Properties of Fine-Grained Concrete with Admixture of Diatomite Powder

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Abstract. The paper reports on the experimental assessment of the physical properties of fine-grained cement-based composites whose composition was enriched with admixture of diatomite powder. Diatomaceous earth, due to its origin and availability, represents highly valuable low cost material that can dispose with high pozzlanic activity. On this account, it can help to mitigate the quantity of used Portland cement, and thus enable to reduce the cost of produced cement-based composites, and accordingly decrease harmful environmental impacts of cement production, especially CO₂ emission. In this work, possible use of diatomite powder as partial Portland cement replacement in concrete composition was studied. The cement replacement ratio was 5 %, 10 %, 15 %, and 20 % by volume. Reference concrete mixture was prepared as well. The water/binder ratio was 0.5, and was kept similar for all prepared composite mixtures. For 28 days water cured samples, bulk density, specific density, strength parameters, and thermo-physical properties were tested. Results of experimental analysis showed high pozzolanic activity of diatomite resulting in reduced porosity and increased mechanical resistance of the developed concretes. The highly porous structure of diatomite particles helped to decrease concrete heat transport properties, despite of low total open porosity of the tested specimens.

1. Introduction
Global warming represents an urgent problem related with greenhouse gases produced due to human activities. Currently, the raised interest is devoted to CO₂ emission generated by traffic and industrial productions [1]. The manufacturing of Portland clinker, the fundamental base of all sorts of cements, is the giant contributor to global warming releasing considerable amount of carbon oxide, when production of each tonne of clinker generates about 950 kg of CO₂ [2]. Supplementary cementitious materials help to lessen the amount of clinker, and thus to mitigate carbon footprint and overall energy required for its production. Clinker replacing materials having fine particulate form are either of the natural origin or in the state of industrial by-products.

Diatomite, defined according to the standard EN 197-1 [3] as a natural pozzolan, represents a sedimentary rock containing predominantly the siliceous fossilized skeleton of diatoms [4]. These skeletons are abundant in opal-A, the source of amorphous silica dioxide. In this respect, diatomaceous earths commonly comprise a major content of SiO₂ (up to 95%), the rest of minor substances is attributed to alumina and iron oxides. Particles with unique porous structure predetermine diatomite as promising material for various kinds of diverse applications due to its specific properties, such as low bulk density, high specific surface area, low thermal conductivity, chemical stability, and inertness [5]. As reported by Ahmadi et al. [6], application of diatomite in cement blends can lead to a comparable strength as
measured on a control material. Authors presented that for 28 days samples was diatomite admixture effective up to 10 % of cement replacement, and for 91 days specimens up to 40 % respectively. Similar observation published, e.g., Kastis et al. [7], who observed that pozzolanic reaction resulted in formation of a higher amount of hydration products, particularly at higher ages of samples. On the other hand, some authors such as Senff et al. [8] described worse workability of fresh composite mixtures, and the subsequent need of higher water demand induced by increased diatomite addition.

As problem associated with the use diatomite in composition of advanced types of construction composites is not fully understood, the effect of diatomite powder as Portland cement replacing material in production of fine-grained concrete was studied in the presented paper.

2. Experimental

The main binder used for preparation of fine-grained concrete was ordinary Portland cement 42.5 R (PC) produced by Heidelberg cement group. This cement, confirming to EN 197-1 [3], was partially substituted by commercially available diatomite powder DP (LB MINERALS, Ltd., Czech Republic).

Both PC and diatomite powder were characterized by their oxide composition with the help of X-ray fluorescence analysis performed with device ED-XRF Quant-X (Thermo Scientific). The testing procedure was based on determination of the content of particular elements of tested powders that was subsequently recalculated to oxide content. In addition, amorphous phase was detected by XRD analysis. Very important information on reactivity of examined powdered materials provided data on particle size distribution and specific surface area. The particle size distribution was measured by a laser particle size analyzer Analysette 22 NanoTec (Fritsch) detecting particles with diameter in the range of 0.08-2 000 µm. For the determination of specific surface area according to the EN 196-6 [9], Blaine device model 62 (Controls, Spa, Italy) was used. Pozzolanic activity of diatomite powder was accessed by the modified Chapelle test that was conducted according to the French standard NF P 18-513 [10].

The physical and chemical parameters of Portland cement and diatomite powder are together with their oxide composition given in table 1. From the obtained data it was evident that studied diatomite had high pozzolanic activity due to its high specific surface area (5.8 times higher compared to Portland cement) combined with high content of amorphous phases (53.9%).

