting these materials to thermal activation, the ceramic particles can be separated from the hydrated cement adhering to them, increasing their availability for reactions with cement hydration products. Therefore, the goal of this section is not to demonstrate that demolition wastes are pozzolanic, but rather to distinguish between the effects on cement hydration of pure concrete waste and mixed waste containing ceramic and glass inclusions.

To evaluate the pozzolanic properties of the studied wastes after thermal treatment, the Chapelle test was conducted, with CS serving as a reference material without any pozzolanic activity (Fig. 10). The results demonstrate that, unlike CS, the reactivity of MS and WM gradually improves with increasing treatment temperature from 301 and 208 mg/g for MSN and WMN to 395 and 353 mg for MS500 and WM500 respectively. Furthermore, MS exhibits a slightly higher capacity for binding calcium hydroxide than WM. However, the reactivity of all investigated materials is not significant, as expected from their chemical composition, as the amount of Ca(OH)<sub>2</sub> fixed by 1 g of the material is lower than the threshold of 436 mg established by Raverdy et al. [73] for materials exhibiting pozzolanic activity.

The slightly improved activity of CS after calcination at 500 °C can be attributed to the transformation of chlorites, which were present in small amounts in the material, into an amorphous phase, as it was

showed by XRD analysis (Table 2 and Fig. 5). Additionally, a Chapelle test was conducted with pure magnetite or iron oxide (II, III) and hematite or iron oxide (III) to examine the influence of iron oxidation state on pozzolanic activity. The results indicate that magnetite only fixes 164.15 ± 21.72 mg of Ca(OH)<sub>2</sub>, while hematite binds 415.85 ± 9.48 mg of Ca(OH)<sub>2</sub>. Therefore, the increased activity of CS can also be attributed to the oxidation of less reactive compounds to more reactive ones in a redox system.

To investigate the influence of heat treatment on waste materials containing ceramic impurities on cement hydration, a TGA was performed on cement pastes containing 20% MSN and MS500 after a 90-day curing. The findings, depicted in Fig. 11, revealed that the quantity of portlandite in both samples was identical. Thus, according to the DTG curves, the weight loss in the temperature range of 400–600 °C caused by the dehydroxylation of portlandite was up to 0.03%/°C in both samples. These results imply that, at this substitution rate, the marginal increase in pozzolanicity caused by the heat treatment of the mixed waste, as indicated by the Chapelle test, had no impact on cement hydration.

#### 5.4. Compressive strength

Fig. 12 presents the compressive strength values of the tested mortar

**Table A1**

Comparison of available works on the use of dehydrated cement in the production of binders.

Source	Main objective of study	Experiments	Precursor	Target temperature, °C	OPC replacement, %	Main conclusion
[40]	To check, whether the hydration of the cement is reversible to have a possibility to identify the original cement used in a concrete and for the recycling of all concrete constituents.	XRD, extraction of free lime by ethyl acetoacetate, Differential Calorimetric Analyses, compressive strength.	Laboratory made cement paste.	600, 900, 1100, 1200, 1400	100	The reactions of clinker hydration are reversible completely at the cement burning temperature, the addition of gypsum form an additional intermediary phase. Portland cement heated at 1100 °C contains high amount of belite and free lime, while Portland cement with high sulfate resistance contains ye'elinite and blast furnace slag cement contains gehlenite.
[54]	Analysis of rehydration capability of concrete powder after thermal treatment.	TGA/DSC, XRD, SEM, compressive strength,	Laboratory made concrete.	200, 500, 800	100	Concrete powder heated at 500 °C consists of sand, dehydrated C–S–H, C2S, CaO, partial CH and dehydrated amorphous phase. Rehydration products have loose microstructure. Fly ash and OPC improves rehydration and mechanical properties of dehydrated cement.
[84]	Investigation of the cementitious characteristics and rehydration capability of dehydrated cement.	Standard consistency, setting time, compressive strength, LOI of dry rehydrated samples, SEM.	Laboratory made cement paste.	300, 400, 500, 600, 700, 800, 900	100	Rehydrated cement required more water for standard consistency and shows short setting time and rapid rehydration rate. These characteristics increase with dehydration temperature. The microstructure of the rehydrated cement is loose because of the fast formation of rehydration products occupying the internal space. The optimal dehydration temperature is 800 °C.
[85]	To study the rehydration of cement pastes with different initial water/binder ratios treated at different temperatures.	XRD, SEM, standard consistency, setting time, compressive strength.	Laboratory made cement paste.	300, 400, 500, 600, 700, 800, 900	100	The water demand increases, and the setting time decreases with increasing of dehydration temperature and initial w/b ratio. Strength development of the recycled cement is attributed to the rehydration of dehydrated phases and the hydration of the initially unhydrated particles and depends on the dehydration temperature and initial w/b ratio. The microstructure of the rehydrated paste is loose because of the fast formation of hydration products occupying the internal spaces.
[86]	To identify factors affecting the cementitious characteristics of the dehydrated cement.	Compressive strength, statistical analysis of 9 factors influence: fineness, w/b ratio, NaOH content, OPC content, dehydration temperature, Silica Fume content, Silica Fume content in the precursor, sand residues in the precursor and clay brick residues in the precursor.	Laboratory made cement paste.	700, 750, 800	100	The following factors have the biggest influence on the strength of rehydrated cement: the degree of hydration of the precursor, the presence of silica fume in the precursor, the fineness of the dehydrated waste, and the replacement of the dehydrated cement with silica fume have a positive effect, while the presence of sand in the precursor and the high temperature of dehydration have a negative effect.
[87]	To investigate the effect of dehydrated cement particle agglomeration on its	XRD, SEM, isothermal calorimetry, standard	Laboratory made cement paste.	650	5, 7.5, 10, 12.5, 15	Dehydrated cement has high water demand due to the water retention in the flaky layers of

