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COMPLEX EVALUATION OF THE MECHANICAL AND FRACTURE PROPERTIES OF CEMENTITIOUS MATERIALS WITH DIFERENT WATER-CEMENT RATIO

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Abstract: This paper is aimed at the development of the physical, mechanical and fracture characteristics of cementitious materials during ageing. Three mortars which differed only in the water-cement ratio (*w*/*c*) were prepared for manufacturing of the test specimens intended for the experiments. All mortars were prepared using the standardized quartzite sand with the particle grain size distribution of 0−2 mm CEN 196-1, CEM I 42.5 R Portland cement (cement plant Mokrá, Czech Republic) and water. The reference *w*/*c* was set to the value of 0.5. To produce two other mortars, this ratio was reduced by approx. 5 and 30 %. This resulted in *w*/*c* ratio 0.47 and 0.35 respectively. All test specimens were not protected intentionally from drying during the whole time of measurement and were stored in laboratory at ambient temperature of 21 ± 2 °C and relative humidity of 60 ± 10 %. The results of the shrinkage, elastic, fracture and strength parameters determined within the time interval from 3 days to 2 years of ageing are summarized and discussed in the paper. The results of performed experiments were partially published within the conference DYN-WIND'17. This paper focuses especially on the evaluation of the results obtained after 2 years of specimens ageing and also provides a complex discussion of the results gathered during the whole time of ageing. The results show a crucial impact of the *w*/*c* on the final values of mechanical and fracture characteristics. The early-age and long-term characteristics were substantially influenced by the poor curing conditions.

1 INTRODUCTION

Cementitious materials belong to the building materials used for a wide range of applications. Even though that they are studied for decades there are still many problems which are worth investigating. The role of water-cement ratio (*w*/*c*) and the problems related to its values were investigated many times for various applications. There are two basic approaches which are used to investigate this problem. The first is to study the influence of the different *w*/*c* on the specific materials' characteristics. The experiments are mostly performed using the test specimens cured under standard conditions – immersion in the water bath or curing in the environment with relative humidity of RH ≥ 95 %. The second approach is the investigation of the materials' characteristics with a specific value of *w*/*c* cured under different conditions, where one of them is the standard curing.

Unfortunately, even though the knowledge in this field is relatively wide, there are still problems with the cracking tendency in practical applications. Many of them are related to the cracking at the early stage of materials' ageing which influences the service life of building materials and structures [1]. The inappropriate curing conditions are one of the factors which increase the risk of shrinkage cracking due to the rapid evaporation of the water from the solidified material [2, 3].

In common building practice, the deterioration degree caused by cracks formation is often evaluated based on the non-destructive monitoring of the development of dynamic modulus of elasticity using an ultrasonic or resonance method. These non-destructive methods are further completed by the results of destructive tests, such as compressive strength, tensile or bending strength tests [4]. As proven in various cases, the above mentioned testing methods are not sensitive enough to evaluate the quality and strength of the particles bonds in the internal structure of the materials and therefore it is advantageous to supplement this results by more complex testing methods such as fracture tests and nanoindentation [5]. These methods are highly sensitive to the micro defects in the internal structure of the materials and are widely used in mechanical engineering.

Currently, both methods are commonly used in the field of building materials testing with the aim of evaluation the resistance of materials to the cracks initiation and propagation [6–9].

2 EXPERIMENTAL INVESTIGATION

The main aim of performed experiments was to determine and evaluate the mechanical and fracture characteristics of cementitious materials during ageing and to find the correlation between the investigated parameters. Intentionally, all test specimens were not treated in any way and was exposed to free desiccation under laboratory conditions during the whole time of measurement. The influence of the water-cement ratio (*w*/*c*) on the development of mechanical and fracture characteristics was investigated. The results of performed experiment were partially published in the conference DYN-WIND'17 [10]. This actual paper focuses especially on the evaluation of the results obtained after 2 years of specimens ageing and also provides a complex discussion of the results gathered during the whole time of measurement.

