Modified fine-grained concretes based on highly filled self-compacting mixtures

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Abstract. In this article the results of study of rheo-technological characteristics of cement-carbonate suspensions and physical and mechanical characteristics of cement composites from highly filled self-compacting mixtures are presented. The optimal dosage of polycarboxylate superplasticizer (Melflux 5581 F) and rheologically active mineral filler (microcalcite) in cement-mineral suspensions when developing self-compacting fine-grained concrete mixtures is defined. The water demand of fine natural quartz sands using the methodology of Skramtayev and Bazhenov is studied. The compositions of self-compacting fine-grained concrete mixtures of workability class SF1 with fine natural quartz sand are developed. High physical and mechanical characteristics of fine-grained concretes from highly filled self-compacting mixtures are fixed: density in normal humidity conditions at the age of 28 days up 2393 kg/m³; bending and compressive strength at the age of 28 days up 14.9 and 115.8 MPa, respectively.

1. Introduction

One of the top-priority strategic trends in modern construction material science is the development of advanced energy-efficient modified cement concretes differing in a number of high physical and technical characteristics [1, 2, 3, 4, 5]. A common feature of modern modified cement composites is high strength (60÷100 MPa and higher), while other strength parameters increase when the content and size of the aggregate decrease reaching maximum when forming a high-strength fine-grained structure characterized by high homogeneity and no destruction hazard in the large aggregate/cement stone interface [6]. The efficiency of the concrete fine-grained structure increasing highly strong cement composites has been proved by the results of own studies [7, 8, 9, 10].

It should be noted that in the development of highly strong heavy and fine-grained concretes, a determinant role has been played by a scientific basis for modifying cement systems with chemical and active mineral additives, which have been formed as a result of multiple studies and proved in practice [11, 12, 13, 14, 15, 16, 17]. Using efficient individual additives (superplasticizing additives, based on polycarboxylate and polyglycol, active pozzolanic additives – microsilica, dehydrated kaolin and finely-dispersed ashes, etc.) and their systems has become a key to solving many process tasks of the concrete science, including manufacture of modified cement composites of increased strength from self-compacting mixtures (SCC) [6, 18, 19, 20].

The term "self-compacting concrete" (SCC, English, Selbstverdichtender Beton (SVB), German, Beton autoplacant (BAP), French) suggested in 1986 by the Japanese Professor Okamura [18] combines concrete mixtures with high workability (slump cone flow above 55-60 cm with a reduced ce-
ment-water ratio down to 0.35-0.4 and less) caused by high deformability of the suspension matrix along with its high resistance to segregation and lamination in displacement [6].

2. Formulation of the problem
A distinctive feature of the formulation of self-compacting heavy or fine-grained concretes with coarse sands is an increased content of rheologically active components, the amount of which in different concretes varies from 40 to 50% [18, 19, 20]. Increasing the share of water-dispersive suspension is achieved by introducing fine powders of rocks of sedimentary, volcanic and metamorphic origin in the amount of up to 100÷150% of the cement weight that are compatible with known plasticizers and are rheologically active in mixture with cement [21] (limestone, marble, dolomite, shredded quartz sand, etc.). However, the issues of manufacturing self-compacting fine-grained concrete mixtures using widely spread fine natural quartz sands with the fineness modulus below 2.0 are almost not investigated, especially in low-cement mixtures with the consumption of the binder up to 400 kg/m³, self-compacting of which cannot be achieved with minimal water and no lamination for the defined filling extent of cement systems due to high inter-grain cavernosity and water demand of the given fine aggregates, as well as low efficiency of plasticizers in such mixtures irrespective of their dosage. The described complications require scientific and theoretical basis to be developed to increase the plasticizing and water-reducing efficiency of modern plasticizers in concrete mixtures based on fine water-demanding sands as well as recommendations for optimal dispersion and concentration of mineral aggregates in such systems to achieve their high mobility with minimal water of mixing.

3. Problem solution
The defined research aim has been reached by solving the following tasks:

- defining the optimal dosage of a polycarboxylate superplasticizer and rheologically active mineral filler in cement-mineral suspensions when developing self-compacting fine-grained concrete mixtures;
- studying the water demand of fine natural quartz sands using the methodology of Skramtayev and Bazhenov;
- developing compositions of self-compacting fine-grained concrete mixtures of workability class SF1 with fine natural quartz sand;
- studying the physical and mechanical characteristics of fine-grained concretes from self-compacting mixtures.