(continued on next page)



Table A1 (continued)

Source	Main objective of study	Experiments	Precursor	Target temperature, °C	OPC replacement, %	Main conclusion
	rehydration and microstructure development.	consistency, setting time, compressive strength.				agglomerated particles. The optimal content of the dehydrated cement in the binder is 10%. Dispersed recycled cement creates additional C–S–H nucleation seeds.
[23]	To use recycled concrete powder received by a new method as Portland cement substitution.	TGA/DSC, XRD, SEM, isothermal calorimetry, flowability, flexural strength, compressive strength.	Laboratory made concrete.	500, 800	10, 20, 30	Dehydration products at 800 °C are C2S and lime. The rehydration occurs in the first hour. The dehydrated cement has higher water demand. Recycled cement can be used to replace up to 20% of the OPC. Dehydrated cement has an activation effect on fly ash and ground granulated blast furnace slag.
[63]	To investigate the rehydration process of the cement with different additives (blast furnace slag, fly ash and limestone).	XRD, standard consistency, setting time, mercury intrusion porosimetry, flexural strength, compressive strength.	Laboratory made cement paste.	200, 400, 600, 800, 1000, 1200	100	Slag and fly ash do not positively affect the strength after rehydration. Samples with higher content of brownmillerite (C4AF) and lower content of larnite (C2S) have higher strength. Limestone is more suitable additive for the rehydration process than slag and fly ash. Dehydration products are C2S polymorphs. Stability and reactivity of alpha-C2S formed at lower temperatures are higher due to smaller crystallite size. Pastes made with cement dehydrated at lower temperatures have higher strengths. Optimal dehydration temperature is 740 °C. Cooling rate had influence on the hydration at later ages.
[45]	To explain the effect of dehydration process parameters on the hydration reactivity of the recycled cement and on the strength of pastes made with it.	XRD, isothermal calorimetry, compressive strength.	Laboratory made cement paste.	699, 740, 800, 860, 901	100	Paste made of cement dehydrated at 450 °C had a similar strength as OPC paste but with a poor workability. Increasing the dehydrated cement fineness and addition of blast-furnace slag improve the workability without reducing the strength.
[51]	Checking the possibility to produce cement which similar physical properties as OPC from a 2-years old waste cement paste.	TGA, XRD, SEM, isothermal calorimetry, rheology measurement (shear stress, viscosity and dynamic yield stress), compressive strength.	Laboratory made cement paste.	120, 450, 750, 1150	100	Rehydrated paste showed higher efficiency than rehydrated concrete powder. Fineness of recycled cement is important. Incorporation of up to 20% of paste powder dehydrated at 650 °C does not affect the properties of mortar.
[52]	Investigation of fresh and mechanical properties of mortars containing dehydrated mortars and concrete, analysis of influence of replacement ratio, target temperature and fineness.	TGA, XRD, standard consistency, setting time, fresh density, flexural strength, compressive strength, ultrasonic pulse velocity, dynamic modulus of elasticity.	Laboratory made cement paste and concrete.	350, 650	20, 50, 75, 100	Dehydrated concrete contains non-hydrated cement, calcium hydroxide, calcium oxide and dicalcium silicate. The calcination temperature had more significant effect on the rehydration of cement than the calcination time.
[88]	To evaluate the effects of mechanical and thermal treatment of concrete rubble on the properties of fine fraction and its potential use as a supplement of Portland cement.	TGA/DTA, XRD, SEM, isothermal calorimetry, flexural strength, compressive strength, water absorbability.	Laboratory made concrete.	288, 350, 500, 650, 712	25	Dehydration product is new nesosilicate. Rehydration products at earliest age are AFm, carboaluminates, sulfoaluminates C–S–H, CH and ettringite. Paste of thermoactivated cement has high volume of hydration products and fine porosity.
[47]	Microstructure development at ages up to 28 days of paste made of rehydrated cement.	TGA, XRD, isothermal calorimetry, SEM, mercury intrusion porosimetry, nitrogen adsorption, fresh density, flexural strength, compressive strength.	Laboratory made cement paste.	700	100	

