
Barbara KUCHARCZYKOVÁ¹, Hana ŠIMONOVÁ², Zbyněk KERŠNER³, Dalibor KOCÁB⁴**SHRINKAGE, WEIGHT LOSS AND FRACTURE PARAMETERS OF SELECTED
POLYMER-MODIFIED CEMENT MORTARS DURING AGEING****Abstract**

Results of pilot experiments focused on monitoring of shrinkage, weight loss and mechanical fracture properties of two selected polymer-modified cement mortars (PCM) during their aging are presented in the paper. Comparison of the measurement results was carried out on the test specimens at the age of 3, 28, and 90 days. The results show that even the specimens made from PCMs, for which the manufacturer declares no shrinkage, shrank considerably. The results also show that the development of mechanical fracture parameters during ageing of these materials differs from generally expected trends and thus the monitoring of these parameters is recommended.

Keywords

Polymer-modified cement mortar, shrinkage, weight loss, modulus of elasticity, fracture toughness, fracture energy.

1 INTRODUCTION

The polymer-modified cement mortars/concretes are often used for the protection and rehabilitation of concrete structures in civil engineering [1]. These composites combine a cement matrix with an organic polymer matrix [2]. They are characterized by high adhesion to the base material, thixotropic properties, and high strength characteristics [3] and often it is declared that they do not evince shrinkage. Such mortars and concretes are commonly used for re-profiling of concrete structures and elements, adjusting of concrete surfaces, grouting and filling of cracks or bonding of special stainless helical reinforcement. Requirements for selection and characteristics of materials used for rehabilitation of structures are defined in the standard ČSN EN 1504, Part 1–10 [4]. The specific use and the technological process of the particular application are described in technical descriptions for individual products given by producers. Some procedures, for example degree of saturation of the base material and a treatment of the surface after application, are, in some cases, specified rather vaguely in these documents which could affect the development of physical and mechanical properties of the material and which can in turn endanger the functionality of the rehabilitation system [5].

2 EXPERIMENTS**2.1 Material, specimens and their maturing**

Two fine-grained polymer-modified cement mortars (PCM) based on Portland cement were chosen for this experiment. Since it was a pilot experiment aimed at monitoring of development of shrinkage and fracture characteristics over time, the trademark of used material is not published in the

¹ Ing. Barbara Kucharczyková, Ph.D., Brno University of Technology, Faculty of Civil Engineering, Veveří 331/95, 602 00 Brno, Czech Republic, phone: (+420) 541 147 527, e-mail: barbara.kucharczykova@vutbr.cz.

² Ing. Hana Šimonová, Ph.D., ditto, phone: (+420) 541 147 381, e-mail: simonova.h@vutbr.cz.

³ prof. Ing. Zbyněk Keršner, CSc., ditto, phone: (+420) 541 147 362, e-mail: kersner.z@fce.vutbr.cz.

⁴ Ing. Dalibor Kocáb, Ph.D., ditto, phone: (+420) 541 147 811, e-mail: dalibor.kocab@vutbr.cz.

paper. For the purposes of evaluation, the PCMs are designated as “V” and “VII” sets. The specimens of the “V” set were made of a two-component mixture wherein a liquid component was an aqueous copolymer dispersion and a powder component contained a mixture of Portland cements and mineral fillers. The specimens of the “VII” set were then made of a single-component powder material, containing among others silica sand, Portland cement, microfibers including plasticisers and polymers, which was mixed with the prescribed dosage of water to prepare the fresh mortar.

The hand-held mixer was used for the preparation of both PCM mortars. The production process, including the mixing time, was proceeded in accordance with the technical description for individual products given by respective producers. After a thorough homogenization of the fresh mixtures, 3 test specimens with a length of 1000 mm and a cross-section of 60×100 mm were made for monitoring of the development of shrinkage and 18 specimens with nominal dimensions $40 \times 40 \times 160$ mm were prepared for the determination of mechanical fracture parameters and informative compressive strength values. All performed measurements were for both PCMs evaluated at the age of 3, 28 and 90 days.

After the test specimens were manufactured, they were stored in the moulds for 72 hours. Then, they were removed from the moulds and stored under laboratory conditions, at a temperature of $(21 \pm 2)^\circ\text{C}$ and relative humidity of $(60 \pm 10)\%$. After the shrinkage values reached the steady state, ca. after 90 days of ageing, the beam specimens with nominal dimensions of $40 \times 40 \times 160$ mm were cut from specimens with length of 1000 mm. These specimens (labelled “R”) were also intended for fracture tests at the age of 90 days.

