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Thermal Properties of High-Performance Concrete Containing Fine-Ground Ceramics as a Partial Cement Replacement

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Some types of industrial waste can be used in concrete as a partial replacement of a cement binder. One such material is fine-ground ceramics, which is a waste produced during brick cutting. The ground ceramic can be used as a pozzolana active material which can improve final properties of concrete. This fine powder was used in this study as a partial replacement of the cement binder up to 60 mass % and its thermal and mechanical properties were studied using the differential scanning calorimetry, thermogravimetry, and thermodilatometry. It was shown that the differential scanning calorimetry is a suitable method for observing thermal changes in concrete samples containing such additives at the microstructural level. In particular, it allows one to investigate the hydration and pozzolanic reaction in the studied concrete. The investigation was performed in the temperature range from 25 °C to 1000 °C.

Keywords: concrete; ground ceramic; differential scanning calorimetry; thermogravimetry; thermal expansion.

1. INTRODUCTION

The properties of concrete containing pozzolanic materials have been studied over the last few decades. Pozzolanic materials can partially replace the cement binder and improve properties of the final concrete product as well as reduce the production costs. These materials can be natural or of an industrial origin [1]. Very popular are industrial byproducts (fly ash or silica fume), metallurgical slags (blast-furnace slag), and natural pozzolans (metakaolin) [2]. The use of waste materials in concrete has obvious benefits in environmental sustainability due to reducing the amount of waste and/or energy in industrial factories.

One of the frequently used pozzolanic materials is ceramic that occurs as waste in the ceramic industry (bricks, roof tiles, electrical insulators or sanitary ware). It can be used as coarse [3 – 7] or fine [8 – 10] aggregates (or their combination [11, 12]) or as pozzolanic admixtures in mortar and concrete [13 – 17].

For coarse aggregate replacement it was determined that its optimal amount is about 10 – 25 mass % [4, 6]. Moreover, it was found that the recycled ceramic aggregate does not interfere in a negative way during the hydration process [5, 6]. It was also found that the microstructure in the interfacial transition zone between recycled ceramic aggregate and paste was more compact than in the case of natural aggregate and paste. The main problem of using ceramic coarse aggregates as substitute of natural aggregates is in its higher water absorption. This shortage can be partially resolved by using a pre-saturation method of the aggregates [3]. On the other hand, the ceramic coarse aggregate has a large abrasion resistance, and so it is more resistant to temperature change than natural coarse aggregate [7].

The use of ceramic waste as fine aggregates was also studied [8 – 10]. The results were similar to the results of ceramic waste coarse aggregates (in terms of workability, durability, abrasion resistance, and water absorption). The replacement of natural sand with fine recycled aggregate from ceramic waste up to 40 mass % did not significantly affect the properties of fresh and hardened low strength mortar, with the exception of density and workability [10].

Finally, the use of ceramic waste as cement replacement was also studied [13 – 17]. The ceramic waste was considered as pozzolanic active material which can participate in cement hydration. For example, it was shown that the waste tiles have pozzolanic properties of the cement and tile conforms to cement standard up to the addition of 35 mass % [13]. In addition, powdered roof tiles were found to be suitable for partial cement replacement (up to 20 – 30 mass %) without a detrimental effect on the strength [14]. As far as the hydration heat development is concerned, blended cements containing fine-ground ceramics as Portland cement replacement were analyzed [17]. It was concluded that the ceramics slightly retards the C_3S hydration process and that above 20 mass % of fine-ground ceramics a substantial part of ceramics does not participate in the hydration process and plays a role of fine filler instead.

In the present paper the basic physical, thermophysical, and mechanical properties of high-performance concrete are studied in dependence on the amount of ceramic waste. We prepared seven different mixtures: a reference concrete without ceramic powder and samples with 10, 20 … 60 mass % replacement of cement binder. The used ceramic waste powder was from the production of bricks.

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2. MATERIALS AND SAMPLES

Samples of high-performance concrete were prepared from Portland cement (CEM I 52.5 R, according to the EN 197-1 standards), finely ground ceramic waste (from HELUZ cihlářský průmysl v.o.s., Hevlín, Czech Republic), natural quartz, basaltic sand (its continuous granulometry was 0 – 4 mm), silica fume, and superplasticizer. The water/cement ratio was chosen very low to achieve a low porosity.