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Table A1 (continued)

Source	Main objective of study	Experiments	Precursor	Target temperature, °C	OPC replacement, %	Main conclusion
[50]	Better knowledge of the cement rehydration by studying of the fresh and hardened properties of mortars with dehydrated cement.	TGA, XRD, isothermal calorimetry, standard consistency, setting time, soundness, flowability, SEM, nuclear magnetic resonance spectroscopy, flexural strength, compressive strength.	Laboratory made cement paste.	400, 500, 600, 800, 900	20, 50, 100	The dehydration product formed at 600–800 °C is a new nesosilicate with a structure of the reactive polymorph $\alpha'$ -L-C2S. The rehydration products are C-S-H with dimer and longer silicate chains and carbonates. The optimal dehydration temperatures are 600–700 °C. The optimal substitution rate is up to 50%.
[48]	Better understanding of the rehydration and microstructure of thermoactivated recycled cement.	TGA, XRD, standard consistency, setting time, SEM, isothermal calorimetry, nuclear magnetic resonance spectroscopy, flowability, flexural strength, compressive strength, mercury intrusion porosimetry.	Laboratory made cement paste.	400, 450, 500, 600, 650, 700, 750, 800, 900	100	Dehydration of cement at temperatures above 600 °C forms C2S polymorphs. Rehydrated cement has high initial hydration heat, high water requirement and high setting time. The hydration products of thermoactivated cement and OPC are similar. The optimal dehydration temperatures are in the range 600–650 °C.
[89]	Studying of hydration activity of thermally activated waste concrete powders.	Differential scanning calorimetry, XRD, SEM, flexural strength, compressive strength.	Laboratory made concrete.	200, 400, 600, 700, 800	30	Thermal treatment decreases the particle size of waste concrete powder. Free calcium oxide was formed after treatment at 800 °C. Concrete powder treated at 700 °C can be used as cement substitution material.
[90]	To study the use of dehydrated cement in the production of ultra-high-performance concrete. Dehydrated cement is used to improve a compact packing structure of the concrete.	XRD, isothermal calorimetry, SEM, flowability, compressive strength, shrinkage, rapid chloride penetration test, mercury intrusion porosimetry, carbon footprint assessment.	Laboratory made cement paste.	650	12.5, 25, 37.5, 50	Dehydrated cement can be used in the production of ultra-high-performance concrete with very dense microstructure. The optimal substitution rate is 25%. Using dehydrated cement can effectively reduce the CO <sub>2</sub> emissions per unit volume of concrete and improve its performances.
[91]	Description and evaluation of the effectiveness of a new method for separation of hydrated cement and aggregates.	Qualitative image analysis, TGA, XRD, acid attack, flexural strength, compressive strength.	Laboratory made cement paste and concrete.	700	100	The method allows to obtain a recycled binder with cement content up to 80 wt% and recycled fine aggregates with cement contamination lower than 3 wt%. The mechanical strength of paste made of recycled binder was about 70% of that of OPC paste.
[92]	Characterization of the durability of concrete containing dehydrated cement.	Slump, fresh density, dry density, compressive strength, capillary absorption, oxygen permeability, rapid chloride migration, carbonation resistance tests.	Laboratory made cement paste and concrete.	650	5, 15, 30, 40, 100	Incorporation of up to 15% of the dehydrated paste or concrete resulted in similar durability to regular concrete. Using more than 30% of dehydrated concrete led to decreased durability compared to samples with recycled paste due to increased w/b. Concrete made of dehydrated cement has similar or better durability compared to regular concrete of the same strength class.
[93]	To evaluate the applicability of recycled cement powder for liquid radioactive waste immobilization.	XRD, compressive strength, leachability, freeze–thaw test.	Laboratory made concrete.	600	100	High water demand of dehydrated cement due to high surface area and low density. Dehydrated cement powder can be used for immobilization of liquid radioactive waste since the specimens with 3 M CoCl <sub>2</sub> and 3 M CsCl meet all the strength and leachability requirements.