Note, that for the purpose of this experiment, all test specimens were not cured during whole time of ageing and their surface was intentionally left to dry freely.

2.2 Shrinkage, weight loss

The measurement of shrinkage was performed using a test device made by the company Schleibinger Geräte Teubert u. Greim GmbH [6]. This test device is primarily designed for shrinkage measurement in the early stage of cement composites setting and hardening. Special moulds of 1000 mm in length and with 60×100 mm in cross-section were used for monitoring the length changes measured along the central axis of the specimens using an inductivity sensor leaning against the movable head of the mould. In this manner, the relative length changes were record simultaneously during about 72 hours. The polyethylene foam mat (MIRELON) of 2 mm thickness was placed on the bottom and along both longitudinal sides of shrinkage moulds in order to ensure free movement of the specimen in the mould. The shrinkage moulds were placed onto a special weighing table [7] that allowed continuous recording of mass losses caused by free drying of the specimen surfaces.

Special markers were embedded into the upper surface of the composite placed in the shrinkage moulds in order to facilitate subsequent long-term measurement of relative deformation. In this way, two gauging bases of 200 mm length were created for further measurement (see Fig. 1). This arrangement enabled the capture of the total relative length changes of the composite since the time the composite is placed into the mould until its long-term ageing after the specimen is removed from the shrinkage mould. Details about the markers types, drawing and their arrangement can be found in [8].

2.3 Fracture tests

The fracture tests were carried out in three-point bending configuration of beam specimens with a notch, depth of notch was approximately 1/3 of the specimen height, located in the middle of the specimen length; span length was 120 mm. Tests were performed on a mechanical testing machine FP10/1 with a measuring range of 1–1000 or 2000 N.

The displacement increment loading test was performed, which allowed to record load versus displacement (deflection in the middle of the span length) F – d diagrams during the tests; loading rate was 0.02 mm/min. The F – d diagrams were used for the determination of elasticity modulus from the initial (almost linear) part of the diagram, and for the calculation of effective fracture toughness using the effective crack extension method [9] and specific fracture energy using work-of-fracture method

[10, 11]. Despite the low loading rate, the stability loss occurred during loading, therefore it was not possible to reconstruct the descending part of $F-d$ diagrams [12]. On that account the work of fracture value W_F^* was determined as an area under the proper part of $F-d$ diagrams before stability loss occurred. Refer to [13] for details about determination of above mentioned mechanical fracture parameters.

The informative value of compressive strength was determined on specimen fragments after the fracture test were performed, see Fig. 2.

