Experimental investigation of the influence of the plasticizing agent on the properties of cement mortars

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Abstract. Negative properties of concrete include the volume changes occurring during their solidification and hardening process. These deformations and cracks lead to reduce the strength and the durability of structures. The most important volume change of concrete is shrinkage resulting from the ongoing physical and chemical processes. The concrete with a high water to cement ratio is especially prone on the drying shrinkage. This article deals with the verification of the properties of cement mortars using selected kinds of plasticizing agents developed for self-levelling cement screeds. Applied agents (MS 3-12, MS 4-12, MS 5-12 and reference Berascreed 101) are based on modified polycarboxylates. The dosage of these agents was determined by the previous research in the amount of 1.5\% by weight of the binder. The use of agents had a positive impact on the consistency of the fresh cement mortars and increased values of the initial and the long-term compressive strength. The negative impact of these agents was noted at the shrinkage and bulk densities of fresh and hardened cement mortars. Based on the achieved results can be concluded, that the plasticizing agent MS 4-12 may be considered a suitable substitute for the reference agent.

1. Introduction

Cement composites, especially concretes are currently the most widely used building materials in the world. The resulting concrete properties in plastic or hardened state are influenced not only by the concrete composition itself, i.e. properties of cement (mineralogical composition, quantity and fineness of grinding), water to cement ratio, properties of aggregates (dosage, granulometry), properties of additives (kind, dosage and composition), as well by a method for dosing of the individual components, mixing them, transport and compacting of fresh concrete and especially by the treatment method.

The origins of cement composites are connected with the knowledge of technology of hydraulic binders. Composites development progressed rapidly and reflecting the increasing demands of civil engineers on the building materials in the implementation of increasingly demanding constructions and often in very complicated conditions\[7\].

Self-Compacting Concrete (SCC) passed shortly rapid development since the creation of the concept of desired characteristics to the normal use of the free market construction work\[7\]. The important progress in the development of concrete with bigger fluidity occurred in Japan in the 80 years of the twenty century, the first significant applications were realized early the 90s. It is characterized by very good workability, stability and texture uniformity and resistance to component segregation. The hardened SCC generally achieves higher strength and durability compared to
conventional concrete [4, 5]. The desirable properties of fresh SCC such as e.g. fluidity, low blocking flow between the reinforcement, the resistance to agitation, and segregation, sweating, good pump ability and mobility can be achieved by suitable composition [7]. The mobility of the concrete is ensured by the addition of a plasticizer (super plasticizer).

Shrinkage may be a serious problem, even if self-leveling cement screeds in which the water to cement ratio is relatively high, even when using high-quality super plasticizers. The basic technological measure for its reduction is the treatment of fresh composite. Often times, the correct method of treating or reducing the water to cement ratio using appropriate additives, do not have to be the prevention of crack formation [10]. This applies especially in the large-scale structures (screeds, floors, ceilings), where there is a very high risk of excessive evaporation of water from the structure and the subsequent formation of shrinkage cracks.

The volume changes of concrete and other cement composites occur as a result of physical and chemical processes in the cement matrix. Reduction of volume causes tensions that may result in cracks or other deformations. We distinguish several kinds of shrinkage. The shrinkage associated with the changes of the water content (moisture content) in the composite structure may be a plastic, which occurs at the initial stages of hydration, or so-called drying shrinkage manifested in composites in hardened condition [9].

A special kind of shrinkage is carbonation shrinkage that occurs in the surface layer of the composite in the long-term reaction of atmospheric carbon dioxide with cement components. The value of total shrinkage of concrete ranges from 0.3‰ to 0.7‰ and mortars shrinkage from 0.7‰ to 1.5‰ [8]. There are many ways of reducing the shrinkage of cement composites. The one of the ways is suitable reinforcement of concrete. Different types of dispersed fibres can be used to eliminating the effects of shrinkage of self-leveling cement screeds [3]. The next option is to use such an additive inorganic material as the fly ash and the shrinkage reducing agent (SRA) [2, 6].