Table 1. Chemical composition of ceramic waste powder

	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	L.O.I.
mass %	50.73	20.26	10.92	6.36	4.75	6.98

The composition of the fine ceramic powder is given in Table 1. This material was used as a partial replacement for the cement binder in the amount 10, 20 … 60 mass %. Thus, six types of samples with a varying amount of cement replacement were prepared to study the effect of the finely ground ceramic waste powder on thermal and mechanical properties of high-performance concrete. The reference material without cement replacement (denoted here as BR3) was also prepared. The composition of the studied materials is given in Table 2. More details on the preparation can be found in [18].

3. EXPERIMENTAL METHODS

The basic physical material characteristics – the bulk density ρ_b ($\mathrm{kg\cdot m^{-3}}$), open porosity (%), and matrix density ρ_{mat} ($\mathrm{kg\cdot m^{-3}}$) – were determined using the water vacuum saturation method (see [19] for details), before and also after heating (from 25 to 1000 °C with a heating rate 1 °C $\mathrm{min^{-1}}$). The size of samples was 50 × 50 × 25 mm.

The flexural and compressive strength were tested according to the standard ČSN EN 196-1 [20]. A tested sample with the dimension of 40 × 40 × 160 mm was placed in the testing apparatus on the supporting rods which were 100 mm from each other. The loading force was applied vertically in the middle of the sample with the rate of $50 \pm 10\ \mathrm{N\,s^{-1}}$ until the sample fractured. Then the halves obtained from the test of the flexural strength were tested for the compressive strength (with an increasing loading force with the rate of $2\,400 \pm 200\ \mathrm{N\,s^{-1}}$ until a fracture occurred).

For the study of the hydration and pozzolanic reaction in the high-performance concrete containing the finely ground ceramic waste we used the differential scanning calorimetry (DSC) and thermogravimetry (TG). For the DSC measurements we prepared powder samples which were placed into an alumina crucible with a volume of 100 $\mathrm{mm^3}$. All experiments were carried out in the temperature range from 25 to 1000 °C with a heating rate 5 °C $\mathrm{min^{-1}}$ in an argon atmosphere and were repeated three times for each sample type. As blank curves, we used the data from a second measurement of the same material under identical conditions. Therefore, any reversible processes that take place in the material cannot be registered.

In the thermodilatometry the length changes of a studied material are measured as a function of temperature or time during a negligible loading. Our samples had the dimensions 15 × 15 × 160 mm and were exposed to a controlled temperature program (from 25 to 1000 °C with a heating rate 1 °C $\mathrm{min^{-1}}$) in a static air atmosphere [18].

4. RESULTS AND DISCUSSION

In Table 3 the results of the basic physical characteristics before and after heating are shown. The bulk density before heating decreased with the increasing amount of ceramics in the samples. The difference between the reference sample (BR3) and sample BC3-60 was about 2 %, and the difference between samples BC3-10 and BC3-60 was about 3 %. The same behavior and differences were observed in the matrix density. The open porosity had variable and relatively low values.

The results after heating showed that the bulk density, matrix density, and open porosity had similar behavior as before heating.

Table 2. Composition of studied high-performance concrete (in $\mathrm{kg\cdot m^{-3}}$)

Material	CEM I 52.5 R	Ceramic waste	Silica powder	Natural quartz and basalt sand				Silica fume	Superplasticizer	Water
				0.1 – 0.6	0.3 – 0.8	0.6 – 1.2	1.0 – 4.0			
BR3	650	0	148	440	281	219	159	148	30	170
BC3-10	585	65								
BC3-20	520	130								
BC3-30	455	195								
BC3-40	390	260								
BC3-50	325	325								
BC3-60	260	390								