2.1 Materials and test specimens

Three cementitious mortars (A, B and C) which differed only in the *w*/*c* were prepared for manufacturing of the test specimens. To minimize the aggregate influence, the standardized sand CEN196-1 was used as an aggregate. The binder consisted of CEM I 42.5 R Portland cement and water in ratio of 0.5, 0.47 and 0.35 for mortar A, B and C, respectively. The aggregate-cement ratio (*a*/*c*) was 3:1 for all mortars. To maintain the workability of the mortar C, the polycarboxylate ether-based superplasticizer in an amount of 1% by cement mass was used for mixture preparation. All mortars were mixed using a mixer with controllable mixing speed and casted into the moulds of two different sizes. The specimens used for shrinkage and mass losses measurement were casted into the moulds with dimensions of 1000 mm in length, 100 mm in width and 60 mm in height. The moulds were equipped with a movable head which enable the early-age measurement of shrinkage. Three test specimens were manufactured from each mortar. Rest of the measurements were performed using the standardized specimen size of 40 × 40 × 160 mm.

All test specimens were cured in the laboratory with a stable temperature of 21 ± 2 °C and relative humidity of 60 ± 10 % and were not protected from drying during the whole time of measurement.

The mechanical and fracture characteristics were evaluated for the mortars at the age of 3, 28, 90 and 730 days. One testing set contained at least 6 test specimens which were tested for a particular characteristic at the specific mortars' age. The modulus of elasticity, fracture toughness and compressive strength were obtained always from one set of the specimens tested at the specific age. Dynamic modulus of elasticity and shrinkage were determined on other sets of specimens.

2.2 Shrinkage and mass losses measurement

The measurement was designed to measure changes in length of the test specimens along their longitudinal axis and to record the changes in mass of the specimens caused by free drying. The measurement was performed in two consecutive stages – early-age measurement (up to age of 72 hours) and long-term measurement (within 72 hours and 730 days). The early-age measurement was carried out on the specimens placed in the moulds where the changes in length were recorded using an inductive sensor leaning against the movable head of the moulds. The early-age measurement started approx. 1 hour after placing the fresh-state materials into the moulds. The moulds were placed on a special weighing table which enabled to record the changes in mass continuously without handling with the test specimens.

All test specimens were removed from the moulds at the age of 72 hours and the long-term shrinkage and mass losses were measured manually. The changes in length were measured using a mechanical strain gauge placed into markers embedded into the upper surface of the specimens during its manufacturing. The changes in mass were measured using a laboratory scale with the resolution of 0.5 g. Refer to [11] for more details about the test procedure.

Due to the early start of the measurement, the shrinkage in the plastic and semi-plastic stage of the solidification process was recorded and reflected in the total values of shrinkage.

2.3 Fracture tests

The fracture characteristics were determined based on the load–deflection (*L*–*d*) diagrams recorded during the three-point bending test of the test specimens with an initial notch. The depth of the notch was, in this case, approx. 1/3 of the specimens' height. The specimens were subjected to the quasi-static loading test with a constant displacement increment of 0.02 mm/min. The modulus of elasticity was calculated from the initial linear part of *L*–*d* diagrams according to [12] using following formula:

$$E_c = \frac{L_i}{4Bd_i}\left(\frac{S}{W}\right)^3 \left[1 + \frac{5qS}{8L_i} + \left(\frac{W}{S}\right)^2 \left\{2.70 + 1.35\frac{qS}{L_i}\right\} - 0.84\left(\frac{W}{S}\right)^3\right] + \frac{9}{2}\frac{L_i}{Bd_i}\left(1 + \frac{qS}{2L_i}\right)\left(\frac{S}{W}\right)^2 F_1(\alpha_0) \qquad (1)$$

where L_i is the load in the initial linear elastic range, d_i is the midspan deflection corresponding to the load level L_i, B and W are the width and height of the specimen, respectively, S is the span length, q is the self-weight of the specimen per unit length and

$$F_1(\alpha_0) = \int_0^{\alpha_0} xY^2(x)dx \qquad (2)$$

with $\alpha_0 = a_0/W$, a_0 is the initial notch depth and $Y(x)$ is the geometry function for a three-point bend beam [12].

The effective fracture toughness K_{Ice} is determined based on the *L*–*d* diagrams using the effective crack model [12]. The effective crack length a_e corresponding to the maximum load $L_{\max}$ and matching the midspan deflection $d_{L\max}$, had to be calculated first. It follows the concept of effective crack model that the a_e can be calculated from the equation (1) using $L_{\max}$ and $d_{L\max}$ instead of L_i and d_i.

Afterwards, the K_{Ice} is calculated using the linear elastic fracture mechanics formula according [12]:

$$K_{Ice} = \frac{3L_{max}S}{2BW^2}Y(\alpha_e)\sqrt{a_e} \qquad (3)$$

with $\alpha_e = a_e/W$.