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Table A1 (continued)

Source	Main objective of study	Experiments	Precursor	Target temperature, °C	OPC replacement, %	Main conclusion
[94]	To characterize the mechanical properties and shrinkage of concrete with various content of dehydrated cement and w/b ratio.	XRD, TGA, slump, fresh density, dry density, compressive strength, splitting tensile strength, ultrasonic pulse velocity, modulus of elasticity, shrinkage.	Laboratory made cement paste.	650	5, 15, 30, 40, 100	15% dehydrated cement can be added to concrete without significantly affecting workability. Dehydrated cement has high rehydration capacity. The optimum incorporation percentage is 15%, and concrete has similar or better characteristics than non-recycled concrete. The performance of recycled cement is improved by the addition of superplasticizer. Dehydrated cement also has a filler effect improving the concrete microstructure.
[44]	To study the co-utilization of dehydrated cement paste and coal gangue.	XRD, SEM, flowability, Fourier-transform infrared spectroscopy, compressive strength.	Laboratory made cement paste.	500, 600, 700, 800, 900	5, 10, 20, 30, 50 (mixed with coal gangue)	Dehydration products of cement paste are lime and $\beta$ -C2S. Mixing it with coal gangue also creates quartz and mayenite. Adding coal gangue improves grinding of recycled cement and binder flowability. The optimum substitution rate was 30%, and the optimum calcination temperature was 800 °C.
[49]	To analyse the rehydration behaviour and phase development of thermoactivated recycled cement pastes at early age.	TGA, XRD, isothermal calorimetry, SEM, nuclear magnetic resonance spectroscopy, flowability, flexural strength, compressive strength.	Laboratory made cement paste.	700	100	The fast early formation of AFm phases and carboaluminate hydrates during rehydration and high reactivity of $\alpha'$ -H-C2S formed in dehydrated cement were confirmed. The amount of hydratable products in dehydrated cement is lower because of excess of carbonates. In the rehydrated paste the same type of C-S-H as is the OPC paste is formed.
[95]	To analyze the effects of the simultaneous replacement of cement by dehydrated concrete fines and natural coarse aggregates by recycled aggregates on the mechanical properties of concrete.	Flexural strength, compressive strength.	Demolition concrete.	400, 650, 900	5, 10, 15	The most influent parameters on the strength are the amount of the recycled aggregates and the particle size of the dehydrated cement. The optimal combination of the parameters is: 20% of recycled aggregates, 5% of dehydrated at 900 °C cement with a size < 75 $\mu$ m.
[61]	To investigate the effect of blast furnace slag on the grinding process and mechanical properties of dehydrated cement paste.	Particle size distribution, particle hardness, semi-adiabatic calorimetry, compressive strength, SEM.	Demolition concrete.	600	100 (mixed with blast furnace slag)	Blast furnace slag enhanced grinding efficiency of dehydrated cement. Recycled cement has better hydration rate than OPC in the early stage, but worse hydration in the later stage. Rehydrated cement showed low strength due to low particle hardness. Slag provides a micro-aggregate framework supporting the cementitious system with high mechanical properties.
[96]	To study the effects of retarders on properties of pastes based on dehydrated cement.	Standard consistency, setting time, compressive strength, semi-adiabatic calorimetry, SEM.	Demolition concrete.	600	100 (mixed with blast furnace slag at cement to slag ratio of 2:1)	The optimal retarder to prolong setting of the rehydrated cement and improve paste microstructure and strength is borax in an amount of 2%. Borax reacts with Ca <sup>2+</sup> and aluminate in dehydrated phase forming a layer, which prevents water penetration into the cement particles.