Table 3. Basic physical characteristics before and after heating

Material	Before heating			After heating		
	Bulk density ($\mathrm{kg\cdot m^{-3}}$)	Matrix density ($\mathrm{kg\cdot m^{-3}}$)	Open porosity (%)	Bulk density ($\mathrm{kg\cdot m^{-3}}$)	Matrix density ($\mathrm{kg\cdot m^{-3}}$)	Open porosity (%)
BR3	2 192	2 302	4.8	2 101	2 592	18.9
BC3-10	2 202	2 318	5.0	2 101	2 595	19.1
BC3-20	2 204	2 304	4.3	2 108	2 609	19.2
BC3-30	2 224	2 308	3.7	2 116	2 588	18.2
BC3-40	2 140	2 254	5.1	2 074	2 583	19.7
BC3-50	2 147	2 269	5.4	2 057	2 562	19.7
BC3-60	2 145	2 265	5.3	2 073	2 594	20.1

The values of the bulk density were lower, however, while the matrix density and open porosity had higher values. The open porosity increased almost four times.

The mechanical properties of the studied materials are given in Table 4. The compressive and flexural strength were measured after 28 days. Concrete BC3-20 had the highest value (132.50 MPa), whereas the lowest value (103.81 MPa) was observed for the concrete containing 60 mass % of the ceramic powder. These results showed that the ceramic powder was applicable up to 20 mass % as a cement replacement and above this amount a substantial part of the ceramic powder did not participate in the hydration process, and acted only as a fine aggregate [17]. It was interesting that the results of the flexural strength were variable and exhibited no obvious trends.

Table 4. Mechanical properties of studied materials

Material	Compressive strength (MPa)	Flexural strength (MPa)
BR3	117.18	12.41
BC3-10	127.65	14.30
BC3-20	132.50	16.37
BC3-30	122.64	14.35
BC3-40	117.23	15.52
BC3-50	114.69	18.30
BC3-60	103.81	15.58

The heat flow and relative mass change at a given temperature are shown in Fig. 1 and Fig. 2 for all studied samples. In Fig. 1 several heat flow peaks occur. The size of these peaks varies with the amount of the ceramic powder.

The heat flow peaks in the temperature interval from 25 to 200 °C correspond to the liberation of physically bound water from pores and also to the dehydration reactions due to the loss of water from calcium silicate hydrates [21, 22]. Usually, two endothermal peaks are present in this temperature interval. One is dehydration of C-S-H gel and the other corresponds to the ettringite decomposition, which usually arises in concrete. Its formation is given by molar ratio of CSH_2/C_3A in concrete [23]. In [21] the dehydration of C-S-H gel and ettringite was observed close to 110 °C, but in [22] it was between 140 – 160 °C. This was probably caused by different ages of the studied concrete, formed hydration products, chosen temperature program, or different sample preparation procedures before measurement. In our case the amount of arising ettringite in concretes was probably too low, because it was not observed on the DSC curves. It is due to the age of samples (about 200 days). A higher amount of the ceramic powder generated a larger peak in this temperature range, which can be caused by the changes in the hydration reaction due to the presence of ceramic powder in the blended binder. The peak positions were between 116 °C (BC3-50) and 123 °C (BR3 and BC3-40). The reactions in this temperature interval are also associated with a significant mass loss (see Fig. 2). For the reference sample it is about 1.9 % and for sample BC3-20 it is about 2.1 %. The mass loss decreases with an increasing amount of the ceramic powder, and it is about 1.4 % for sample BC3-60.

The next peak is in the temperature interval from 390 to 450 °C. This temperature interval is associated with the decomposition of portlandite $Ca(OH)_2$ [24 – 26]. The peak moves to higher temperatures with an increasing amount of the ceramic powder. For sample BC3-40 the peak is at about 449 °C. The decomposition is not observed once the ceramic powder replacement has 50 or 60 mass %. The decomposition of portlandite is also accompanied by a mass loss. This effect is the most visible for reference concrete for which it is about 0.32 %. With an increasing amount of the ceramic powder in concrete the mass loss decreases until it completely vanishes.

The next relevant temperature point for our studied material is 573 °C when the α→β transformation of quartz takes place. This reaction is accompanied by a sharp endothermic heat flow peak and a volume change (about + 0.68 %) [27]. However, this reaction should not be visible in Fig. 1 because it is reversible (and thus cannot be detected in the DSC measurements, as is explained in the Experimental methods section). The small peaks visible in Fig. 1 around 573 °C are attributed to measurement errors. During the quartz transformation there is no change of mass.

