HPC cement materials prepared by mixing under reduced pressure

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Abstract. This article deals with HPC (High Performance composite) materials whose main advantages are increased mechanical properties, durability and stability. The limiting parameter for such materials is the presence of air bubbles that cannot be removed just by the compacting. Mixing of cement mixtures under reduced pressure can be used to reduce porosity and eliminate the presence of air bubbles. Moreover, it is associated by the improvement of the mechanical properties. The influence of preparation of HPC by mixing under the atmospheric pressure and by mixing under reduced pressure to parameters like mechanical properties, porosity, microstructure was studied in this paper. The porosity of the samples was analysed in a cross-section using an optical method. The effect of the reduced pressure on the resulting strength of the samples was observed.

1. Introduction

Concrete is the most common building material elsewhere on the world. The typical concrete usually consists of Ordinary Portland Cement (OPC) mixed with water and coarse aggregates which is accompanied with a low homogeneity. The nowadays society focuses not only on the utility properties of the materials but on the environmental and economic aspects as well. The clinker production process is related with rapid CO₂ emission (about 7% of total world’s production), thus it is not surprising that scientists all around the world are trying to find some alternatives, whose can help to eliminate production of greenhouse gases. The most common is probably utilization of secondary raw materials. Considering their utilization (blast furnace slag, fly ash, microsilica) as a partial replacement of clinker in cements improvement of mechanical properties, durability same as the reduction of the size of the particles can be achieved [1].

For the same amount or reduction of Portland cement and increase of mechanical properties of the material, it is possible to reduce the size of the particles or add reactive components (such as blast furnace slag, microsilica, fly ash). Another important factor, which influences mechanical parameters of concrete, is the water to binder ratio (w/b). The reduction of water is achieved by plasticizing additives. These types of concrete are typically classified as HPCs (high-performance cement composites). Concrete prepared from a large amount of Portland cement is characterized by a high hydration temperature. This cause in micro-cracks. Reduction of microcracks formed in concrete due to this effect can be achieved by water curing [1, 2].

RPC (Reactive powder concrete) is a new field of development of cement materials and it belongs to the UHPC (Ultra – high performance composite) category. UHPC materials were developed due to various aspects. The main ones were high price of OPC and environmental burden associated with...
the cement production beside the superior mechanical properties and durability when compared with traditional concrete based on OPC. The RPCs allow us to produce material with improved mechanical properties, with lower price and environmental burden than the traditional concrete (with OPC). Instead of traditional aggregates, very fine sand is used in this case. Fine sand with a maximum particle size of 0.4 mm is used. The low w/b is compensated by the addition of a superplasticizer. RPC materials use very small highly homogenous particles without aggregates to form dense and homogenous matrix. Due to these properties, micro-cracks are limited and shrinkage is very small. It is possible to achieve compressive strengths around 200 MPa after 28 days [2, 3, 4, 5, 6].

The pozzolanic reaction of this material may be similar to traditional compositions. The hydration processes depend on the type of curing (dry, water) same as on the type of reactive component of concrete. An undesirable property of this material is increased brittleness. Fibres (steel, carbon, PVA) can be doped into the matrix to increase the toughness and material durability [1, 7, 8].

The microstructure is an important factor in concrete preparation. To improve the microstructure, it is possible to realize aging of concrete at elevated temperatures or to improve the mixing process. Mixing under reduced pressure causes a high reduction of the porosity. Porosity can cause local stress concentration which can be accompanied by the material rupture. Vacuum mixing can limit these problems. The porosity of concrete with standard composition is around 5–10%. In samples under reduced pressure, it is possible to achieve 1/10 porosity [9, 10, 11, 12].

This article deals with the preparation of RPC materials with the aim to compare two mixing methods – standard mixing and mixing under reduced pressure. The influence of the mixing procedure on the microstructure, porosity and mechanical parameters was studied and evaluated. Reduced porosity can be expected to increase mechanical resistance.

2. Materials and experimental part

White Portland cement CEM I 52.5 R (Aalborg), silica fume RW-Füller Q1 (RW silicium, Germany), limestone SBL CL 90-Q (Kotouč Štramberk, spol. s r.o., Czech Republic), fine sand (Filtrační písky, spol. s r.o., Czech Republic), superplasticizer Master Glenium ACE 446 (BASF Stavební hmoty Česká republika s.r.o., Czech Republic) and deionised water were used in this study. The chemical composition of cement is given in table 1, the particle size distribution of sand, limestone, silica fume and cement shows table 2.