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Table A1 (continued)

Source	Main objective of study	Experiments	Precursor	Target temperature, °C	OPC replacement, %	Main conclusion
[56]	To analyze the effects of dehydrated cement and recycled sand on the properties of cement mortar.	XRD, SEM, TGA/DSC, setting time, flowability, shrinkage, total porosity of mortars heated at 200 and 500 °C, flexural and compressive strength of mortars heated at 200 and 500 °C.	Demolition waste of unknown origin.	500	5, 10, 15, 20, 30, 50	The finer fraction of recycled concrete contains more reactive components than the coarser one. The addition of 10% of dehydrated cement does not have a significant effect on the workability. The optimal contents of the recycled sand and dehydrated cement for the mechanical properties of mortars are 20% and 15% respectively. Dehydrated cement increases the total porosity that decreases the heat resistance of the mortar.
Present research	To investigate the potential of fine mixed concrete-ceramic waste and mud from the recycled aggregates washing for use as SCMs and understand their behavior after thermal treatment.	DTA/TGA, XRD, isothermal calorimetry, standard consistency, setting time, Chapelle test, compressive strength, SEM.	Concrete screening fines, mixed screening fines, mud from recycled aggregates washing.	450, 500	20	Uncalcined MS and WM, containing gypsum, increase setting time. Mortars with 20% of all three waste types had similar mechanical properties. Thermal treatment of waste slightly improves mortar's mechanical properties. Dehydrated at 500 °C mixed wastes have weak hydraulic activity, but no pozzolanic activity.

and reference Portland cement mortar specimens. All investigated materials exhibited similar mechanical properties development. Thus, the strength of the mortars after 90 days of curing was  $52.9 \pm 0.9$ ,  $51.3 \pm 0.4$ , and  $52.6 \pm 0.9$  for mortars containing CSN, MSN, and WMN, respectively, and  $59.5 \pm 0.8$ ,  $57.9 \pm 0.5$ , and  $56.4 \pm 0.5$  for mortars containing CS500, MS500, and WM500, respectively. The strength of mortars containing CS and the other two materials did not show a significant difference, indicating that the presence of ceramic and glass impurities in waste powders does not affect the mechanical properties of the mortars at the substitution rate of 20%.

All samples met the European standard EN 197-1 for blended cement, with a minimum strength of 32.5 MPa and an initial setting time threshold of  $\geq 75$  min. Additionally, they even met the strength class of 42.5 MPa, demonstrating that all materials can be used as SCMs for blended cement production. The waste powders contributed to the strength of the mortars due to the filler effect, and in the case of heat-treated materials, due to the combined action of the filler effect and rehydration of the products of partial C-S-H dehydration.

Fig. 13 shows the SAI of the mortar specimens. There is no single standard that defines the threshold, so the researchers usually use 75% as the threshold, as defined by the ASTM C311 standard [60] for fly ash and natural pozzolans. All investigated materials met this threshold, despite not being pozzolans. The SAI values of the studied materials were consistent with data obtained by other researchers for the substitution rate of 20% by recycled cement (RC), limestone filler (LF), and quartz filler (QF), which are given in Table 3. Thus, after 7 days of curing, the ranges of SAI values were 75–77% for unheated materials and 81–82% for materials treated at 500 °C. For RC, LF, and QF, the SAI values were  $77 \pm 11\%$ ,  $85 \pm 6\%$ , and  $83 \pm 12\%$ , respectively. After 28 days of curing, the SAI values were 77–78% for unheated materials, 85–89% for materials calcined at 500 °C, and  $72 \pm 6\%$ ,  $81 \pm 6\%$ , and  $74 \pm 6\%$  for RC, LF, and QF, respectively. Finally, after 90 days of curing, the SAI values were 78–80% for unheated materials, 86–90% for materials calcined at 500 °C, and  $72 \pm 8\%$ ,  $83 \pm 1\%$ , and  $79 \pm 4\%$  for RC, LF, and QF, respectively.