Plasticizing agents are substances that affect the dosage of water. They can reduce the amount of mixing water with unchanged consistency or contrariwise, at the same dosage of water are able to improve the workability, i.e. the consistency of fresh concrete. Reducing the water to cement ratio has the effect on increasing the compressive strength and improving hardened concrete properties. Reducing the water to cement ratio using plasticizing agents leads to a significant reduction in shrinkage and creep of the concrete. The application of plasticizing agents with constant water to cement ratio has not significant influence on the shrinkage of concrete. Special kind of plasticizing agents are superplasticizers characterized by a very strong fluidity effect. They significantly affect the consistency of fresh concrete. Increasing dosage of plasticizing agents results in a lower consistency (better workability). The dosage of agents ranges from 0.6 to 1% by weight of agents solutions from the weight of cement. Superplasticizers have not direct impact on the shrinkage, creep or durability of concrete [1].

2. Materials and methodology

2.1. Materials

Cement CEM II A/S 42.5 R, in according with STN EN 197-1 [15], with following properties: compressive strength after 2 days 21.6 MPa, after 28 days 49.7 MPa, initial setting time 200 min, setting time 250 min and soundness (expansion) by Le Chatelier 7.2 mm.

Aggregate. Natural quarried aggregate meeting the requirements of STN EN 13139 [11] divided by sieve analysis according to STN EN 12620+A1 [12] on fraction 0/1, 1/2 and 2/4 mm.

Fly ash is characterized as the grey powder with apparent density 700 to 900 kg.m⁻³; activity index after 28 days min 75%; activity index after 90 days min 85% and other characteristics declared by producer. The chemical compositions of fly ash (wt. %) are: SiO₂ (38.76); Al₂O₃ (16.08); Fe₂O₃ (3.21); CaO (21.28); MgO (1.98); TiO₂ (0.51); SO₃ (13.99).

Water supplied by the public water supply company was used in the mixtures, in according with STN EN 1008 [13]. All cement mortars had w/c ratio = 0.55.
Plasticizing agents. To improve the flow ability of the cement mortars were applied four kinds of super plasticizing agents based on modified polycarboxylates, which differ from each other by the type (molecule) of polycarboxylate. The agent Berascreed 101, characterized as the yellow-brown homogeneous solution by density at 20°C 1 070 kg.m⁻³; pH value at 20°C 6.7±0.5; dry matter content 24% ± 1.2%, was used for reference mortar (labelled as REF). The agent MS 4-12 was used for mixture labelled as P1; for mixture labelled as P2: MS 3-12 and for mixture labelled as P3: MS 5-12. These agents have the same properties declared by producer: density at 20 °C 1 060 kg.m⁻³±20 kg.m⁻³; pH value at 20°C 6.0±2.0; dry matter content 20%±1%; chloride content max 0.10% by weight of agent. Furthermore, the stabilizing agent BR stabilizer 101 with density 1 026 kg.m⁻³, 3.6% dry weight and pH 9.5 was used to reduce the segregation of the cement mortars.

2.2. Mixture design
The design of cement mortar composition was based on commercially produced self-levelling cement screeds (mortars). The general composition of cement mortar is given in table 1. The composition of the mortars were each differed only in the kind of plasticizing agents, wherein the dose was estimated from previous research in an amount of 1.5% by weight of the binder for super plasticizing agent and 1.6% of the weight of the binder for stabilizer.

| Component   | Dosage (kg m⁻³) |
|-------------|-----------------|
| Cement      | 243.15          |
| Fly ash     | 154.00          |
| Aggregate   |                 |
| - fraction 0/1 | 518.72         |
| - fraction 1/2 | 207.49         |
| - fraction 2/4 | 311.23         |
| Agent       |                 |
| - Plasticizing | 6.00           |
| - Stabilizing | 6.24           |
| Water (w/c=0.55) | 218.84     |

2.3. Measuring methods
Cement mortars were mixed according to STN EN 196-1 [14] in a normalized laboratory mixer. After that, the consistency and the bulk density of the fresh cement mortars were determined. The consistency was determined by a flow test on the Haegermann flow table as the average value of two perpendicular measurements of the cake diameter resulting from the spill of mortar after lifting the mould. Then was followed the change in the consistency of the mortar with a time of maturing. The consistency was monitored every 30 minutes from the completion of mixing of the mortar to the time, that the mortar do not longer show signs of flow ability, i.e. it was not possible to measure its spill.