Chemical composition was measured by manufacturer by Heidelberg Cement group and it is shown in table 1.

| Chemical composition of the applied white cement determined by XRF. |
|---------------------------------------------------------------|
| C₃S | C₃S | C₃A | C₄AF | MgO | SO₃ | Cl | Na₂O |
| CEM I 52.5 R Aalborg white | 73 | 16 | 5 | 1 | 0,6 | 1.8-2.3 | ≤0.04 | ≤0.3 |
Particle size distribution of CEM I 52.5 R Aalborg cement and silica fume measured by laser diffraction analyzer (HELOS KR Sympatec) are shown in table 2.

| Distribution of particle sizes |
|-------------------------------|
| D50 [µm]                      |
| CEM I 52.5 R Aalborg white     | 8.7 |
| Quartz flour ST 09             | 6   |
| Fine sand                     | 140 |
| Silica fume                   | 1.9 |

The dry components were homogenized in the mixer (KitchenAid) for 1 minute. After that mix 75% of water and superplasticizer were added. The mixture was stirred for 5 minutes. After that 25% of the remaining water was added and stirred for 3 minutes. The mixture was transferred to molds with a test specimens’ dimension of 40 × 40 × 160 mm. These forms were compacted by vibrating table for 45 seconds.

The mixture was placed into the vacuum mixer and stirred intensively for 1 minute, before compaction in case of samples under the reduced pressure. Subsequently, the mixtures were placed in molds and compacted, as in the previous samples without the use of the vacuum. After 24 hours, the experimental specimens were demoulded and placed in temperature-controlled (laboratory temperature 25°C) water curing for optimal concrete aging.

Processability of the mixture was tested by Hägermann-flow table for the flow test. Firstly, filled cone was removed and spread was measured. Second test was measured after 15 strokes.

Mechanical properties were tested by DESTTEST, BETONSYSTEM (FCH BUT). Flexural strength (3-point arrangement) and compressive strength tests were performed after 1, 7 and 28 days. Furthermore, the density of concrete samples was measured. After 28 days, samples were sawed and the level of porosity was determined. A white cement paste was prepared to fill the pores. The prepared samples were photographed. The porosity was evaluated using an ImageJ program. The efficiency of mixing under the reduced pressure was evaluated based on the results obtained (compressive and flexural strengths; porosity).

3. Results and discussion

3.1. Preparation of RPC material

The solid phase composition was the same for all mixture experiments. The highest water to binder ratio used was 0.22. Other samples were prepared with a 0.02 reduction step up to the workability limit. The mixture with a w/b = 0.22 was very fluid and easy to process. During forming, easy degassing was possible due to the fluidity of the system. The next mixture was w/b = 0.20, where the viscosity increased but the workability of the mixture was optimal. The last step was the mixture with w/b = 0.18, when the reduction of the amount of water in the mixture was ended, because the processability and the possibility to homogenize the mixture were difficult. Spread data are shown in table 3. The compositions of the individual mixtures are given in table 4.
Table 3. Summary of Hägermann-flow table test.

| w/b       | Before strokes [mm] | After 15 strokes [mm] |
|-----------|---------------------|----------------------|
| 0.18      | 105                 | 130                  |
| 0.2       | 105                 | 145                  |
| 0.22      | 110                 | 180                  |

Table 4. Composition [g] of measured samples.

|                      | w/b 0.18 | w/b 0.20 | w/b 0.22 | w/b 0.18 vac | w/b 0.20 vac | w/b 0.22 vac |
|----------------------|----------|----------|----------|--------------|--------------|--------------|
| CEM I 52.5 R [g]     | 225      | 225      | 225      | 225          | 225          | 225          |
| Silica fume [g]      | 75       | 75       | 75       | 75           | 75           | 75           |
| Quartz flour ST_09 [g]| 75      | 75       | 75       | 75           | 75           | 75           |
| Fine sand [g]        | 360      | 360      | 360      | 360          | 360          | 360          |
| Limestone SBL CL 90-Q [g] | 15     | 15       | 15       | 15           | 15           | 15           |
| Superplasticizer ACE 446 [g] | 7.5   | 7.5      | 7.5      | 7.5          | 7.5          | 7.5          |
| Deionised water [g]  | 54       | 60       | 66       | 54           | 60           | 66           |

As shown in table 4, the same mixtures were stirred under reduced pressure. The mixture was transferred to a mixer for stirring under reduced pressure and stirred intensive for 1 minute. The pressure was measured with a barometer. The pressure in the mixer was adjusted to approximately 1/10 atmospheric pressure using an oil pump. After stirring, the mixture was homogeneously transferred to molds and compacted.

3.2. Testing of mechanical parameters
Samples were demoulded and put into a water curing after 24 hours to achieve a better final property of the material.

The flexural strength of the sample with w/b = 0.18 was about 8 MPa after 1 day of hydration. When compared to a sample under reduced pressure, influence of mixing under the reduced pressure on the increase in flexural strength was obvious. Same trend was observed for samples measured after 7 and 28 days. As expected, the maximum flexural strength was obtained by samples prepared using the mixing under the reduced pressure after 28 days of hydration. The flexural strengths measured were up to 32 MPa as shown in figure 1. The compressive strength of the samples was measured after 1 day without using vacuum mixing and reached 87 MPa. The maximum value (190 MPa after 28 days) was measured for the samples using reduced pressure mixing [9, 12, 13].
The values of flexural and compressive strength of the specimens with w/b 0.18 are shown in figure 2. The obtained values were lower than the ones of mixtures with w/b = 0.2. It can be related to the higher w/b coefficient since the sufficiency of water is necessary to obtain the desired strength. The flexural strength of samples with w/b = 0.20 was 7 MPa after 1 day, which is not as much as in case of w/b = 0.18. The flexural and compressive strength after 28 days were 27 MPa, respectively 190 MPa for the samples prepared using the vacuum technique. No significant difference was observed between the samples with 18 and 20% water. The only difference was a better workability for the mixture with the 20% water content.
Figure 2. Flexural and compressive strength of the specimens w/b = 0.20.

