Parameters of mortars supplemented with chalcedonite powder

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Abstract. Laboratory tests were conducted of mortars supplemented with 0%, 5%, 20%, 35%, 50% of chalcedonite powder. Chalcedonite powder replaced the percentage amount of cement. The subject of tests was impact of admixture on properties of hardened mortars including: compressive strength, bending strength, water absorption and capillary rising. Tests of compressive strength were conducted following 2, 7, 14, 28, 56 and 90 days, whereas water absorption, capillary rising were tested following 28 days. The mortar demonstrated the biggest compressive strength where cement was replaced with admixture in 5% and following curing period of 90 days it was 72.1MPa. Mortars where cement was replaced in 5%, 20%, 35% and 50% demonstrated decrease of bending strength comparing to the reference mortar. During the test regarding water absorption, 50% admixture of powder resulted mass increase by 6.1% in relation to the reference mortar, whereas 20% admixture of chalcedonite powder resulted in mass increase by 3.9% in relation to the reference mortar. Water absorption resulted from capillary rising during tests was below 3.5%.

1. Introduction

Pursuant to PN-EN 206-1:2014 Standard a concrete admixture relates to “fine grained component used to enhance some concrete properties or to ensure specialized properties of concrete”. The Standard considered two types of inorganic admixtures: almost inert admixtures (Type I), e.g. filler aggregate, dyes, and other, as well as of pozzolanic properties or hidden hydraulic properties (Type II) for example fly ash, silica fume, blast furnace slag. PN-EN 206-1:2014 Standard recommended initial laboratory tests to determine impact on some parameters of admixtures in mortars considering its quantity and type. The most commonly used admixtures in mortars include: grinded blast furnace slag, fly ash and silica fume added mainly in quantity over 5% of cement mass. It is most often applied as cement equivalents at proper proportion. Precise selection of admixtures has positive impact on rheological properties and parameters of hardened mortar during long time of hardening. Lately there was growing interest [1-3] in latest admixtures which modify rheological properties and parameters of hardened mortars. Many authors of scientific papers raised the issue regarding management of wastes generated during production of crushed aggregates. Those admixtures include: chalcedonite powder and ash, as well as mineral ash and powder. Use of new wastes in practice requires assessment regarding its performance. Use of wastes as admixtures during production of concrete mixtures and mortars fits into Sustainable Development Strategy. Proper quantity of admixture in mortar enables to optimize consumption of cement clinker, saves natural raw materials and reduces negative impact on...
natural environment thanks to reduction of CO₂ emission [1-5]. Due to quantity of wastes generated during production of crushed aggregates, it is necessary to find methods for management thereof. However safe use thereof required broad know-how based on results of laboratory tests considering environmental and technical aspects, as well as changes in the legislation. Therefore analyses will be necessary regarding impact of chalcedonite powders and ashes on performance of cement composites taking into account their composition, and the results thereof will be indispensable at design phase of mortars purposed for specific use [9-12].

2. Materials and Methods

Laboratory tests were conducted with five series of mortar containing chalcedonite powder admixture. Chalcedonite powder replaced the percentage amount of cement, namely 0% (batch 0), 5% (batch 5), 20% (batch 20), 35% (batch 35), 50% (batch 50). Mortars were manufactured on basis of Portland cement CEM I 42.5R (Table 1), quartzite sand 0-2mm, as well as water of constant ratio w/s=0.50.

| No. | Cement type  | Chemical composition, %  | Blaine, cm²/g  | Resistance, MPa |
|-----|--------------|--------------------------|----------------|-----------------|
| 1   | CEM I 42.5R  | SiO₂ 19.49 | CaO 62.3 | MgO 2.08 | Fe₂O₃ 3.25 | Al₂O₃ 4.75 | 4053 | R₂ 32.0 | R₂8 53.0 |

Table 1. Information on basic properties of the cement, [www.dyckerhoff.pl].

Chalcedonite powder relates to waste generated by crushed aggregate quarries. This admixture is made from chalcedonite rock and it mainly consists silica. The production process includes mainly repeated crushing, sifting and drying, to obtain mineral powder free of humidity and of appropriate grade. The end product relates to sand and chalcedonite gravel.

