

Development of shrinkage and fracture parameters in selected fine-grained cement-based composites

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Abstract. The paper summarizes results of a pilot study aimed at the evaluation of an experimental investigation focused on determination of the material characteristics development of selected fine-grained cement-based composites during their ageing. The composition of composites being investigated differed only in a water to cement (w/c) ratio and in amount of superplasticizer. Quite extensive experiments were performed with the aim to determine shrinkage, dynamic a static modulus of elasticity and fracture properties on test specimens exposed to free drying during the whole time of its ageing (including the early stage of setting and hardening). The article presents especially results (including their statistical evaluation) of shrinkage and fracture parameters development within 90 days of composites' ageing. Experimental results show the dependence of the investigated characteristics on the value of w/c ratio. The most visible effect was observed in the case of shrinkage development. The curing conditions were reflected especially in high variability of the test results.

1 Introduction

Cement-based composites are one of the most frequently used materials in building practice. In order to achieve even better physical and mechanical characteristics, the composition of the cement-based composites is continuously developing. The main aim of this effort is to obtain a building material with a high quality and high durability.

One of the most commonly monitored characteristics is their resistance to the formation and propagation of cracks during whole time of ageing. Generally speaking, the first source of cracking are volume changes which take place in the early stage of setting and hardening. Shortly after the pouring of fresh mixture into the moulds plastic shrinkage occurs. Its magnitude strongly depends on the ambient conditions and appropriate curing conditions have to be maintained to eliminate the cracking. Other substantial source of early cracking, mainly in composites with a low water to cement (w/c) ratio, can be also the occurrence of autogenous shrinkage especially when it is restrained [1, 2]. However, the major causes of shrinkage cracking are probably deformations due to drying which start after the removal the final structural element from the mould and appropriate curing conditions are not

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maintained [3, 4]. The above mentioned sources of cracking may be causes of decrease in strength as well as durability of structural members [5, 6].

To quantify degree of material deterioration, an appropriate approach has to be applied. Commonly used tests for determination of materials' strength parameters are not sufficient to describe the actual degree of their inner structure deterioration. In this case, more suitable approaches are those based on the quantification of the fracture parameters which are strongly dependent on the damage of macro- and micro-bonds in the inner structure of the tested material [7].

2 Experimental part

2.1 Materials

The test specimens used in the experiment were manufactured from three different fine-grained cement-based mixtures (A, B and C). The composition of the investigated composites differed only in a w/c ratio and in an amount of superplasticizer, rest of components was always the same. A design of mix proportions originates from the standard ČSN EN 196 - 1 [8]. The fresh composites were made using the standardized quartzite sand (s) with maximum nominal grain size of 2 mm, CEM I 42.5 R Portland cement (c) and water (w) at a proportions mentioned in Table. 1. "C" mixture contains also polycarboxylate ether-based superplasticizer SIKA SVC 4035 in an amount of 1% by cement mass. A mixing device with controllable mixing speed was used to prepare the fresh mixtures. Basic information about mix proportions and parameters of fresh-state composites are shown in Table 1. The workability was determined according to ČSN EN 1015-3 [9] using the flow table with a calibrated scale designed for the testing of fresh mortars. Bulk density of the fresh composite was determined using a calibrated vessel with the volume of 1 dm³ in accordance with ČSN EN 1015-6 [10].

Table 1. Mix proportion and properties of fresh concrete.

	Units	Composite A	Composite B	Composite C
$s:c:w$	–	3:1:0.5	3:1:0.47	3:1:0.35
Plasticizer	% by cement mass	–	–	1.0
w/c ratio	–	0.5	0.47	0.35
Mixing speed	revolutions/min	20	30	40
Workability	mm	145	120	130
Bulk density	kg/m ³	2200	2230	2270

Two groups of test specimens were prepared from each mixture. The first group contained test specimens with dimensions of 100×60×1000 mm intended especially for

measurement of shrinkage development (6 specimen from each mixture). The second group contained test specimens with dimensions of 40×40×160 mm which were subjected, among others, to fracture tests (12 specimen from each mixture).

2.2 Test procedures

2.2.1 Shrinkage measurement

The test procedure intended for determination of shrinkage development was designed to measure relative length changes along the central axis of the test specimens placed in the moulds with dimensions of 100×60×1000 mm. The measurement was performed using an inductive sensor leaning against the movable head of the mould whereas the head on the other side was fixed. The measurement started approx. 1 hour after the fresh composite was placed into the moulds. In this way the early relative length changes (up to ca. 72 hours of specimens' ageing) were recorded. Once early measurement was finished, the specimens were extracted from the moulds, placed on the table, and left to dry freely. The long-term measurement was performed using a strain gauge which was fixed onto the markers embedded into the upper surface of the test specimens during their manufacturing. The spacing of gauging points was 200 mm. Refer to [11] for more details about the test procedure.