The test samples to determine compressive strength were cylindrical with diameter and heights of 30 mm and were cured in humid environment. The compressive strength of hardened mortars was measured after 2, 28 and 90 days. The test samples to determine shrinkage were beams of 40 x 40 x 160 mm with glass contacts in front. They were cured for 24 hours in humid environment. After this time, samples were unmoulded and the initial length measurements were done. Subsequently, the samples were kept in a laboratory environment (temperature 20 °C±5 °C, relative air humidity φ = ca 50%). The drying shrinkage of cement mortars was determined as loss of length compared to the initial length determined after 24 hours of curing in moist environment. Shrinkage was measured after 2, 4, 7, 14, 28, 56 and 90 days of curing in the laboratory environment. The length measurement was done using the Graf-Kaufmann device. The weight loss was measured on the same samples used for the shrinkage measurements.
3. Results and discussion
Achieved results of monitored parameters of fresh and hardened cement mortars are given in table 2.

The application of four kinds of plasticizing agents had various impacts on physical and mechanical properties of cement mortars, which can be seen in figure 1 to 3.

|        | Physical and mechanical properties of testing cement mortars. |
|--------|---------------------------------------------------------------|
|        | Fresh cement mortars                                         | Hardened cement mortars |
|        | Bulk density (kg m\(^{-3}\))                                 | Bulk density (kg m\(^{-3}\)) | Compressive strength (MPa) |
|        | Consistency (mm)                                              | 2 days | 28 days | 90 days | 2 days | 28 days | 90 days |
| REF    | 2 030                                                        | 1 960  | 1 920   | 1 920   | 6.77   | 19.26   | 20.80   |
| P1     | 2 120                                                        | 2 160  | 2 010   | 1 990   | 9.85   | 24.38   | 26.00   |
| P2     | 2 070                                                        | 2 170  | 2 010   | 2 030   | 7.55   | 17.80   | 23.10   |
| P3     | 2 080                                                        | 2 125  | 1 980   | 1 970   | 8.86   | 21.64   | 25.55   |

3.1. Consistency
The use of plasticizing agents resulted in a high fluidity of cement mortars. Flow diameter ranged from 229.5 mm to 282.5 mm. The most liquid initial consistency was achieved using the MS 4-12 plasticizer (P1). Its value was higher by about 2.7% compared to the reference mortar.

The consistency of cement mortars were gradually changed with a time of their maturing. All of the observed mortars showed even bigger fluidity during the first 60 minutes. The further course of the change in consistency depended on the type of used plasticizer, which was resulted to different rates of loss of workability (Figure 1). The fastest loss of workability was achieved for reference mortar after about 210 minutes. Mortars tested for new plasticizing agents were still workable after 4 hours. Mortar with the plasticizing agent MS 5-12 (P3) has maintained the longest ability for workability.

![Figure 1. The change in the consistency of the cement mortar with a time of maturing.](image-url)
3.2. Bulk density and weight loss
The values of density of fresh mortar were almost identical to the reference mortar, which reached a value of 2030 kg.m\(^{-3}\). Some difference was observed for sample P1, whose bulk density was 2120 kg.m\(^{-3}\). Bulk density of hardened cement mortars was gradually declined with a time of keeping the samples in humid environment. The reference mortar reached the lowest value of the bulk density, i.e. 1920 kg.m\(^{-3}\) (90 days of hardening). The values of bulk density of cement mortars with new plasticizing agents in the same reference period ranged from 1970 kg.m\(^{-3}\) to 2030 kg.m\(^{-3}\).