The compressive strength was determined on the fragments of the prismatic specimens obtained after the fracture tests were finished.

2.4 Dynamic modulus of elasticity

The resonance method was used for determination of the dynamic modulus of elasticity. The dynamic material property was evaluated using the natural frequencies of longitudinal vibration according to the following formula:

$$E_{crL} = 4l^2 \cdot f_L^2 \cdot D \qquad (4)$$

where E_{crL} is the dynamic modulus of elasticity in MPa, l is the specimen length in m, f_L is the natural frequency of longitudinal vibration in kHz and D is the bulk density of the material. Refer to [4] for more details about this testing method.

3 RESULTS AND DISCUSSION

The results of performed measurements are displayed in Fig. 1 – Fig. 6. The average values accompanied by the sample standard deviations are shown for all investigated characteristics.

The differences in *w*/*c* are reflected especially in the absolute values of particular characteristics – the highest are recorded for mortar C. The slowest initial increase in this characteristics is observed for mortar A.

The poor curing conditions are reflected in the higher variability of the results and in the long-term development of the values of investigated characteristics – the lower *w*/*c*, the higher impact on the variability of the results. In the case of mortar C (the lowest *w*/*c*) the variability of the results increases with the age of material significantly (see Fig. 4, 5 and 6).

This effect is related to the process of desiccation, expressed herein by the process of mass losses (see Fig. 3). All materials exhibit a rapid decrease in the mass within the 28 days of ageing which means a substantial loss of the water, which is needed for cement hydration. The absence of water leads to the weakening of the bonds between the particles in the internal structure of the mortars. The desiccation leads also to formation of the higher internal porosity of the mortars.

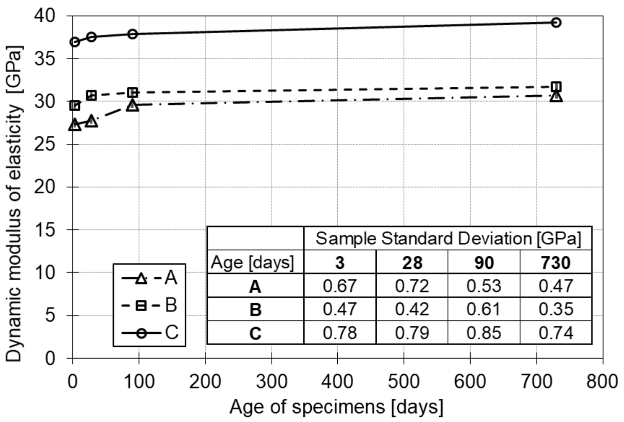

Figure 1: Dynamic modulus of elasticity

Based on the results of the dynamic modulus of elasticity (see Fig. 1), it can be stated that the weakness of the particle bonds does not lead to the formation of the significant cracks. The crack formation is reflected in the decrease in the value of dynamic modulus of elasticity.

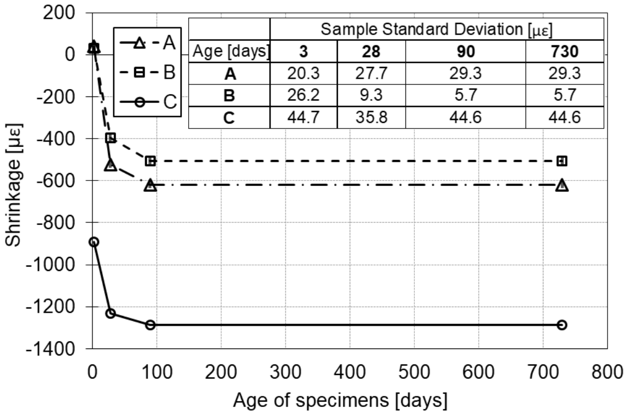

Figure 2: Shrinkage

The rapid drying leads also to the rapid shrinkage – the highest is recorded for mortar C (see Fig. 2). At the age of 28 days, the shrinkage values reach about 80% of the steady-state value for mortar A and B and 95 % for mortar C. The most negative impact of the curing conditions is observed in the case of mortar C for which the total shrinkage value measured at the age of 3 days is about 900 με which is approx. 70% of the steady-state value determined at the age of 730 days. Another two mortar exhibit a slight expansion strengthened by the initial bleeding after placing the fresh-state material into the moulds. This expansion reduces the early-age shrinkage values.