All measurements were performed in a climate control chamber at a stable temperature of 21 ± 2 °C and relative humidity of $60 \pm 10\%$. The measurements were finished approx. at the specimens' age of 90 days.

2.2.2 Fracture parameters evaluation

The mechanical fracture parameters were determined from the records captured during the fracture test of beam specimens with a central edge notch in three-point bending. All specimens were subjected to the fracture test using the test machine with controllable speed of displacement increment which was, for the purpose of experiment, set to value of 0.02 mm/min. The record in form of load versus displacement (deflection in the middle of span length) diagrams were evaluated using the effective crack extension method [12] and work-of-fracture method [13]. The first (almost linear) part of the load versus displacement ($L-d$) diagrams were used for the determination of the elasticity modulus value. Because of stability loss during loading, it was not possible to reconstruct the descending part of $L-d$ diagrams. Therefore, the work of fracture value W_F^* is determined as area under $L-d$ diagrams before stability loss occurred. Refer to [14] for details about determination of above mentioned mechanical fracture parameters.

3 Results and discussion

The results of performed tests are introduced in following figures in form of boxplots. Six specimens for shrinkage and twelve specimens for the rest of investigated parameters were used in the experiment. Boxplots provide information about the distribution of obtained results. Boxplot shows median, 25th and 75th percentiles, and the range of data. The monitored materials' parameters were following: bulk density D , shrinkage ϵ , modulus of elasticity E_c , effective fracture toughness K_{Icc} and specific fracture energy G_F^* (determined using already mentioned work of fracture W_F^* value) and informative compressive strength f_c determined on the fragments of test beams after fracture test were finished.

Fig. 1 shows increase in values of bulk density of "C" composite which corresponds to the reduced value of a w/c ratio (see Table 1). This reduction was reflected in the development

of shrinkage (see Fig. 2) where the increased value of early shrinkage for “C” composite was recorded.

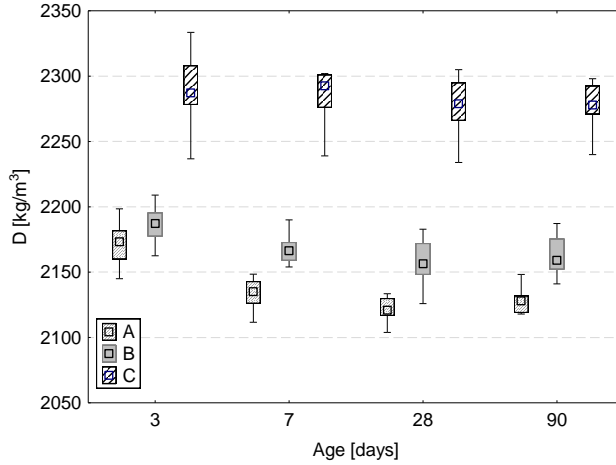


Fig. 1. Bulk density at 3, 7, 28 and 90 days of composites’ ageing.

This rapid increase affected also the final steady-state value of total shrinkage which was more than 2 times higher compared to “A” and “B” composites. Initial expansion of “A” and “B” composites was recorded within first 3 days of ageing. This expansion is connected with a rather high *w/c* ratio. “A” and “B” composites show very similar shrinkage process despite the reduced *w/c* ratio for “B” composite. This reduction was reflected slightly in the values of bulk density (Fig. 1).

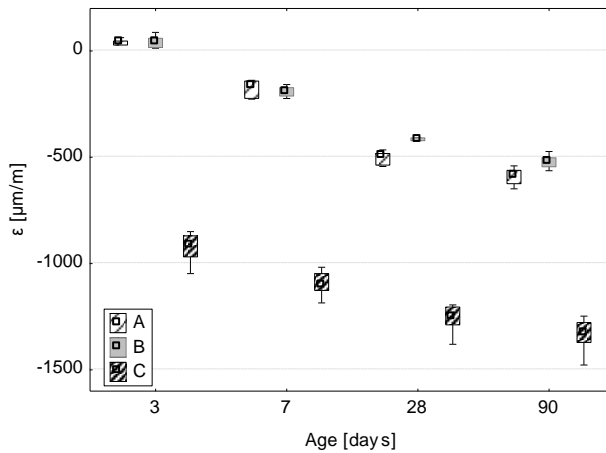


Fig. 2. Shrinkage values at 3, 7, 28 and 90 days of composites’ ageing.