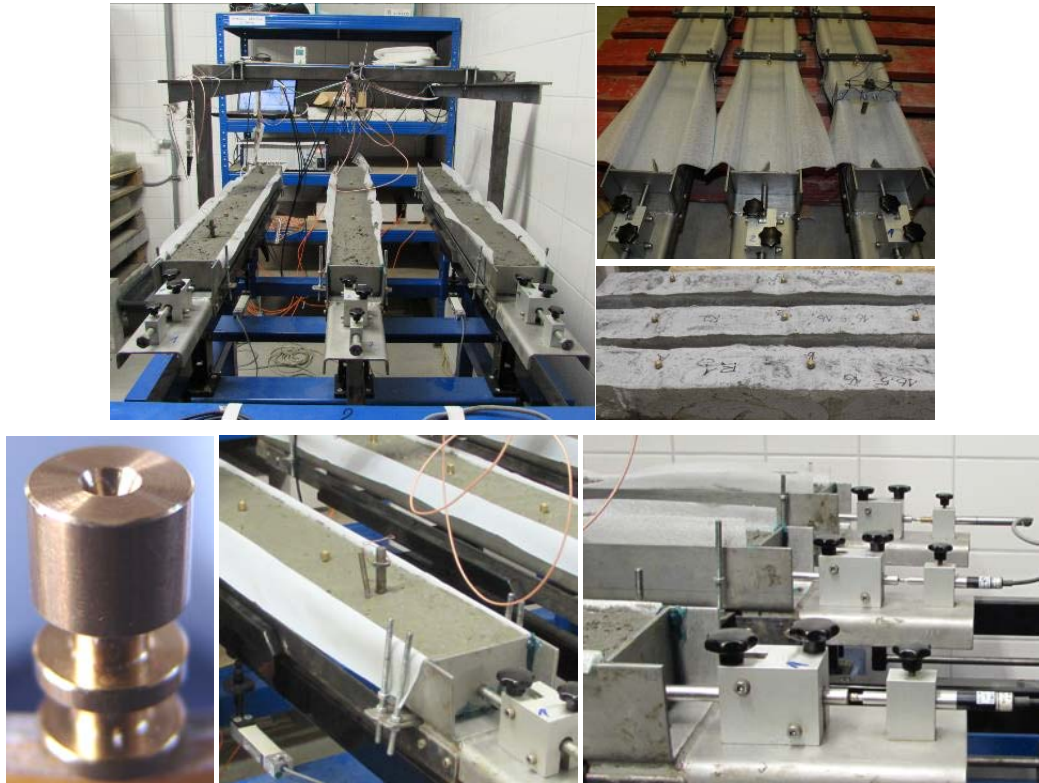


Fig. 1: Illustration of shrinkage, weight loss and relative length changes measurement



Fig. 2: Arrangement of three-point bending fracture test (left) and determination of an informative value of compressive strength

3 RESULTS

The results of performed measurements and their evaluation are shown in bar graphs, in which the height of the bar represents the mean value and error bars represent the sample standard deviation of corresponded parameter, and compared in subsequent tables. The set of three test specimens was used for evaluation of the shrinkage and weight loss development for both PCMs. Fracture parameters of both PCM mortars were obtained from the tests performed on the set of six test specimens at the ages of 3, 28 and 90 days. The informative compressive strength values were determined on the beams' fragments after the fracture tests were completed (twelve test specimens of each age and each mortar).

Fig. 3 shows that both investigated mortars showed a relatively high value of shrinkage at the selected “treatment”. The weight losses recorded during ageing were approximately the same for both mortars (max. difference was around 15 %, see Fig. 4). The shrinkage values obtained for mortar labelled “VII” were approx. 40 % higher than the values obtained for the mortar “V” (see Tab. 1). The highest difference was obtained at the age of 28 days. Mortar “VII” also showed greater variability of results.