Figure 3 summarizes the flexural and compressive strengths of specimens with w/b = 0.22. The reduction of compressive strength was observed in case of the specimen with w/b = 0.22 in comparison with specimens with w/b = 0.20 and 0.18. The compressive strength of the sample with w/b = 0.22 prepared under the reduced pressure is 160 MPa, which is 30 MPa less than in the case of the specimens with w/b = 0.20 and 0.18. This can be attributed to the relatively high water content in the mixture connected with excessive fluidity, which caused additional air capturing during the further processing (removing from the mixer, molding process) and thus the increase of porosity. Therefore, the optimal water coefficient for the tested mixtures was determined to be 0.2.
3.3. Measure of porosity

The porosity was significantly reduced by the mixing under the reduced pressure as can be seen in the figure 4. Figure 4 (a) is the sample prepared by mixing at atmospheric pressure where the pores were filled with white cement paste. Samples were photographed and analysed using ImageJ programme. The calculated porosity for the unvacuated samples with a water coefficient w/b = 0.18 was 7.3%, after stirring under reduced pressure only 0.67%. Such a significant decrease in porosity positively influences the mechanical resistance, which correlates with the data in figure 7 [9].
Figure 4. Analysis of porosity of the specimens w/b = 0.18, a) atmospheric pressure, b) reduced pressure.

The data in table 5 show similar level of porosity for specimen w/b = 0.20 and w/b = 0.18. Significant decrease of porosity of the samples was observed when the vacuum was applied during the mixing process, as can be seen in figure 5. An apparent positive influence of vacuum mixing on the physico-mechanical properties, in particular the strength and porosity, was also reported by [11].

Figure 5. Analysis of porosity of the specimens w/b = 0.20, a) atmospheric pressure, b) reduced pressure.

In the figure 6, a higher porosity is visible, when compared with the samples prepared under reduced pressure. In table 5, the porosity values are shown and it can be seen that the mixture was probably too fluid and thus new porosity was formed in the samples during moulding. This correlates with the measured mechanical strength of the specimen w/b = 0.22, which did not achieve as high resistance as the samples with lower water coefficient. Similar results were obtained by Zdeb T., who described a significant porosity reduction after mixing with reduced pressure [14].
Figure 6. Analysis of porosity of the specimens w/b = 0.22, a) atmospheric pressure, b) reduced pressure.

Table 5. Summary of measured porosity of specimens.

| Porosity [%]     |
|------------------|
| w/b 0.18         | 7.3 |
| w/b 0.20         | 8.98|
| w/b 0.22         | 6.48|
| w/b 0.18 vac     | 0.67|
| w/b 0.20 vac     | 0.42|
| w/b 0.22 vac     | 1.58|

In the figure 7, the effect of porosity on the mechanical strengths of samples is presented. The principle is similar to that described by Malecot et all [15]. This figure is a summary of results and influence of porosity on mechanical strength is apparent. The workability of the mixture is also different. When the water content was 22%, mixture was too fluid and it was not possible to achieve such a low porosity. Further reduction of the water coefficient under w/b = 0.18 would make homogenization impossible.
Figure 7. Summary of the influence of porosity on compressive strength after 28 days of hydration.

As described in figure 7, reduced porosity increases compressive strength, which is consistent with the results published by Edwin et al. [9]. The effect of mixing under reduced pressure on the resulting strength is apparent.

4. Conclusion
The series of samples of RPC mixtures were prepared with a goal to determine the influence of the type of mixing (standard/vacuum) and the water content on the mechanical properties and the total porosity of the designed RPC. The selected w/b ratios varied from 0.18 up to 0.22 with a 0.02 step. Considering the workability and final mechanical parameters, the w/b ratio of 0.2 was determined as optimal for the proposed RPC mixtures. The results indicate, that the large increase in flexural (up to 37%) and compressive (up to 34%) strengths can be achieved by applying vacuum during the mixing process. Significant decrease of porosity of the samples prepared by vacuum mixing was observed. The porosity was reduced from 7.3%, 8.98% and 6.48% to 0.67%, 0.42% and 1.58% in case of samples with w/c = 0.18, 0.20 and 0.22 respectively. It was proven, that applying the vacuum mixing technology in production process can significantly improve the homogeneity and load bearing capacity of the RPC materials, which makes them more suitable for several demanding applications as ballistic protective structures or thin shell structures.
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