Concerning the mechanical fracture parameters, it can be suggested that these increased values of shrinkage (for “C” composite) did not cause a sharp drop in any of monitored parameters (see Figs. 4–6). Note, that relatively high variability was recorded for all results of performed tests (including the values of informative compressive strength – see Fig. 3). The variability can be connected with the curing conditions. Note also that no special wet or water curing conditions were maintained during the ageing. All tested specimens (from Set “A”, “B” and “C”) were approx. 3 days in moulds while the upper surface was exposed to free drying. After removing from the moulds, the specimens were stored in a laboratory and

left to dry freely during the entire time of ageing. This fact may be reflected in magnitudes of mechanical parameters [6], [15] and also in variability of the tests results. Following aspects must be taken into account in relation to the results interpretation. First, the water needed to full cement hydration evaporated too early – a decrease in mechanical parameters can be expected. Second, there was a non-uniform distribution of water in volume of the particular test specimens during their ageing, especially within first 3 days the upper surface dried much more. Third, these “un-cured” composites are also more sensitive to micro-cracking. All above mentioned aspects are not always the cause of the decrease in absolute value of investigated parameters, but are reflected in the variability of results [16].

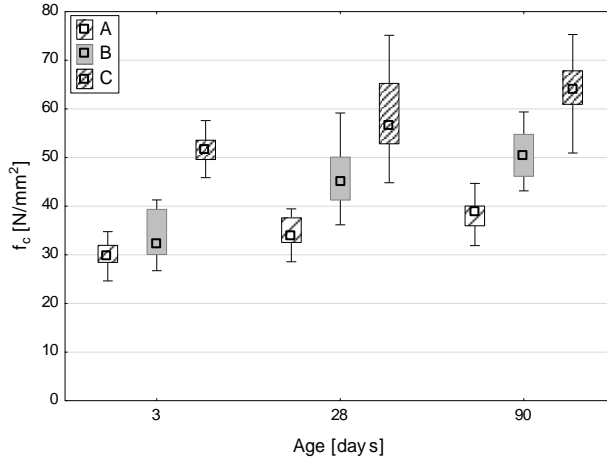


Fig. 3. Compressive strength at 3, 28 and 90 days of composites’ ageing.

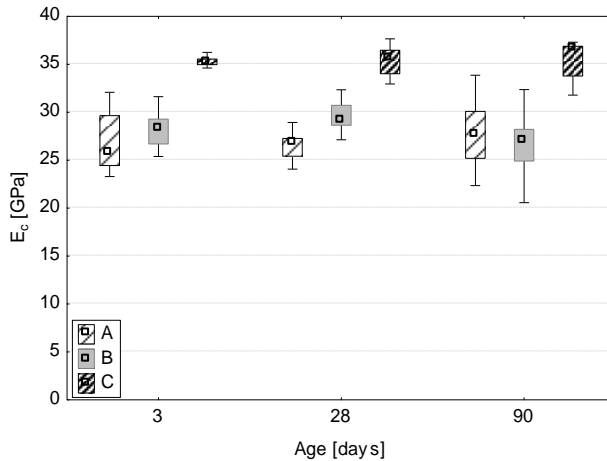


Fig. 4. Modulus of elasticity values at 3, 28 and 90 days of composites’ ageing.

The high variability of modulus of elasticity values is rather remarkable, especially in the case of “A” and “B” composites. This phenomenon may originate in composition and workability of fresh mixtures. In the case of “A” composite, bleeding was observed after pouring the moulds during the manufacturing of test specimens. In the case of “B” composite, the fresh mixture exhibited poor workability. Both these technological aspects can essentially influence overall development of physical and mechanical parameters of hardened

composite. Evaluation of the modulus of elasticity from ($L-d$) diagram recorded during the fracture tests can be more sensitive to these technological aspects.

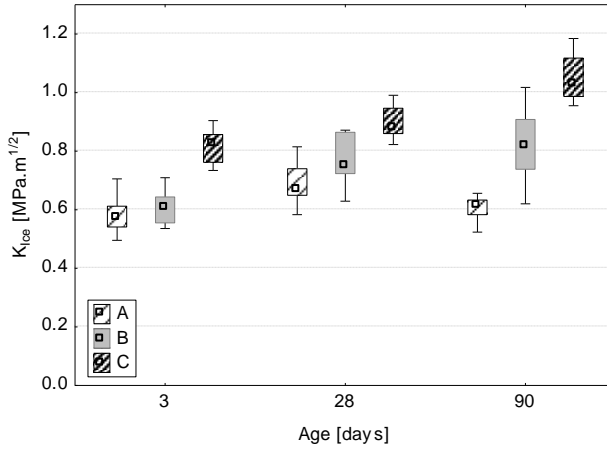


Fig. 5. Effective fracture toughness values at 3, 28 and 90 days of composites' ageing.