Volume changes in cement mortar had started to show from the start of drying. The biggest weight loss occurred in the first days of setting and subsequent hardening of the mortars. After 28 days of hardening, there was observed only a minimal weight change of mortars and after 56 days the weight has not changed practically. The biggest weight loss compared to the reference mortar was reached on the cement mortar (P3), i.e. 8.8%.

3.3. Shrinkage
Course of shrinkage of cement mortar (Fig. 2) corresponded with the weight loss of the sample. The biggest shrinkage was reflected in the first days, gradually decreased and after 28 days was changed minimally. The weight of reference mortar increased in the first days, i.e. there was a swelling and not shrinkage of the mortar. The overall lowest shrinkage was found just at the reference mortar, i.e. 0.66‰ after 90 days of hardening. The lowest shrinkage values of the new tested plasticizing agents reached cement mortar P1 with a value of 0.76‰ (90 days).

3.4. Compressive strength
The influence of plasticizing agents on the compressive strength of cement mortars can be seen in figure 3. Using these agents had only a minimal effect on an increase the initial (2-day) and the long term (28 and 90 days) compressive strength of cement mortars compared to the reference mortar. The resulting values of compressive strength in the monitoring period ranged from 7.55 MPa to 26.00 MPa. A positive increase of strength characteristics of testing mortars was observed by the application...
plasticizer MS 4-12 (P1). Due to the achieved lower compressive strengths, the plasticizing agent MS 3-12 (P2) appears to be the least suitable.

![Figure 3](image)

**Figure 3.** The compressive strength of cement mortars in the application of four kinds of plasticizing agent.

4. Conclusion

Based on the results of the experiments can be concluded that the use of new types of plasticizing agents had only a slightly different effect on the observed properties of cement mortars compared to the reference additive.

Application of plasticizing agents resulted in a high fluidity of cement mortars. Flow diameter of testing mortars ranged from 229.5 mm to 282.5 mm. In the case of use the plasticizer MS 4-12, the initial consistency was slightly improved, the effectiveness of other ingredients (MS 3-12 and MS 5-12) was lower. Changing the mortars consistency with time of maturing was during 210 minutes almost identical in all cases. Another course of the change in consistency depended on the kind of used plasticizing agent, which was resulted to different rates of loss of workability. Mortar with the plasticizer MS 5-12 has maintained the longest ability for workability.

The values of fresh mortar’s densities were almost identical with the reference mortar, which reached a value of 2030 kg.m⁻³. Some difference was observed on mortar with plasticizer MS 4-12, whose bulk density was 2120 kg.m⁻³. Bulk density of hardened cement mortars was gradually declined with a time of keeping the samples in the humid environment. The reference mortar reached the lowest value of the bulk density, i.e. 1920 kg.m⁻³ (90 days). The values of bulk density of cement mortars with new plasticizing agents in the same reference period ranged from 1970 kg.m⁻³ to 2030 kg.m⁻³.

The biggest shrinkage of mortars was reflected in the first days. It was decreased gradually and after 28 days was changed minimally. The overall lowest shrinkage was observed at the reference mortar, i.e. 0.66‰ after 90 days. The lowest shrinkage values of new plasticizing agents reached a mortar with the plasticizer MS 4-12, i.e. 0.76‰ (90 days).

The results showed a positive effect of the tested plasticizing agents on the initial and the long-term compressive strength of mortars. All new plasticizers resulted to higher values of compressive strength as a reference sample. The one exception is mortar P2 (plasticizer MS 3-12) at the age of 28 days, the strength value of which was higher as a reference sample. However, this difference was minimal. The compressive strength values ranged from 7.55 MPa to 9.85 MPa after 2 days of hardening and from 23.10 MPa to 26.00 MPa after 90 days of hardening.
Due to the achieved results, the plasticizer MS 4-12 seems to be a suitable replacement for plasticizing agent Berascreeed 101.

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