Particle size of chalcedonite powder was measured with laser diffraction spectroscopy.

\[
x_{10} = 0.28 \ \mu m \quad x_{50} = 3.37 \ \mu m \quad x_{90} = 25.53 \ \mu m \quad SMD = 0.90 \ \mu m \quad VMD = 9.50 \ \mu m
\]

\[
x_{16} = 0.44 \ \mu m \quad x_{54} = 22.38 \ \mu m \quad x_{99} = 34.99 \ \mu m \quad \Sigma V = 6.64 \ \text{m}^3/\text{cm}^3 \quad \Sigma m = 66392.20 \ \text{cm}^2/\text{g}
\]

Figure 1. Particle size of chalcedonite powder.

Precise grading of powder was determined on basis of laboratory tests. Quantity of grade sized up to 2µm is 17.5%, whereas those sized from 2 to 10 µm make for 22.5%. Grades sized from 10 to 40 µm make for the most quantity equal to 42.5%. Actually 99% of grades are sized below 72µm.
Figure 2. Chalcedonite powder microstructure.

Table 2. Chalcedonite powder chemical composition.

| Chemical compound | Quantity pierwiastków, [%] |
|-------------------|---------------------------|
| F                 | 0.06                      |
| Na₂O              | 0.09                      |
| MgO               | 0.15                      |
| Al₂O₃             | 3.04                      |
| SiO₂              | 91.8                      |
| P₂O₅              | 0.04                      |
| SO₃               | 0.05                      |
| K₂O               | 0.42                      |
| CaO               | 0.45                      |
| TiO₂              | 0.11                      |
| Fe₂O₃             | 1.28                      |
| ZrO₂              | 0.01                      |
| BaO               | 0.04                      |

Table 3. Mortar composition.

| Sample | Cement [g] | Chalcedonite powder [g] | Sand [g] | Water [g] |
|--------|------------|-------------------------|----------|-----------|
| 0      | 450        | 0                       | 1350     | 225       |
| 5      | 427.5      | 22.5                    | 1350     | 225       |
| 20     | 360        | 90                      | 1350     | 225       |
| 35     | 292.5      | 157.5                   | 1350     | 225       |
| 50     | 225        | 225                     | 1350     | 225       |

Laboratory tests considered also impact of mortar hardening period (2, 7, 14, 28, 56, 90 days) and quantity of admixture on some parameters (compressive strength and bending strength, water absorption during capillary rising).
Table 4. Bulk density of tested mortars.

| No. | Series | Density [kg/dm³] |
|-----|--------|-----------------|
| 1   | 0      | 1.95            |
| 2   | 5      | 1.89            |
| 3   | 20     | 1.84            |
| 4   | 35     | 1.79            |
| 5   | 50     | 1.73            |

The more cement was replaced with chalcedonite powder the higher decrease followed of mortar bulk density.

Compressive strength has been tested in accordance with PN-EN 206-1:2014 Standard. Following forming the samples were stored in water at temperature of +18°C during the whole period of hardening. Tests regarding compressive strength were conducted following 2, 7, 14, 28, 56, 90 days [8].

Tests regarding capillary rising were conducted according to PN-88/B-06250 Standard with samples sized 4x4x16cm. Following forming the samples were stored in water at temperature of +18°C during period of 7 days. During next days the samples were stored in dry air environment at temperature +18°C. Increase of sample mass was determined following 28, 56 and 90 days of hardening period. Before tests samples were placed inside the conditioning chamber for 72 hours. Then mass increase was determined following 15 min, 30 min, 1h, 4h since sample contacted water. Next measurements were taken every 24 h. Samples were immersed at level of ca. 3mm.

Tests regarding water absorption were conducted pursuant to PN-88/B-06250 Standard with samples sized 4x4x16cm. The samples were stored for 7 days in water at temperature +18°C, and for the next days in the air at temperature +18°C. The samples underwent drying for 72 h inside the conditioning chamber. Next samples were immersed in water up to half of their height for 24 hours. Next water was refilled to ensure level +1cm over sample height. Increase of mass was determined every 24 h, until two identical result were obtained [4].

3. Description of research

Tests regarding cement setting time were conducted pursuant to PN-EN 196-1:2006 Standard.

![Figure 3. Beginning and end of cement setting time.](image)

The analysis regarding the beginning of setting indicated that cements containing chalcedonite powder admixture demonstrated delayed beginning of setting comparing to the cements without this admixture. Quantity of chalcedonite powder had impact on change of time regarding beginning and end of setting.
Figure 3 demonstrated an average increase of compressive strength of mortars with chalcedonite power admixture in 5%, 20%, 35%, 50% and the reference product.