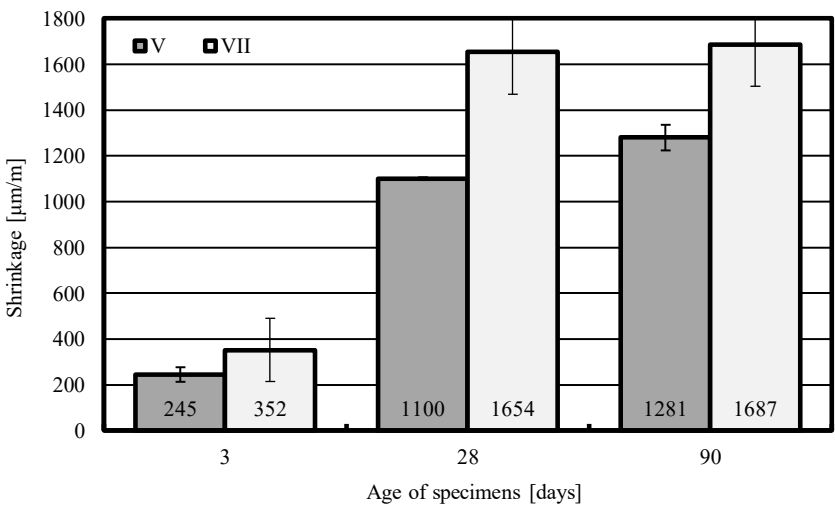


Fig. 3: Shrinkage values depending on specimen age

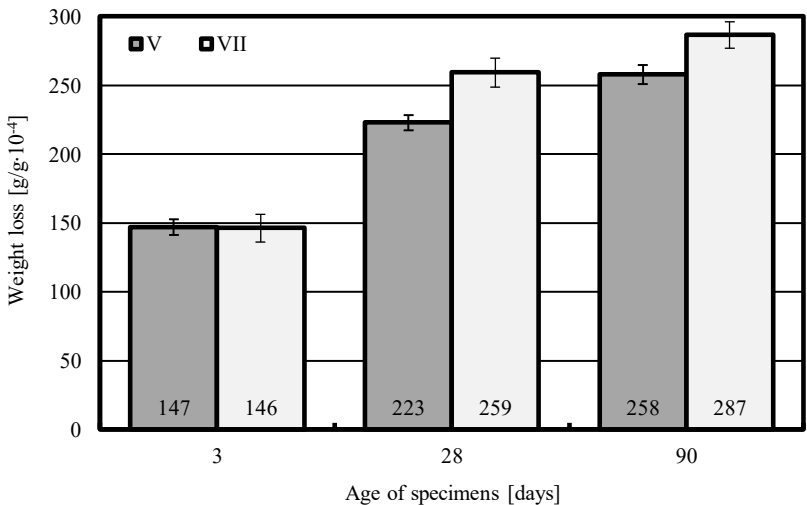


Fig. 4: Weight losses depending on specimen age

Fig. 5 shows the development of informative compressive strength values determined on specimens' fragments after the fracture tests were performed. The results showed an increase in strength over the time for both investigated mortars, where generally higher values were monitored in the case of mortar labelled "VII" in comparison with mortar "V". Exception was observed for the value of compressive strength at the age of 3 days, which was about 25 % lower in the case of set "VII" than the value of set "V" (see Tab. 1). In case of specimens made by cutting the value of compressive strength decreased about 20 % in comparison with specimen made in moulds for mortar "V". The opposite trend was observed in case of mortar "VII", where the compressive strength value was about 18 % higher for specimens made by cutting. It should be noted that high variability of test results was observed for all investigated parameters of both mortars at all ages.

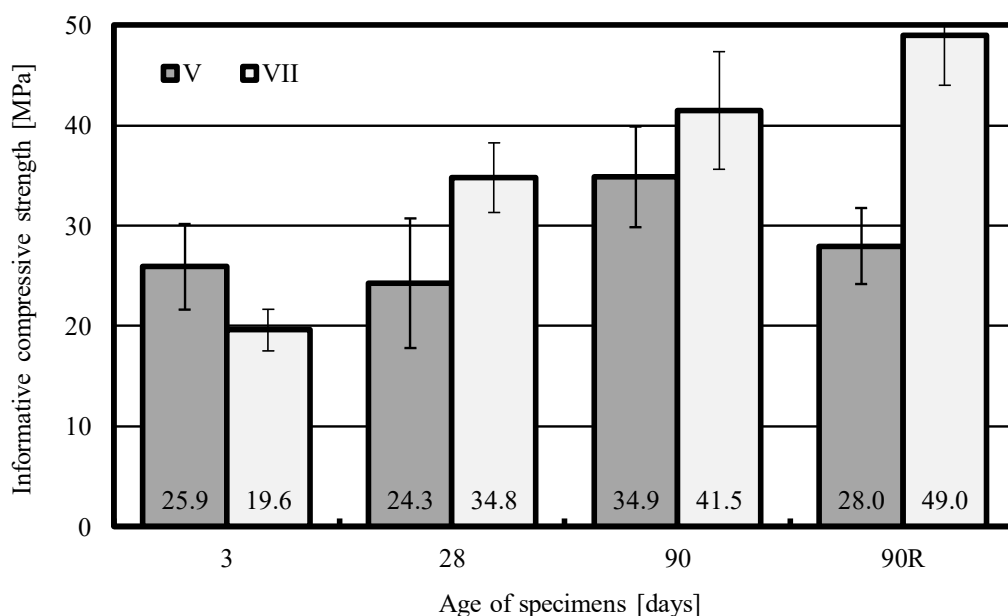


Fig. 5: Informative compressive strength value depending on specimen age

Fig. 6–8 show the results obtained from the fracture tests – development of values of the modulus of elasticity, effective fracture toughness and specific fracture energy. The results show that the values of monitored parameters sharply decreased in the case of mortar "VII" at the age of 90 days (up to 70 %) compared with the results obtained at age of 28 days. In the case of mortar "V", rather different development of fracture parameters was recorded – the modulus of elasticity value is almost the same for all investigated ages of test specimens (difference of about 5 %), the effective fracture toughness and specific fracture energy values decreased by approx. 7 % at the age of 90 days, compared to the results obtained at the age of 28 days. The effective fracture toughness value decreased approx. 15 % and specific fracture energy value about 20 % in the case of test specimens labelled "R" (also compared to the results obtained at the age of 28 days). All monitored mechanical fracture parameters were lower in case of specimens made by cutting. The differences were up to 15 % in case of mortar "V". In case of mortar "VII" the higher differences were observed, the modulus of elasticity value was lower even more than 50 % for specimens made by cutting.