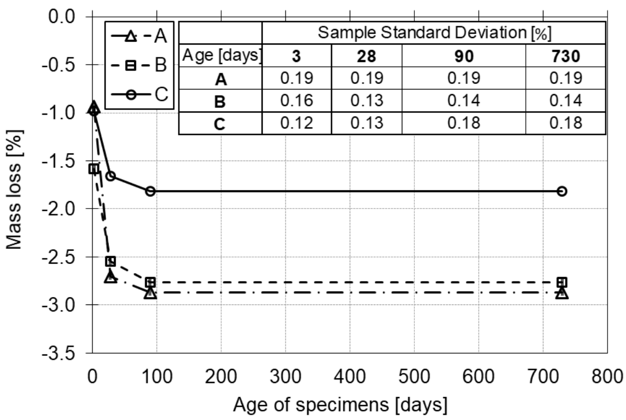

Figure 3: Mass losses

If the results of effective fracture toughness and modulus of elasticity determined from the *L–d* diagrams are studied in details, the relations between these characteristics can be observed.

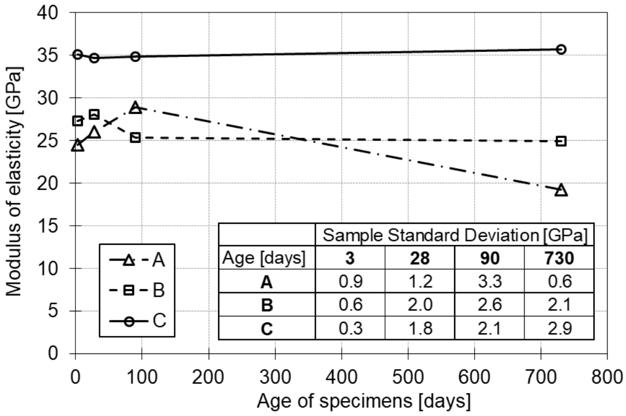

Figure 4: Modulus of elasticity from *L–d* diagrams

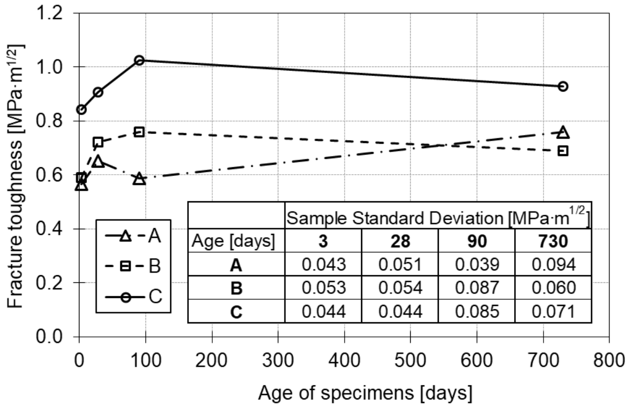

Figure 5: Effective fracture toughness

The most visible effect is observed for mortar A with the highest *w/c* and high porosity caused by desiccation. The value of the modulus of elasticity increases within the age-interval from 3 to 90 days (see Fig. 4). However, the value of effective fracture toughness increases only up to the age of 28 days (see Fig. 5). It can be supposed that at the age of 90 days the particles bonds are not able to withstand the applied load which is reflected in the decrease in the value of effective fracture toughness. The weakness of the particles bonds together with a high porosity caused by desiccation lead to the decrease in the value of elastic modulus. In relation to the calculation method, this softening leads to the increase in the value of effective fracture toughness. In the case of mortar C, with a lowest *w/c* and lowest porosity, the weakness of the particles bonds is observed at the age of 730 days (see Fig. 4 and 5).