4. Results and discussion

4.1. Defining the optimal dosage of polycarboxylate superplasticizer and rheologically active mineral filler in cement-mineral suspensions
To prepare suspensions, Portland cement CEM I 32.5R (C) by Mordovtsement PJSC was used. The mineral part included a carbonate filler from KM100 microcalcite (MKM) by Polipark LLC with the dosage of 0÷300% of Portland cement weight (0÷75% of solid phase weight) with the variability pitch of 100%. Melflux 5581 F (SP) polycarboxylate superplasticizer by BASF Construction Solutions (Trostberg, Germany) was used as a plasticizer.

The study was carried out with the fixed water-solid ratio W/S = 0.15 with the varying factors being:

- ratio MKM /C, \( x_1 = 0÷3.0 \) relative units;
- ratio SP/(C + MKM), \( x_2 = 0÷1.5\% \).

The study results were used to develop an experimentally statistical (ES) model describing the changes in the flow diameter of cement-carbonate suspensions (Portland cement + microcalcite) [mm] from the Hegermann cone from the content of the varying factors \( x_1 \) and \( x_2 \). ES-model was set as polynome being indicated below
\[ y = 288.9 + 52.9 \cdot x_1 + 64.8 \cdot x_2 + 19.8 \cdot x_1 \cdot x_2 - 51.33 \cdot x_1^2 - 89.3 \cdot x_2^2 + 37.97 \cdot x_1^3 + 32.34 \cdot x_2^2 - 59.06 \cdot x_1 \cdot x_2^2 - 15.19 \cdot x_1^2 \cdot x_2 + 31.64 \cdot x_1^2 \cdot x_2^2, \]  

(1)

where \( x_1 \), \( x_2 \) are the values of variable factors.

Using the numerical values of the coefficients of the polynomial (1), isolines have been built, which reflect the changes in the flow diameter of cement-carbonate suspensions from the Hegermann cone depending on the content of microcalcite and Melflux 5581 F superplasticizer (Figure 1). It has been found that for the constant water/solid ratio of \( W/S = 0.15 \) relative units, an increase in the dosage of the superplasticizer and the mineral filler (microcalcite) causes a significant increase in the flow diameter of the cement-mineral suspension.

The analysis of the ES-model (1) and Figure 1 found the optimal levels of the varying factors that allow reaching the self-compacting of suspensions for the flow diameter from the Hegermann cone above 280 mm and the water/solid ratio of 0.15 relative units: the dosage of Melflux 5581 F superplasticizer is 0.5÷1.0% of the solid phase weight; the filling degree of suspension with microcalcite is at least 105% of the Portland cement weight. An increase in the plasticizer content to 1.5% of the Portland cement weight does not cause a significant growth of the rheo-technological parameters. Reaching the self-compacting of the dispersive system with reduced dosages of the plasticizer equal to 0.45÷0.6% of the solid phase weight is possible for the aggregate content above 180% of the Portland cement weight.

4.2. Studying the water demand of fine natural quartz sands

The water demand of fine and large natural quartz sands was defined upon the methodology of Skramtayev and Bazhenov [22] based on selecting the mixtures of the same mobility with comparative tests of the cement paste and mortar with a fine aggregate. To find the water demand of sand \( (W_s) \), the at the initial stage water content of the cement paste was found \((W/C)_c\), for which it has the flow diameter from the Hegermann cone of about 170 mm, which corresponds to normal cement paste density. The water-cement ratio of the mortar was then found for the composition of sand/cement = 1/2 \((W/C)_m\), for which it has the same flow diameter from the Hegermann cone (170 mm). The water demand of sand (%) was calculated under the formula:

\[ W_s = \frac{(W/C)_m - (W/C)_c}{2} \cdot 100\%. \]  

(2)
As a binder for the preparation of the cement paste and mortar, the above Portland cement CEM I 32.5R (C) was used with the normal cement paste density 26.5% and the water-cement ratio \((W / C)_{c} = 0.265\).