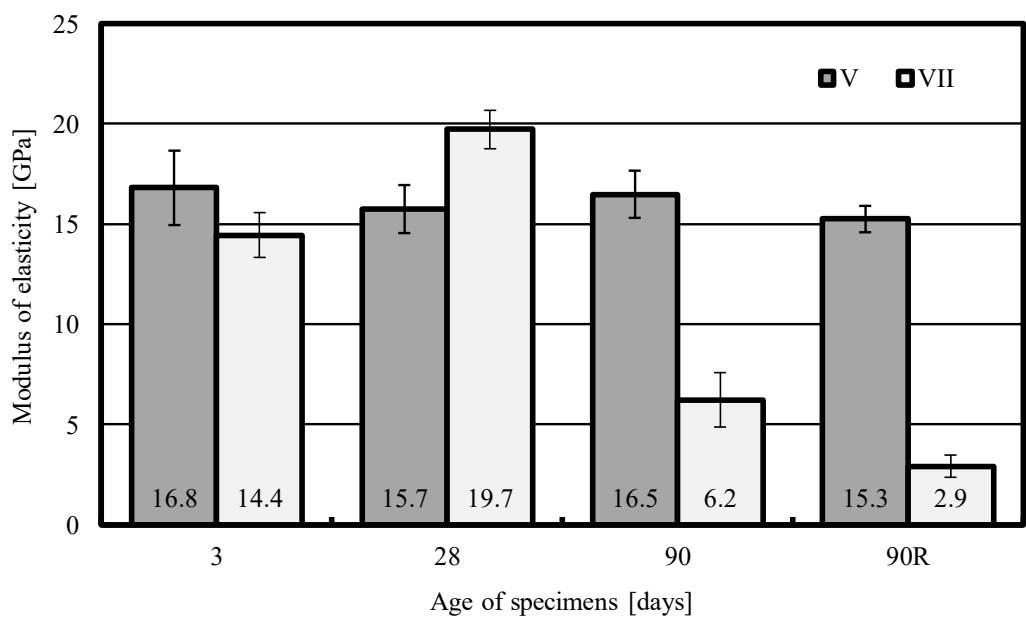


Fig. 6: Modulus of elasticity value depending on specimen age

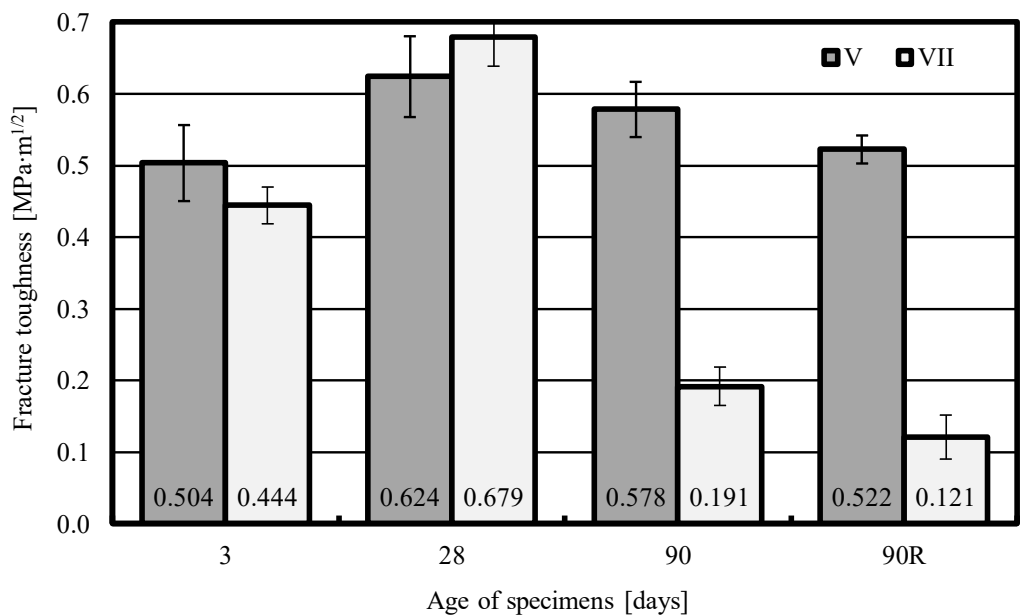


Fig. 7: Effective fracture toughness value depending on specimen age

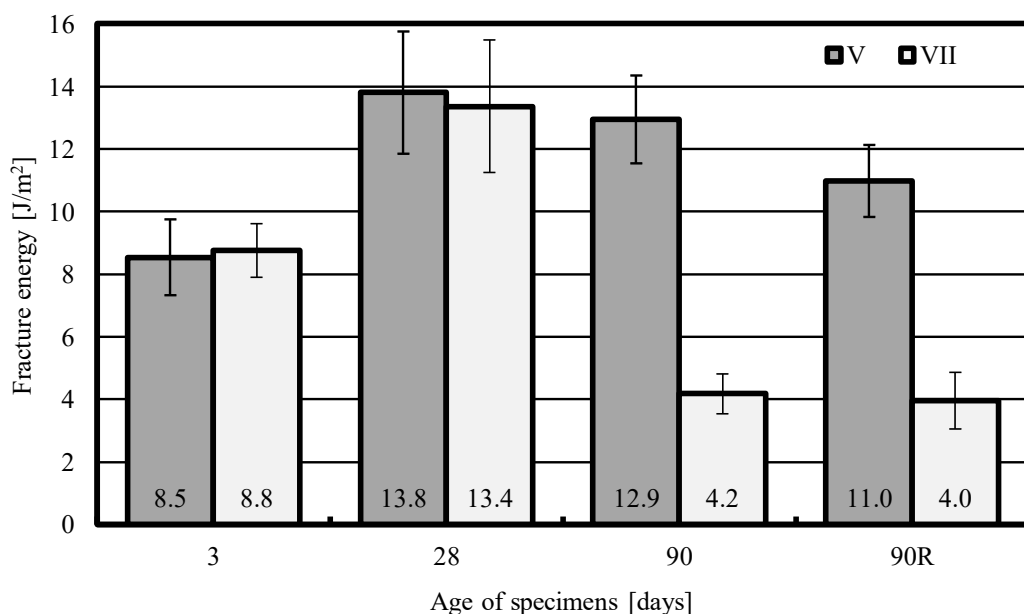


Fig. 8: Specific fracture energy value depending on specimen age

4 CONCLUSIONS

The paper presents the results of pilot experiments aimed at monitoring of development of shrinkage, weight losses and mechanical fracture parameters of two selected polymer-modified cement mortars during their ageing. It is important to note that all the test specimens were stored in laboratory conditions, at a temperature of $(21 \pm 2) ^\circ\text{C}$ and relative humidity of $(60 \pm 10) \%$. During the first 72 hours, all specimens were stored in the moulds (only upper surface of specimens was left to dry freely) and after that, they were removed from the moulds and placed onto prepared shelves without further treatment (all their surfaces were intentionally left to dry freely). For the purpose of experiment evaluation, results obtained for set “V” and “VII” were compared to each other. The results