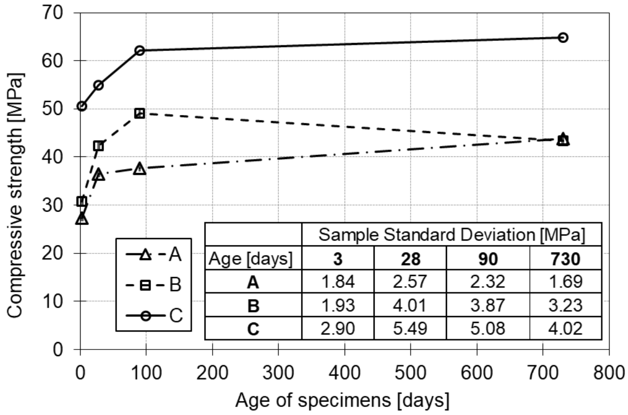

Figure 6: Compressive strength

The experimental data were further used to find the correlation between the investigated characteristics. Because there is only one variable in the composition of investigated materials (different *w/c*), Fig. 7 – Fig. 9 display all data of particular characteristics obtained for materials A, B and C tested at the age of 3, 28, 90 and 730 days. For a better visualization of the results distribution, the data representing particular materials are displayed with different markers – triangle, square and circle are used for material A, B and C, respectively. At least 32 values of investigated characteristics for each mortar were used as an input data for the correlation evaluation.

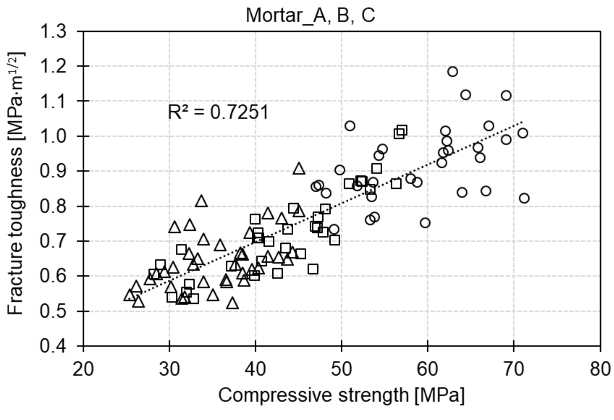

Figure 7: Correlation between compressive strength and fracture toughness

As shown in plots, relatively strong correlation is found only between the compressive strength and fracture toughness. The correlation is affected by the increased variability of the results caused by the curing conditions.

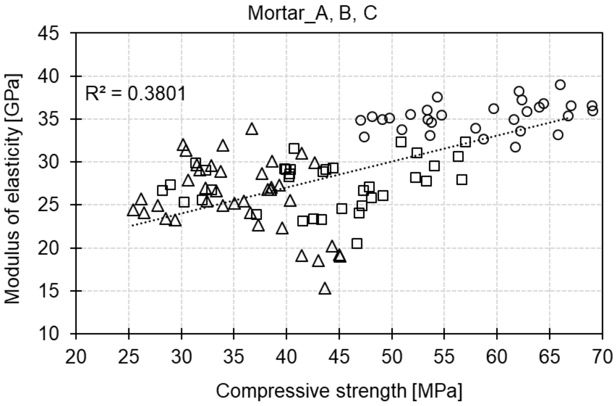

Figure 8: Correlation between compressive strength and modulus of elasticity from L–d diagrams

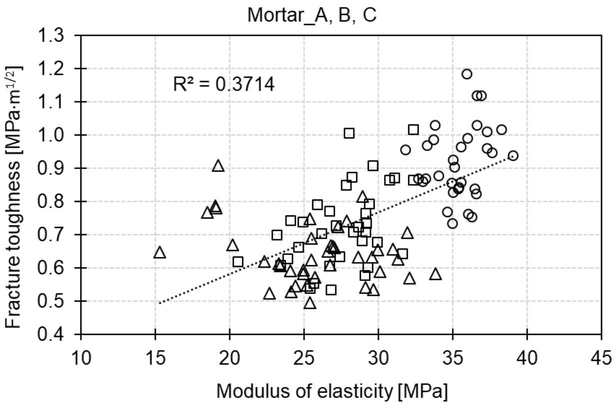

Figure 9: Correlation between modulus of elasticity and fracture toughness

In spite of the fact that there is a relation between the effective fracture toughness and modulus of elasticity related to the weakening of the particles bonds, the general correlation between these characteristics is not proven (see Fig. 9). This is related to the evaluation approach of both characteristics (see paragraph 2.3).

Another three correlations of the experimental data were verified, namely the correlation between the compressive strength, dynamic modulus of elasticity, shrinkage and effective fracture toughness (see Fig. 10 – 12). Because the particular characteristics were obtained from the measurement performed on different sets of specimens, the average values of investigated characteristics, determined at the age of 3, 28, 90 and 730 days, are displayed in following figures.