As a fine aggregate, the natural quartz sand of the Mordovia Novostepanovsky pit was used with the coarseness modulus \(M_{coar} = 1.6\) and the content of pulverulent and clay particles of 1.6% (Russian GOSTs 8735 and 8736). As a result of sand fractioning and selecting fraction below 0.63 mm, the coarseness modulus changes within \(M_{coar} = 1.4 ÷ 1.6\). In the study, this type of a fine aggregate was used in two versions: as a selected fraction below 0.63 mm with \(M_{coar} = 1.4\) and average grain size \(d_{av} = 0.23\) mm (S1); in natural occurrence with the grain size below 5 mm, \(d_{av} = 0.29\) mm and \(M_{coar} = 1.6\) (S2).

The results of calculating the water demand of the studied types of sand are given in Table 1.

| Type of applied sand | Marking of sand | Dispersivity and coarseness modulus of sand | \((W / C)_{m}\), relative units | \(W_s\), % |
|----------------------|-----------------|------------------------------------------|-------------------------------|----------|
| natural quartz sand  | S1              | fraction below 0.63 mm, \(d_{av} = 0.23\) mm and \(M_{coar} = 1.4\) | 0.430                         | 8.3      |
| of the Mordovia      |                 | natural occurrence with the grain size   |                               |          |
| Novostepanovsky pit | S2              | below 5 mm, \(d_{av} = 0.29\) mm, \(M_{coar} = 1.6\) | 0.413                         | 7.4      |

4.3. Developing of compositions of self-compacted fine-grained concrete mixtures with fine natural quartz sand

The study results of the water demand of sands and rheological efficiency of formulations of cement-carbonate suspensions were used to develop self-compacting fine-grained concrete mixtures of workability class SF1 (the flow diameter from the Hegermann cone nevertheless 260÷280 mm, slump and flow of the standard Adams cone above 25÷25.5 cm and 500÷550 mm, respectively) with fine natural sand S1 (Table 1), consumption of Portland cement CEM I 32.5R (C) within 239÷739 kg/m³ of the concrete mixture, MKM shares – 45÷321% of Portland cement weight, SP Melflux 5581 F and water contents – 0.7% and 0.202÷0.218 relative units of the mixture \((C + MKM)\), respectively. These high-mobility mixtures are highly filled systems, characterized by an increased content of fine rheologically active components \((C + MKM)\) and rheologically active suspension \((C + MKM + mineral additive + water): 340÷360 and 580÷600 l/m³ of the concrete mixture, respectively, ensuring a sufficient interlayer between particles of fine sand.

To increase the dispersive phase of the binder, in addition to microcalcite, three types of mineral additives were used: condensed non-compacted microsilica MC-85 (MC) from Kuznetskiye Ferrosplavy JSC; highly active white metakaolin (HAWM) from Meta-D LLC; integral crystalline waterproofing additive Penetron Admix (Admix) for concrete mixture from Penetron Waterproofing Factory LLC.

4.4. Studying the physical and mechanical properties of fine-grained concretes from highly filled self-compacting mixtures

At the final stage, 7 compositions of highly filled self-compacting fine-grained concrete mixtures with fine natural quartz sand S1 were selected (Table 2) for experimental studies of the physical and mechanical properties of modified cement composites: density in normal humidity conditions at the age of 28 days, bending strength and compressive strength (cube strength) at the age of 1, 7 and 28 days. The research results are given in Table 2 and Figure 2.
Table 2. Compositions, physical and technical properties of modified fine-grained concretes from highly filled self-compacting mixtures with fine sand S1.

| Components | Prescription and technological parameters | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|------------|------------------------------------------|---|---|---|---|---|---|---|
| MKM / C, relative units | 0.45 | 0.45 | 0.45 | 0.45 | 1.11 | 1.63 | 3.21 |
| C, kg/m³ | 701÷739 | 239÷485 |
| S1 / C, relative units | 1.50 | 1.50 | 1.50 | 1.50 | 2.23 | 2.92 | 4.68 |
| Additive (A), % of weight (C + A) | - | 10 | 10 | 10 | 10 | - | - |
| SP, % of weight (C + MKM) | 0.7 | 0.7 | 0.7 | 0.7 | 0.7 | 0.7 | 0.7 |
| W / C, relative units | 0.29 | 0.30 | 0.32 | 0.32 | 0.44 | 0.57 | 0.89 |
| Flow diameter from the Hegermann cone, mm | 295 | 260 | 280 | 280 | 260 | 290 | 300 |