obtained for individual sets of specimens were also compared in terms of development of the parameter values in the time and method of preparation of test specimens. The results show that even the specimens made from PCM mortars, for which the manufacturer declares no shrinkage, shrank considerably. The results also show that the development of mechanical fracture parameters during ageing of these materials differs from generally expected trends and thus the monitoring of these parameters is recommended.

Tab.1: The relative values of monitored parameters (1 = parameter for mortar “V”)

Parameter	Set	Age of specimens [days]			
		3	28	90	90 R
Shrinkage [-]	V	1.00	1.00	1.00	–
	VII	1.44	1.50	1.32	–
Weight loss [-]	V	1.00	1.00	1.00	–
	VII	1.00	1.16	1.11	–
Informative compressive strength [-]	V	1.00	1.00	1.00	1.00
	VII	0.76	1.43	1.19	1.75
Modulus of elasticity [-]	V	1.00	1.00	1.00	1.00
	VII	0.86	1.25	0.38	0.19
Effective fracture toughness [-]	V	1.00	1.00	1.00	1.00
	VII	0.88	1.09	0.33	0.23
Specific fracture energy [-]	V	1.00	1.00	1.00	1.00
	VII	1.03	0.97	0.32	0.36

Tab.2: The relative values of monitored parameters (1 = parameter at the age of 28 days)

