Effect of Fine-Ground Ceramic Admixture on Early Age Properties of Cement Paste

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Abstract—Properties of cement pastes with fine-ground ceramics used as an alternative binder replacing Portland cement up to 20% of its mass are investigated. At first, the particle size distribution of cement and fine-ground ceramics is measured using laser analyser. Then, the material properties are studied in the early hardening period up to 28 days. The hydration process of studied materials is monitored by electrical conductivity measurement using TDR sensors. The changes of materials' structures within the hardening are observed using pore size distribution measurement. The compressive strength measurements are done as well. Experimental results show that the replacement of Portland cement by fine-ground ceramics in the amount of up to 20% by mass is acceptable solution from the mechanical point of view. One can also assume similar physical properties of designed materials to the reference material with only Portland cement as binder.

Keywords—Fine-ground ceramics, cement pastes, early age properties, mechanical properties, pore size distribution, electrical conductivity measurement.

I. INTRODUCTION

RECENT development in manufacturing of building materials is focused on application of secondary raw materials having pozzolanic properties. Many industrial byproducts possessing pozzolanic properties are produced in large amounts worldwide, and despite the current trends of environmental protection and sustainable construction, their production volume still greatly exceeds their industrial reuse. Therefore, there are compelling reasons to extend their practical application in building industry.

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P. Volfová is with the Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague, 166 29 Prague, Czech Republic (phone: +420-2-2435-4688; fax: +420-2-2435-4446; e-mail: petra.volfová@fsv.cvut.cz).

M. Keppert is with the Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague, 166 29 Prague, Czech Republic (phone: +420-2-2435-4688; fax: +420-2-2435-4446; e-mail: martin.keppert@fsv.cvut.cz).

R. Černý is with the Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague, 166 29 Prague, Czech Republic (phone: +420-2-2435-5044; fax: +420-2-2435-4446; e-mail: cernyr@fsv.cvut.cz). These materials can be beneficially applied above all in concrete industry, where they can be used as partial Portland cement replacement in concrete and mortars. In this way, the concrete industry will produce environmentally more friendly materials especially from the point of view of reduction of CO2 production. One possible source for such a pozzolana is calcined clay that is usually product of ceramic industry [1]. A portion of ceramic products formed within the burning illite-group minerals is discarded as scrap, thus constitutes industrial waste. These waste products having suitable fineness can then become active pozzolana [2], [3]. Therefore, waste ceramic materials may have a potential to become a cheaper but almost equivalent alternative to metakaolin as supplementary binder in mortar and concrete [4].

In this paper, fine-ground ceramics is used as an alternative binder in cement pastes mixtures replacing Portland cement in the amount of up to 20% of mass.

II. EXPERIMENTAL

A. Materials and Samples

The researched cement pastes were prepared with Portland cement CEM I 42.5 R as the main binder. Its specific surface area was 341 m²/kg. A part of cement was replaced by fineground ceramics, which specific surface area was 336 m²/kg. The differences in particle size distribution were observed by laser particle size analyser Analysette 22 MicroTec plus. The particle size distribution of used binders is shown in Figs. 1, 2.



Fig. 1 Particle size distribution of applied binders



Fig. 2 Particle size distribution of applied binders

Looking at these results, one can see that the CEM 42.5 R is slightly finer than used ceramics. On the other hand, the fineness of grinding of the applied ceramics is sufficient for optimal pozzolanic reaction.

The composition of pastes mixtures is given in Table 1. Here, the reference mixture without ceramics addition is denoted RP. P10 and P20 are mixtures where the part of the Portland cement is replaced by fine-ground ceramics. As superplasticizer, Mapei Dynamon SX was used. It is based on modified acrylic polymer and allows greater water reduction, along with high mechanical strength and prolonged workability.

TABLE I Composition of Tested Pastes

Mixture	RP	RP10	RP20	
	Amount of material (kg/m ³)			
CEM I 42.5 R	484	435.6	387.2	
Fine-ground ceramics	-	48.4	96.8	
Superplasticizer	5.3	5.3	5.3	
Water	200	200	200	

The measurement of materials properties was done in the early hardening period up to 28 days. The following specimens were used in the experiments for each of the studied materials: compressive strength -3 specimens of dimensions 100 x 100 x 100 mm, monitoring of hydration process using electrical conductivity sensors -3 specimens of dimensions 100 x 100 x 100 x 100 mm, pore size distribution -1 specimen cut from the cubic specimens for the electrical conductivity measurements.