### 5.5. Microstructure of mortar samples

To investigate the effect of waste powders on the microstructure development of cementitious mortars, scanning electron microscopy (SEM) analysis was employed. Fig. 14 shows the SEM micrographs of the reference mortar and mortars containing 20% waste materials calcined at 500 °C after curing for 90 days and storage for approximately 1 year. EDX microanalysis of the mineralogical phases is given in the appendix to this article.

Observations of the microstructure of cement mortars confirmed that the waste powders were embedded perfectly in the cement matrix, forming a compact microstructure. There were no significant differences in the microstructure of the four specimens, except that the microstructure of the mortars with waste powders appeared slightly looser than that of the reference mortar, which may explain the strength loss. However, the pictures show that the pores in the cement matrix with waste powders are filling with hydration products, which explains the growth of SAI with age.

### 5.6. Rationality of SCMs thermal treatment

To assess the rationality of thermal treatment of SCMs, as well as to compare the two drying methods from the economic and environmental aspects, approximate calculations of specific energy consumption and specific CO<sub>2</sub> emissions were made for the production of 1 kg of binder with a substitution rate of Portland cement of 20%. For the calculation, the following literature-based data valid for European Union countries were used:

- Energy demand for heating up to 1450 °C: 1715 kJ per 1 kg of Portland cement clinker [80];
- Energy demand for drying of raw materials with moisture content of 10%: 2315 kJ per 1 kg of Portland cement clinker [80];
- CO<sub>2</sub> emissions related to limestone decarbonation: 0.53 kg CO<sub>2</sub> per 1 kg of Portland cement clinker [81];