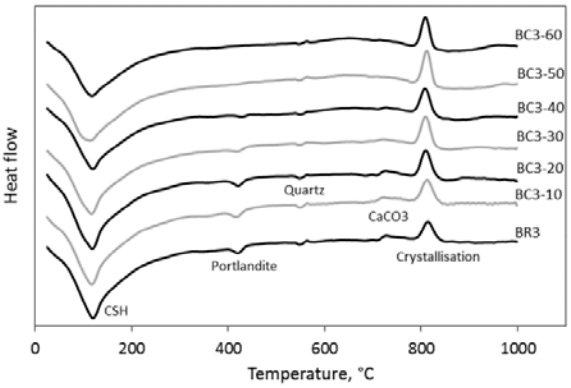

Fig. 1. The DSC thermograms of the high-performance concrete containing 0 – 60 mass % of the ceramic waste powder replacement

The $CaCO_3$ decomposition is initiated in the temperature interval 722 – 731 °C [21]. It is observed for materials with up to 40 mass % of the ceramic powder, while for samples with 50 and 60 mass % it disappears. Note that the reaction is accompanied by about two times larger mass loss than in the case of the $Ca(OH)_2$ decomposition. This is an apparent consequence of the relatively high age of the samples.

At temperatures above 800 °C, there is one significant exothermal peak corresponding to a crystallization process [21, 24]. In [21] a similar peak was obtained, but it was caused by the transformation of metakaolin to spinel. In our case, however, the peak is caused by the pozzolanic reaction between Portland cement and silica fume, and its size corresponds to the degree of crystallization between Ca and Si [24]. It was observed that it is growing with the curing age. In our study the peak corresponding to crystallization is located at a slightly lower temperature, which could be caused by the age of samples, complexity of composition, or a different procedure of concrete preparation.

The enthalpy of reaction occurring in the studied concrete was determined from DSC peaks (see Fig. 1).

Table 5. The enthalpy results for the corresponding peaks in the high-performance concrete containing various amount of the ceramic powder

Material	C-S-H decomposition		Portlandite decomposition		Calcite decomposition		Crystallization	
	Temperature	Enthalpy	Temperature	Enthalpy	Temperature	Enthalpy	Temperature	Enthalpy
	°C	$J \cdot g^{-1}$	°C	$J \cdot g^{-1}$	°C	$J \cdot g^{-1}$	°C	$J \cdot g^{-1}$
BR3	123.08	143.71	440.22	2.70	729.16	6.73	834.76	-9.70
BC3-10	120.58	138.26	437.04	2.91	726.71	6.77	833.24	-12.28
BC3-20	121.22	153.19	441.12	2.75	730.31	5.22	831.03	-13.78
BC3-30	119.87	133.68	439.44	1.38	721.94	3.79	830.66	-13.69
BC3-40	122.53	130.92	448.88	0.71	731.27	1.15	828.99	-12.70
BC3-50	115.96	130.35	–	–	–	–	832.87	-14.19
BC3-60	129.79	122.51	–	–	–	–	829.72	-12.47

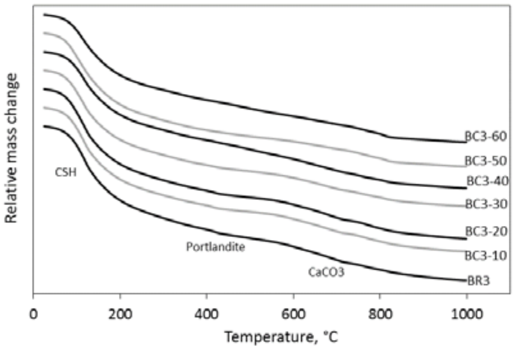

Fig. 2. The relative mass change of the high-performance concrete containing 0 – 60 mass % of the ceramic waste powder replacement