Parameter	Set	Age of specimens [days]			
		3	28	90	90 R
Shrinkage [-]	V	0.22	1.00	1.16	–
	VII	0.21	1.00	1.02	–
Weight loss [-]	V	0.66	1.00	1.16	–
	VII	0.56	1.00	1.11	–
Informative compressive strength [-]	V	1.07	1.00	1.44	1.15
	VII	0.56	1.00	1.19	1.41
Modulus of elasticity [-]	V	1.07	1.00	1.05	0.97
	VII	0.73	1.00	0.32	0.15
Effective fracture toughness [-]	V	0.81	1.00	0.93	0.84
	VII	0.65	1.00	0.28	0.18
Specific fracture energy [-]	V	0.62	1.00	0.94	0.80
	VII	0.66	1.00	0.31	0.30

Tab.3: The relative values of monitored parameters (1 = parameter for specimens made in moulds)

Parameter	Set	Specimens made in mould	Specimens made by cutting
Informative compressive strength [-]	V	1.00	0.80
	VII	1.00	1.18
Modulus of elasticity [-]	V	1.00	0.93
	VII	1.00	0.47
Effective fracture toughness [-]	V	1.00	0.90
	VII	1.00	0.63
Specific fracture energy [-]	V	1.00	0.85
	VII	1.00	0.95

ACKNOWLEDGMENT

This outcome has been achieved with the financial support of the Ministry of Education, Youth and Sports of the Czech Republic under the “National Sustainability Programme I” (project No. LO1408 AdMaS UP).

LITERATURE

- [1] MALORNY, W. & PLATH, M. Investigations on Properties Determining Durability of Novel PCC. In: DROCHYTKA, R., VANĚREK, J. *Advanced Materials Research: Proceedings of the Conference on the Rehabilitation and Reconstruction of Buildings CRRB 2012*. Switzerland: 2013, Vol. 688, pp. 130–138, ISSN 1662-8985, ISBN 978-3-03785-679-6.
- [2] OHAMA Y. *Handbook of polymer-modified concrete and mortars properties and process technology*. 1st ed. Park Ridge, N.J: Noyes Publications; 1995. 246 pp. ISBN 978-08155-1358-2.
- [3] ŁUKOWSKI, P. Polymer-Cement Composites Containing Waste Perlite Powder. *Materials* [online]. 2016, Vol. 9, Issue 10, p. 839. ISSN 1996-1944. doi:10.3390/ma9100839
- [4] ČSN EN 1504, PART 1–10 (73 2101). Products and systems for the protection and repair of concrete structures. Prague: ČNI, 2006. In Czech.
- [5] COURARD, L., LENAERS, J.-F., MICHEL, F & GARBACZ, A. Saturation level of the superficial zone of concrete and adhesion of repair systems. *Construction and Building Materials* [online]. 2011, Vol. 25, Issue 5, pp. 2488–2494, ISSN 0950-0618.
- [6] Schleibinger Testing Systems, <http://www.schleibinger.com>.
- [7] VYMAZAL, T., DANĚK, P., KUCHARCZYKOVÁ, B. & MISÁK, P. *Continuous measurement method of cement composite weight losses in early phase of setting and hardening, and apparatus for making the same*. 2015, Czech Republic, CZ 304898 B6 Patent, Granted 26. 11. 2014, Written 7. 1. 2015. In Czech.
- [8] KUCHARCZYKOVÁ, B., DANĚK, P., MISÁK, P. & VYMAZAL, T. *Apparatus for measuring relative deformation of concrete and cement composites*. 2011, Czech Republic, CZ 21600 U1 Utility model, Granted 12. 1. 2011. In Czech.
- [9] KARIHALOO, B. L. *Fracture Mechanics and Structural Concrete*. New York: Longman Scientific & Technical. 1995. ISBN 0-582-21582-X.
- [10] RILEM TC-50 FMC (Recommendation, 1985) Determination of the fracture energy of mortar and concrete by means of three-point bend test on notched beams. *Materials & Structures*. 1985, Vol. 18, pp. 285–290.
- [11] VESELÝ, V. *The Role of Process Zone in Quasi-brittle Fracture*. Brno, 2015. Habilitation thesis. Brno University of Technology, Faculty of Civil Engineering, Institute of Structural Mechanics.
- [12] FRANTÍK, P. & MAŠEK, J. 2015. GTDiPS software, <http://gtdips.kitnarf.cz/>.
- [13] ROVNANÍK, P. ŠIMONOVÁ, H., TOPOLÁŘ, L., BAYER, P., SCHMID, P. & KERŠNER, Z. Carbon nanotube reinforced alkali-activated slag mortars. *Construction and Building Materials*, 2016, vol. 119, pp. 223–229.