![Graph showing increase of compressive strength over time for different admixtures.]

**Figure 4.** Increase of mortar bending strength.

**Table 5.** Average compressive strength versus time.

| Tested batch | Compressive strength [MPa] |
|--------------|----------------------------|
|              | 2 days | 7 days | 14 days | 28 days | 90 days |
| 0            | 23.5   | 51.4   | 59.4    | 62.8    | 70.3    |
| 5            | 22.6   | 56.8   | 62      | 65.3    | 72.1    |
| 20           | 24.6   | 51.7   | 56.1    | 58      | 62.6    |
| 35           | 17.2   | 38.1   | 40.6    | 41.5    | 43.3    |
| 50           | 10.2   | 17.2   | 23.1    | 26.5    | 27      |

Analysis of obtained results indicated that mortars containing 5% of chalcedonite powder (5 batch) demonstrated the biggest increase of compressive strength. Following 90 days compressive strength was obtained of 72.1MPa. Samples with chalcedonite powder at quantity of 20%, 35% and 50% featured with decreased compressive strength comparing to mortars without admixture. The tendency was evident that the more cement was replaced with admixture of chalcedonite powder the more decrease followed on compressive strength. Mortars with chalcedonite powder in quantity of 50% demonstrated the biggest decrease of compressive strength that actually was 61.6%. Note also that samples containing powder quantity of 5% featured with higher increase of compressive strength comparing to the reference concrete since 2 day of hardening period.
Figure 5. Increase of bending strength.

The reference mortar demonstrated the biggest bending strength that actually was 2.4 MPa following 90 days of hardening. The mortar where cement was replaced with chalcedonite powder in 5% demonstrated bending strength decrease by 12.5% in relation to the reference mortar. The mortar where cement was replaced with chalcedonite powder in 50% demonstrated bending strength decrease by 91.6%.

Figure 6. Increase of mortar mass during water absorption test following 28 days of hardening period.

The analysis of obtained results indicated that the admixture where cement was replaced caused increase of mass during test regarding water absorption. The admixture with chalcedonite powder in 50% resulted in mass increase by 6.1% comparing to the reference mass, whereas admixture containing 20% of chalcedonite powder resulted in mass increase by 3.9% comparing to the reference mortar.
Figure 7. Mass increase during test regarding mortar capillary rising following 28 days of hardening period.

Quantity of absorbed water during capillary rising was below 3.5\% in case of tested batches. The smallest water absorption was recorded for the batch where cement was replaced with chalcedonite powder in 5\% and actually it was 1.2\%.

Figure 8. CEM I 42.5R cement mortar X-ray examination at point 1, 2, 3.
Figure 9. X-ray examination of mortar with chalcedonite powder in 50% at 1, 2, 3 point.

Examination of CEM I 42.5R cement mortar microstructure (Fig. 8) and the mortar where cement was replaced with chalcedonite powder in 50% (Fig. 9) performed with scanning electron microscope indicated that the admixture was active in hydration. This confirmed absence of clear boundaries between powder particles and C-S-H fraction, and the Figure 9 demonstrated high increase for silicone and decrease for calcium.

4. Conclusion
Test results provided basis for the following conclusions:
1. The highest increase of compressive strength was recorded for mortars where cement was replaced with chalcedonite powder in 5%. Replacement of cement with chalcedonite powder in 20% resulted in compressive strength by 10.95%. The higher content of admixture in the mortar the higher decrease followed of compressive strength.
2. Chalcedonite powder admixture results in decrease of bending strength for all tested batches. The highest bending strength was recorded in case of reference mortar that was 2.4MPa following 90 days of hardening.
3. Test regarding water absorption demonstrated the highest increase of mass in mortars with chalcedonite powder added in 50% that actually was 6.1%. Water absorption below 5% was demonstrated by mortars supplemented with chalcedonite powder in 5% and 20%.
4. Replacement of cement with chalcedonite powder did not result in water absorption over 3.5% in tested mortars.
5. Chalcedonite powder resulted in delayed beginning and end of cement setting. The more cement was replaced with this admixture, the biggest delay of setting beginning followed and extension of setting period.
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