Physical and mechanical properties at the age of 28 days

| Density, kg/m³ | 2393 | 2343 | 2320 | 2367 | 2320 | 2323 | 2344 |
| Bending strength, MPa | 12.3 | 12.8 | 10.6 | 14.9 | 9.9 | 7.4 | 5.3 |
| Compressive strength, MPa | 92.5 | 104.2 | 82.7 | 115.8 | 73.8 | 47.5 | 28.3 |

Experimental studies found high densities of cement composites made from highly filled self-compacting mixtures with sand S1 amounting to 2320÷2393 kg/m³ (Table 2). Strength at the age of 28 days for the studied modified fine-grained concretes with the Portland cement consumption of 239÷739 kg/m³ of the concrete mixture vary within a wide range: bending strength – 5.3÷14.9 MPa; compressive strength – 28.3÷115.8 MPa.

Figure 2. Kinetics of growth of bending (a) and compressive (b) strength of compositions of modified fine-grained concretes from highly filled self-compacting mixtures using fine sand S1.
The studied compositions of modified cement composites from highly filled self-compacting mixtures differ in high rates of strength gain: the bending strength at the age of 1 and 7 days, respectively, was $28\div66$ and $75\div86\%$, and the compressive strength at the age of 1 and 7 days was $28\div44$ and $71\div81\%$ of the design strength at the age of 28 days (Figure 2).

The data analysis of Figure 2 and Table 2 found an opportunity to produce highly strong fine-grained concretes with no active mineral additives with the Portland cement consumption of 739 kg/m$^3$ of the concrete mixture: composition No. 1 with the ultimate bending and compressive strength at the age of 28 days: 92.5 and 12.3 MPa, respectively.

By studying groups of cement composites with increased consumption of Portland cement ($701\div739$ kg/m$^3$ of the concrete mixture), it has been found that introducing active mineral additives of condensed non-compact microsilica (MC) and highly active white metakaolin (HAWM) into the formulation results in the increased strength of composites. In particular, the strength characteristics of fine-grained concretes with MC and HAWM of compositions Nos. 2 and 4 are higher than the similar indicators of the control composite No. 1 non-modified with mineral additives with almost the same consumptions of Portland cement: up to 21 and 25% for bending strength and compressive strength, respectively.

Introducing Admix (composition No. 3) into the formulation promotes some decrease in the strength of fine-grained concretes at the design age (28 days) as compared to composites of the control composition No. 1 with almost the same consumptions of Portland cement: by 14 and 11% for bending strength and compressive strength, respectively. This effect can be explained by a somewhat increased water demand of fine-grained concrete mixtures with this mineral additive (Table 2) and the specifics of the phase composition of composites produced on their basis.

5. Conclusion
The results of experimental studies were used to develop compositions of modified fine-grained concretes of compression strength classes from C16/20 to C80/95 made of highly filled self-compacting mixtures with the Portland cement consumption of $239\div739$ kg/m$^3$ and the water content of $212\div226$ l/m$^3$ of the concrete mixture, including high strength concretes of class C45/55 and higher with the Portland cement consumption of $485\div739$ kg/m$^3$ of the concrete mixture, including carbonate filler, Melflux 5581 F superplasticizer, active mineral additives (if necessary), fine natural quartz sand with the coarseness modulus of 1.4. These cement composites are characterized by the following quality indicators: density in normal humidity conditions at the age of 28 days $2320\div2393$ kg/m$^3$; bending and compressive strength at the age of 28 days $5.3\div14.9$ and $28.3\div115.8$ MPa, respectively. Mass use of such materials forms pre-requisites to implement into the production complex resource-saving, energy-saving and more environmentally friendly technologies by reducing the buildup of material and energy-consuming production of Portland cement, reducing emissions of harmful gases to the atmosphere, decreasing the energy and fuel consumption for laying and forming concrete mixtures, reducing the volumes of automobile and railroad transportation of raw materials and replacement of expensive imported aggregates with cheap local sands.

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