B. Monitoring of Hydration Process

To study the effect of fine-ground ceramics addition on the process of hydration of cement pastes, monitoring of electrical conductivity was done. For that purpose, two rods waveguide electrical conductivity sensors LP/ms by Easy Test were applied. The sensors are made of two 53 mm long parallel stainless steel rods, having 0.8 mm in diameter and separated

by 5 mm. The particular sensors were placed into the cubic moulds containing fresh mixtures of studied pastes and connected to the cable tester TDR/MUX/mts produced by Easy Test. This apparatus was developed in the Institute of Agrophysics PAS, Lublin, Poland, and allows measurement of electrical conductivity, temperature and relative permittivity [5], [6]. The monitoring of electrical conductivity changes was performed for time interval of 28 days.

C. Measurement of Compressive Strength

The compressive strength tests were performed according to ČSN EN 12390-3 [7] on samples 1 and 28 days old. Using this measurement we were able to observe the hardening process of studied materials together with the strength growth.

D.Pore Size Distribution

There are several methods of the experimental assessment of porosity and the relevant quantities such as pore size distribution [8]. In this paper, pore size distribution of investigated materials was measured by mercury intrusion porosimetry using apparatus Pascal 140 and 440 (Thermo). The measurement by mercury porosimetry is based on the physical principle that a non-reactive and non-wetting liquid (in our case mercury) will note penetrate pores until sufficient pressure is applied to force its entrance [8]. As narrow pores must be filled up, high pressure must be applied. The relationship between the applied pressure and the pore size into which the mercury will intrude is given by the Washburn equation [9]

$$P = \frac{2\gamma(t)}{r}\cos\theta,$$
(1)

where *P* (Pa) is the applied mercury pressure, *r* (m) the pore radius, γ (t) the surface tension of mercury, (at 20°C it is equal to 480 mN/m), *t* (°C) temperature, and θ (°) contact angle of mercury and pore wall. In our experimental setup we assumed contact angle 130°. The pore size distribution measurement was performed on dried samples in 7 days intervals.

III. RESULTS AND DISCUSSION

The results of electrical conductivity measurement are presented in Figs. 3, 4. We can observe an increase of the electrical conductivity after the addition of batch water into the studied pastes mixtures, which is due to the increase of temperature of the mix induced by hydration heat generation. Looking at the data from the quantitative point of view, the highest values of electrical conductivity were measured for pure cement paste without ceramics additions. On the other hand, the lowest values were reached for paste P20. The results measured for modified paste P10 were in the middle. The decrease of the amount of free water in the mix with increasing ceramics amount was apparently due to the wetting of ceramics particles.



Fig. 3 Changes of electrical conductivity of studied pastes within first 5 days from casting



Fig. 4 Electrical conductivity of studied pastes measured for time interval of 28 days



The data of electrical conductivity are in agreement with compressive strength results presented in Fig. 5.

As high is the electrical conductivity at the beginning of the hardening process, as high is the material mechanical strength. Clearly, the supposed pozzolanic reaction in the mixes containing ceramics was not yet complete.

The highest values of compressive strength exhibited the pure cement paste. However, the values of compressive strength measured for pastes containing ceramics are also very promising, especially taking into account fact that the part of Portland cement was replaced by waste material.

The results of mercury porosimetry measurements are given Figs. 6-8. The data demonstrate the process of materials' hardening and strength growth.



Fig. 6 Pore size distribution in reference paste RP



Fig. 7 Pore size distribution in paste P10

In Fig. 9, there is presented comparison of pore size distribution of all the tested materials measured for 28 days. We can see that the measured cumulative pore volumes correspond with the results of compressive strength and electrical conductivity measurement. The lowest pore volume exhibited reference paste. Also in this case, the application of fine-ground ceramics did not lead to the significant decrease of

pastes quality and the tested materials with ceramics can be generally considered as acceptable.



Fig. 8 Pore size distribution in paste P20



Fig. 9 Comparison of pore size distribution of all testes materials measured for 28 days

IV. CONCLUSIONS

In this paper, experimental testing of new types of cement pastes containing fine-ground ceramics was presented. The tested materials with partial Portland cement replacement revealed good mechanical strength that was only partially reduced in comparison with the reference paste without ceramics addition. Also the measured pore size distribution confirmed the reasonable quality of the developed materials.

For the practical application of the developed materials in building industry, it will be necessary to perform series of durability tests in order to obtain information on materials' long term behaviour at harmful climatic conditions.

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