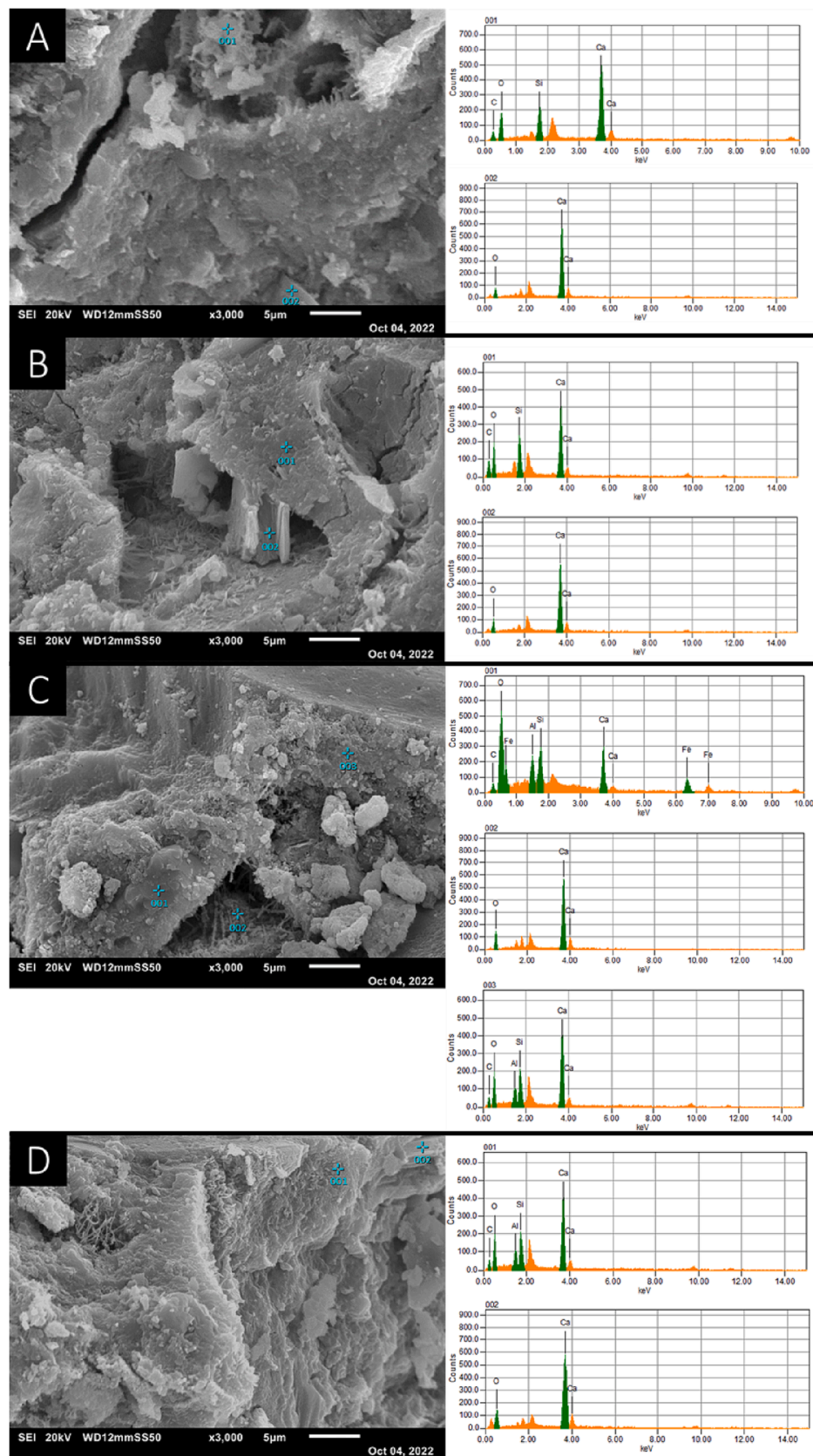


Fig. B1. EDX microanalysis of the mineralogical phases identified by SEM: A – reference OPC mortar, B – CS500, C – MS500, D – WM500.



- 65% of CO<sub>2</sub> emitted during cement production is associated with limestone decarbonation and 35% of CO<sub>2</sub> associated with fuel combustion [82,83];
- 100% fossil fuel was taken as energy source.

The composition of Portland cement was considered as 95% clinker and 5% gypsum, the moisture content in CS and MS was assumed as 10%, and the moisture content in WM was 20%. Energy consumption for crushing, grinding and sieving was not taken into account. The calculation results are given in Table 4.

As it can be seen from Table 4, the drying of raw materials at elevated temperatures is the most energy-intensive process in the production of cement components. Moreover, the production of blended cement using WM dried by heating requires more energy than the production of ordinary Portland cement due to the high moisture content in the mud. In the case of ambient drying the energy consumption is significantly reduced.

Considering CO<sub>2</sub> emissions, all studied blended cements have an advantage over ordinary Portland cement, and the drying of raw materials without heating greatly enhances this advantage.

To assess the rationality of calcination the wastes at temperatures of 400 °C and 500 °C, the increase in energy consumption and CO<sub>2</sub> emissions was compared with the increase in strength of specimens containing 20% thermally treated SCMs after 28 days of curing relative to specimens containing unheated SCMs. The comparison results, given in Table 5, show that drying CS and MS at elevated temperatures improves the strength of the samples, but at the same time it is associated with an important increase in energy consumption and CO<sub>2</sub> emissions. Calcination of the wastes at 500 °C significantly improves the strength of samples containing SCMs, while the increase in energy consumption and CO<sub>2</sub> emissions is not much higher than for specimens with uncalcined wastes. In addition, the differences between the strengths of samples containing SCMs treated at 400 °C and at 500 °C are more pronounced than the differences in energy consumptions and CO<sub>2</sub> emissions for the same samples, which suggests that calcining the waste at 500 °C is more appropriate.

The energy effectiveness indices, which are the ratio of the strength gain with the associated energy consumption increase, as well as the CO<sub>2</sub> reduction effectiveness indices, which are the ratio of the strength gain with the associated CO<sub>2</sub> emission increase, both illustrate the advantage of drying at natural condition rather than at 105 °C. While it is obvious that natural drying has a clear environmental advantage over drying by heating, this method is much more time consuming and requires a lot of space, which is difficult to carry out on an industrial scale. Perhaps this problem could be solved by the use of mechanical drying methods such as centrifugal drying, or changes to the storage method for fine demolition wastes to reduce their moisture content. Calcining materials at relatively low temperatures up to 500 °C contributes significantly less to energy consumption and CO<sub>2</sub> emissions than drying them by heating for a long time.