Fig. 5 and Fig. 6 show development of effective fracture toughness and specific fracture energy values. These two parameters should be most sensitive to materials' inhomogeneity and micro-cracks. A decrease of both fracture parameters was recorded at the age of 90 days for "A" composite (the one with the highest w/c). This decrease was not recorded in any other monitored mechanical parameters.

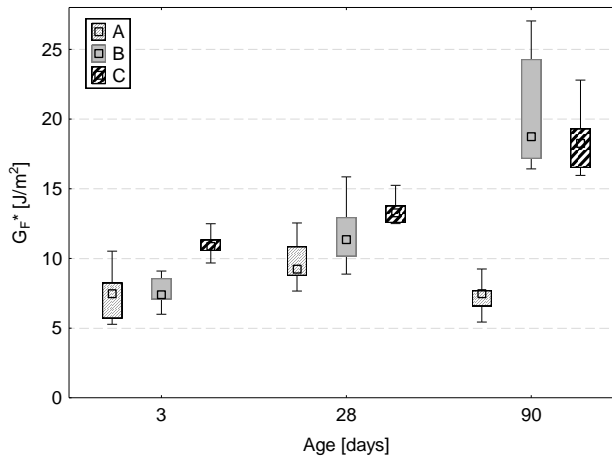


Fig. 6. Specific fracture energy values at 3, 28 and 90 days of composites' ageing.

4 Conclusion

The main aim of the performed experiment was to determine the development of shrinkage and fracture parameters for three cement-based composites which differed in the value of a w/c ratio and presence of plasticizer. To observe the influence of insufficient curing on the development of investigated parameters, all of the test specimens were exposed to free desiccation (drying) during whole time of their ageing, including the early stage of setting and hardening. The experimental results show that the actual value of a w/c ratio substantially

influences the magnitude and development of all investigated parameters. The most visible effect was observed in shrinkage development which was reflected in increased values of shrinkage during the whole time of ageing. The high shrinkage recorded at the age of 3 days (approx. 900 $\mu\text{m/m}$) for mixture "C" (the lowest w/c with addition of plasticizer) may lead to crack formation if it is restrained. The poor curing conditions were reflected especially in high variability of the test results and in the decrease of fracture parameters.

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References

1. P. Lura, O. M. Jensen, J. Weiss. *Mater Struct* **42**, 8 (2009)
2. L. Østergaard, PhD Thesis, Denmark, supervisor: L. Damkilde, H. Stang (2003)
3. L.Liu, X. Wang, H. Chen, Ch. Wan, M. Zhang, *Computers & Structures* **178** (2017)
4. P. Havlásek, PhD Thesis, Prague, supervisor: M. Jirásek (2014)
5. M. N. Hassoun, A. A. Al-Manaseer, *Structural concrete: theory and design*, 6th ed. (Wiley, USA, 2015)
6. P.-C. Aïtcin, *36th Conference on OUR WORLD IN CONCRETE & STRUCTURES*, <http://cipremier.com/100036002> [online], (Singapore, CI-Premier PTE LTD, 2011)
7. Z.P. Bažant, *5th International RILEM Symposium on Creep and Shrinkage of Concrete (ConCreep 5)*, [online], (E & FN Spon, London, 1993)
8. ČSN EN 196-1, *Determination of strength*, (Prague: ČNI, 2005)
9. ČSN EN 1015-3, *Determination of consistence of fresh mortar*, (Prague: ČNI, 2000)
10. ČSN EN 1015-6, *Determination of bulk density of fresh mortar*, (Prague: ČNI, 1999)
11. B. Kucharczyková, L. Topolář, P. Daněk, D. Kocáb, P. Misák, *Adv. Mater. Sci. Eng.* (2017)
12. B. L. Karihaloo, *Fracture mechanics and structural concrete* (Wiley, New York, 1995)
13. RILEM TC-50 FCM Recommendation, *Mater. Struct.* **18** (1985)
14. P. Rovnaník, H. Šimonová, L. Topolář, P. Bayer, P. Schmid, Z. Keršner, *Constr. Build. Mater.* **199** (2016)
15. D. Kocáb, M. Králíková, P. Cikrle, P. Misák, B. Kucharczyková, *Experimental Analysis of the Influence of Concrete Curing on the Development of its Elastic Modulus over Time*, *Mater. Technol.*, (MIT, Slovenia, to be published 2017)
16. P. C. Aïtcin, *High-Performance Concrete*, Czech version (IC ČKAIT, Prague, 2005)