2.1. Physical and chemical analysis

Table 1. Oxide composition and basic properties of used cement and diatomite.

| Oxide composition (mass %) | PC  | DP  |
|---------------------------|-----|-----|
| SiO₂                      | 19.00| 74.50|
| Al₂O₃                     | 4.31 | 11.00|
| Fe₂O₃                     | 2.40 | 1.34 |
| TiO₂                      | 0.28 | 0.60 |
| CaO                       | 62.90| 0.16 |
| MgO                       | 1.80 | 0.23 |
| K₂O                       | 0.82 | 0.78 |
| Na₂O                      | 0.14 | 0.12 |
| SO₃                       | 3.24 | -    |
| Amorphous phase (mass %)  | -   | 53.90|
| Apparent density (kg·m⁻³) | 3 129| 2 416|
| Powder density (kg·m⁻³)   | 970 | 302  |
| Specific surface (m²·kg)  | 360 | 2 095|
| Pozzolanic activity (mgCa(OH)₂·g⁻¹) | - | 1 441 |

In order to reveal the contribution of diatomite powder admixture to concrete properties, five different concrete mixtures have been formulated and prepared. Reference mixture (RC) consisted of PC and silica sand mix of three fractions 0.0/0.5; 0.5/1.0 and 1.0/2.0, mixed in mass ratio 1/1/1. In other
concrete samples, PC was substituted by diatomite in the range from 5 % to 20 % by volume. The amount of batch water remained same for all prepared concrete mixtures. The water/binder ratio was 0.5. Composition of the examined concrete mixtures is presented in table 2.

| Substance | Content (kg·m⁻³) PC 1 | DP | Sand | Water |
|-----------|------------------------|----|------|-------|
| RC        | 496.2                  | -  | 1488.6 | 248.1 |
| DP 5      | 490.3                  | 7.7| 1494.0 | 249.0 |
| DP 10     | 481.6                  | 15.8| 1492.2 | 248.7 |
| DP 15     | 470.6                  | 24.5| 1490.1 | 248.4 |
| DP 20     | 462.4                  | 34.3| 1485.3 | 247.6 |

The input raw materials were mixed with mixer Spar D 200-B with vertical axis. The casted specimens were prisms having dimensions of 40 × 40 × 160 mm, and 70 mm cubes. After 24 hours of initial hardening in laboratory at temperature of (22 ± 1) °C and relative humidity of (45 ± 5) %, samples were demoulded and stored in water for overall time of 28 days.

The rheology of concrete fresh mixtures was measured according to the EN 1015-9 [11] with the help of penetrometer BS PNE 02 (Beton System, Ltd.). The values of the determined penetration time corresponded to penetration pressure of 0.5 MPa induced by iron cylinder.

For 28 days cured samples, basic structural, mechanical, and thermo-physical properties were accessed. Bulk density was determined according to the standard EN 1015-10 [12]. Specific density values were accessed by helium porosimeter Pycnomatic ATC (Thermo-Scientific). Total open porosity was calculated using the bulk density and matrix density values. The relative expanded uncertainty of the porosity assessment was 5 %. Flexural strength was measured in three point bending test arrangement on 40 × 40 × 160 mm prisms. On the rest of broken prisms (loading area 40 × 40 mm), compressive strength was tested [13]. The expanded measuring uncertainty of the both strength tests was 2 %. Among mechanical parameters, dynamic Young’s modulus was also measured. For this purpose, device Dio 562 NLF (Starmans Electronics) was applied. The relative expanded uncertainty of the dynamic Young’s modulus measurement was 2 %. Thermo-physical properties, such as thermal conductivity, thermal diffusivity, and volumetric heat capacity were tested on dried 70 mm cubes by the use of a portable device ISOMET 2114 (Applied Precision) equipped with plate circular probe. The measuring range of the thermal conductivity was 0.015-6.0 W·m⁻¹·K⁻¹ with the measurement accuracy 5 % of reading + 0.001 W·m⁻¹·K⁻¹. For the volumetric heat capacity was the accuracy 10 % of reading, and the reproductibility was 3 % of reading + 1·10³ J·m⁻³·K⁻¹ [14]. The tests of thermal parameters were conducted at laboratory conditions at temperature (22 ± 1) °C.