## 6. Conclusions

This study provides an assessment of the potential use of real demolition waste, including mixed concrete-ceramic screening fines and mud from recycled aggregate washing, in eco-cement production. The following conclusions are drawn based on the research:

- (1) The XRD and DTA/TGA analysis of the investigated materials showed that all three samples are mainly composed of quartz and calcite. Small amounts of gypsum were observed in MS and WM, accounting for 4.2% and 2.1% of the respective samples crystalline phase weights. No portlandite was detected in any of the investigated materials due to its transformation into calcite over time as a result of carbonation processes.

- (2) The investigated waste materials showed only an initial heat release during the first 2 h of hydration. The addition of waste powders improved cement hydration due to their filler effect, but also increased water demand by 2% to 11% depending on the type of waste and treatment temperature. CS and calcined MS and WM had no significant impact on setting time, whereas uncalcined MS and WM caused retardation due to the presence of gypsum.
- (3) The reactivity of MS and WM gradually improves with increasing treatment temperature, but remains low even at the highest treatment temperature. The amount of Ca(OH)<sub>2</sub> fixed by 1 g of the material is lower than the threshold for materials exhibiting pozzolanic activity, ranging from 208 mg/g to 353 mg/g for unheated WM and WM calcined at 500 °C, and from 301 mg/g to 395 mg/g for unheated MS and MS calcined at 500 °C. The ceramic inclusions in the waste powders do not appear to affect the strength of mortars when the substitution rate is 20%.
- (4) The mortars with 20% of all three waste types had similar mechanical properties and is at least 37.93 MPa, 46.25 MPa and 51.33 MPa after 7, 28 and 90 days of curing, respectively. Thermal treatment of wastes improves the mechanical properties of the mortars, and increases the SAI after 90 days of curing at 10% for the mortars with CS and MS and at 6% for the mortars with WM. The contribution of waste powders to the strength of mortars is explained by their filler effect and the presence of partially dehydrated C-S-H products in the calcined materials.
- (5) By using the CDW fines investigated as SCMs to replace 20% of Portland cement, CO<sub>2</sub> emissions can be reduced by 10.7 to 20%. However, the process of drying wash mud by heating leads to higher energy consumption compared to the production of ordinary Portland cement due to the material's high moisture content. Therefore, alternative drying methods should be considered for this material.

The study showed that real demolition waste, despite containing high levels of inactive components like quartz, can be utilized to produce eco-efficient blended cement. However, it's crucial to assess the long-term properties and durability of building materials that contain these wastes when exposed to environmental factors. In addition, one of the main challenges of utilizing demolition waste in producing new building materials is uncertainty about the stability of their composition. Thus, conducting systematic studies comparing the properties and composition of different demolition waste, over time and across various locations, is essential. To carry out such studies, a comprehensive database is necessary, and continued research on demolition waste is required to expand and update this database.

## CRedit authorship contribution statement

**Anna Tokareva:** Conceptualization, Methodology, Validation, Formal analysis, Investigation, Resources, Writing – original draft, Visualization. **Sinan Kaassamani:** Conceptualization, Methodology, Validation, Resources, Writing – review & editing. **Danièle Waldmann:** Conceptualization, Methodology, Validation, Resources, Writing – review & editing, Supervision, Project administration, Funding acquisition.

## 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

The authors do not have permission to share data.

## Acknowledgments

We sincerely thank Mr. Marc Seil, Mr. Ed Weyer, Mr. Gilbert Klein and Mr. Mehdi Saeidi from the Solid Constructions Laboratory at the University of Luxembourg, Mr. Markus Schlien from the Electrochemistry Laboratory of the University of Luxembourg, and the team of the Laboratory of Ponts et Chaussée (Luxembourg) for their technical support and training. We are also very grateful to Mr. Robin Chapelle and Mr. Nicolas-Alexandre Eugene from Tradecowall company (Belgium) for providing the researched materials, Dr. Claude Simon and Mr. Sven Jung from Cimalux company (Luxembourg) for providing cement and technical assistance in laser granulometry analysis, Dr. Baptiste Luzu from the University of Lorraine for technical assistance with microcalorimetry, Dr. Neven Ukrainchuk from the Technical University of Darmstadt for technical assistance with XRF, XRD and TGA/DTA analyses, and Mrs. Zornitza Tosheva from the Department of Physics and Materials Science at the University of Luxembourg for the training of the SEM use.

## Appendix A

## Appendix B

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