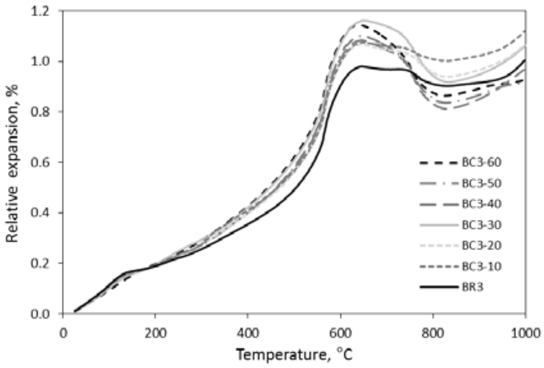

Fig. 3. The relative expansion of the high-performance concrete containing 0 – 60 mass of the ceramic waste powder replacement

The results are summarized in Table 5. They show that the temperature and enthalpy of reactions are changing with an increasing amount of the ceramic powder. The first studied reaction was associated with the C-S-H dehydration. The temperature of the peak increases and the enthalpy of this reaction decreases with amount of the ceramic powder in studied samples. The enthalpy of sample BR3 is 143.71 J g^{-1} and that of sample BC3-60 is 122.51 J g^{-1}. The second studied reaction was associated with the portlandite decomposition. The temperature and also enthalpy have the same trend as it was for the C-S-H dehydration. The enthalpy of sample BR3 is 2.70 J g^{-1} and for sample BC3-40 it is 0.71 J g^{-1}. If the amount of the ceramic powder is the ceramic powder is more than 40 mass%, the peak vanishes. Finally, we investigated the reaction associated with the crystallization process. It was observed that the temperature of the process decreases and the corresponding enthalpy increases with the amount of the ceramic powder in samples.

The length changes of high-performance concrete during heating up to 1000 °C were also studied (see Fig. 3). Clearly, there are many differences in these results between the samples with different amounts of the ceramic powder. This behavior may be described within several intervals.

The first interval is up to about 170 °C. This limit is probably given by water evaporation and C-S-H dehydration (the samples were not dried before measurement). These effects caused a slight delay in the expansion of samples. The second interval is from 200 °C to 573 °C. BC3 samples have a higher relative expansion its modification from α to β.

This material change is accompanied by a thermal expansion which becomes larger with a higher amount of the ceramic powder (for BR3 it is 0.8 %, while for BC3-60 it is 0.9 %). Above the temperature 573 °C, there are many differences in the relative expansion between the studied materials. In fact, the materials with a higher amount of the ceramic powder have a higher relative expansion. Above temperature 650 °C the expansion decreases for all studied materials. The shrinkage is higher with the increasing amount of the ceramic powder in concrete. Finally, above the temperature 830 °C the relative expansion again increases for all materials. This trend was observed up to 1000 °C.

5. CONCLUSIONS

The properties of high-performance concrete containing 0 – 60 mass % of ceramic waste powder were studied. Experimental results showed that ceramic waste powder can be successfully used as a supplementary material of cement binder in the production of high-performance concrete. The main findings can be summarized as follows: 1) The bulk and matrix density decreased with the increasing amount of ceramic powder. The open porosity had variable values that were relatively low before heating for all materials. The trend of increasing porosity probably affected the mechanical properties; 2) The basic physical characteristics were changed after heating. The bulk density decreased with an increasing amount of the ceramic powder, while the matrix density and open porosity were increasing. The open porosity increased almost four times; 3) The mechanical properties confirmed that the ceramic replacement of cement binder works until 20 mass % and above this value it behaves only as a fine aggregate. With this amount of ceramic powder, a high-performance concrete with the compressive strength above 130 MPa can produced. The flexural strength increased with an increasing amount of the ceramic powder; 4) Thermal properties showed changes in the chemical composition of the studied materials at high temperatures. The temperature and enthalpy of the C-S-H dehydration, Portlandite decomposition, the α→β transformation of quartz, and the calcite decomposition were distinguished and described. The last modification at temperature about 830 °C was attributed to a crystallization process which corresponded to the reaction between Ca and Si during ageing of the composite.

It was shown that it is possible to design and produce high-performance concrete containing fine ceramic waste powder. An appropriate amount of the ceramics replacement is about 20 mass %.

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