3. Results and discussion

Particle size distribution of both binder components is shown in figure 1. Obtained distribution curves had practically similar shape, however, diatomite with particles diameter reaching up to 100 µm was coarser compared to cement used. At first sight, the coarser particles of DP could partially retard the progress of pozzolanic reaction in spite of their high reactivity. Nevertheless, in combination with other mix components, they may properly supplement overall particle distribution and partially contribute as filler.
Figure 1. Particle size distribution of PC and DP

Basic physical properties measured on hardened specimens after curing period are summarized in table 3. The incorporation of diatomite powder resulted in the decreased total open porosity, whereas the most obvious reduction in porosity of about 11.6 % was observed for concrete DA 5. For other diatomite enriched samples, the drop in porosity was lower. In a closer look one can see that concrete with 20% volumetric cement substitution reached almost similar porosity as concrete with 10% PC replacement. This fact can be attributed on the one hand to the considerable pozzolanic activity of diatomite powder and on the other hand to the filler effect of its coarser particles. These grains allow better redistribution of batch water in the sense of filling gaps between binder and sand particles, and thus help to create denser and durable concrete structure.

Table 3. Basic physical properties of tested concretes.

| Mixture | Bulk density (kg·m⁻³) | Specific density (kg·m⁻³) | Total open porosity (%) |
|---------|------------------------|---------------------------|-------------------------|
| RC      | 2 079                  | 2 514                     | 17.3                    |
| DA 5    | 2 216                  | 2 497                     | 15.3                    |
| DA 10   | 2 089                  | 2 487                     | 16.0                    |
| DA 15   | 2 052                  | 2 476                     | 17.1                    |
| DA 20   | 2 062                  | 2 463                     | 16.3                    |

Strength parameters, dynamic moduli, and penetration time data is presented in table 4. The Portland cement substitution with diatomite powder led to the gradual shortening of penetration time proportional to the increasing content of diatomite in concrete mixture. The worsened workability of concretes with admixed diatomite we assign to its pozzolanic activity and porous particles of high specific surface requiring higher amount of batch water for their wetting. The flexural strength, compressive strengths, and dynamic moduli data corresponded well with the total open porosity results. From the quantitatively point of view, the highest compressive strength was recorded for concrete samples containing diatomite in the dosage of 5 % by volume. The compressive strength of this material was approximately of about 32 % higher than obtained for reference concrete. Similar and considerable compressive strength exceeding 65 MPa reached also hardened samples of concretes DA 10 and DA 20. On the other hand, concrete DA 15 having the highest porosity among the diatomite enriched composites, exhibited the lowest mechanical parameters, nevertheless, still significantly higher compared to reference material.

The dependence of thermo-physical parameters of tested materials on the dosage of diatomite in concrete composition is apparent from table 5. Obtained results show clear contribution of highly porous diatomite particles to the decrease of both the thermal conductivity and thermal diffusivity values of concretes with cement/diatomite blends compared with ordinary reference concrete. Accordingly, also the volumetric heat capacity decreased with increasing content of diatomite in concrete composition.
The slowest heat transport exhibited concrete with 15% PC substitution with diatomite, what was due
to its highest porosity and high content of porous DP particles.

### Table 4. Mechanical parameters and penetration time.

| Composite | Flexural strength (MPa) | Compressive strength (MPa) | Dynamic moduli (GPa) | Penetration time (min) |
|-----------|-------------------------|---------------------------|---------------------|------------------------|
| RC        | 9.6                     | 54.1                      | 30.1                | 150                    |
| DA 5      | 11.8                    | 71.6                      | 33.6                | 130                    |
| DA 10     | 10.9                    | 66.9                      | 32.5                | 125                    |
| DA 15     | 10.2                    | 60.1                      | 30.3                | 115                    |
| DA 20     | 10.4                    | 65.6                      | 31.4                | 110                    |

### Table 5. Thermo-physical characteristics.

| Material | λ / (W·m⁻¹·K⁻¹) | a × 10⁻⁶ / (m²·s⁻¹) | c × 10⁶ / (J·m⁻³·K⁻¹) |
|----------|-----------------|---------------------|-----------------------|
| RC       | 2.58            | 1.40                | 1.85                  |
| DA 5     | 2.36            | 1.32                | 1.82                  |
| DA 10    | 2.13            | 1.18                | 1.80                  |
| DA 15    | 1.95            | 1.16                | 1.62                  |
| DA 20    | 2.05            | 1.19                | 1.71                  |

### 4. Conclusion

Obtained experimental results revealed high value of diatomite for use as cement replacement in
manufacturing of fine-grained building composites. It was due to diatomite considerable pozzolanic
activity that resulted in the decreased concrete porosity and thus improvement of mechanical resistance
even for its dosage up to 20% by volume of PC. Nevertheless, the best mechanical resistance exhibited
concrete with 5% PC substitution. The only negative effect that brought diatomite use in concrete
composition was worsening of fresh concrete mixtures workability due to the high specific surface and
rugged surface of diatomite particles. However, specific hollow and structured diatomite particles
reduced concrete thermal conductivity. In summary it can be concluded, diatomaceous earth in the form
of fine powder represents valuable and effective material for cement-based composites manufacturing.

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