Strong correlation is found between the compressive strength and dynamic modulus of elasticity (see Fig. 10). The correlation displayed in Fig. 11 shows the relation between the compressive strength and shrinkage values. The results show that the reduction of the *w*/*c* value leads to the increase of the strength accompanied by the high shrinkage without significant internal cracking.

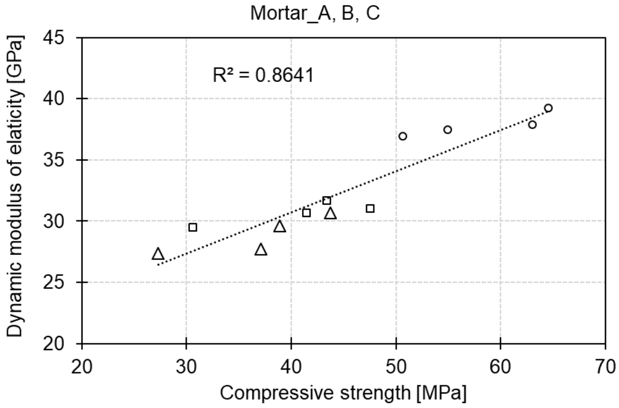

Figure 10: Correlation between compressive strength and dynamic modulus of elasticity

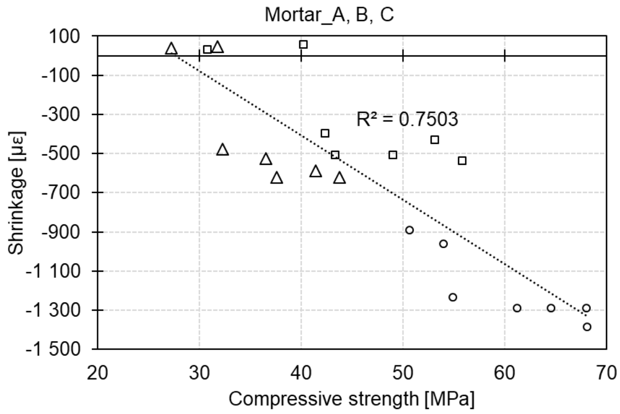

Figure 11: Correlation between compressive strength and shrinkage

The last correlation displays the relation between the effective fracture toughness and shrinkage (see Fig. 12). The coefficient of determination is lower than for other two correlations. Based on the previous explanations, it can be stated that this relation is affected by the quality of the particles' bonds which leads to more complex approach to investigate and evaluate this problem.

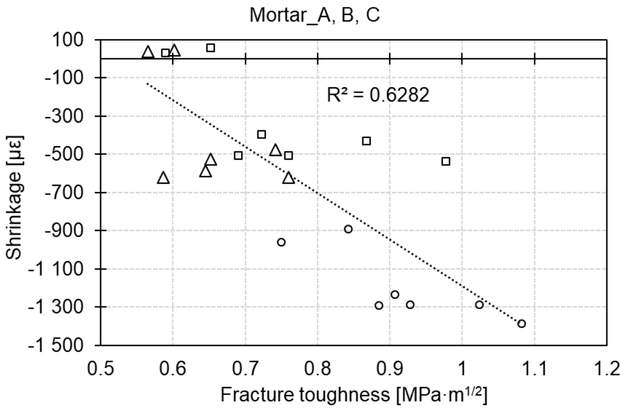

Figure 12: Correlation between effective fracture toughness and shrinkage

4 CONCLUSIONS

The results of the experimental analysis aimed at the development of selected mechanical and fracture characteristics are presented in the paper. To simulate the inappropriate curing conditions, the specimens manufactured from mortars with a different *w/c* are intentionally left to dry freely during the whole time of ageing and tested in pre-defined ages – at the age of 3, 28, 90 and 730 days. It can be suggested that, in this case, the differences in the mechanical and fracture characteristics are more visible from the long-term point of view.

The results show that the magnitude of *w/c* in combination with poor curing conditions are reflected in the absolute values of the investigated characteristics and especially in the high variability of the results which increase with the age of materials – the lower *w/c*, the higher impact on the variability.

The impact of *w/c* and curing conditions is also reflected in the increase of the shrinkage values and in the decrease of the fracture characteristics. The lower *w/c* is, the more rapid shrinkage the material exhibits, especially during the first 3 days of ageing. On the other hand, the higher *w/c* is, the earlier decrease in the value of effective fracture toughness occur.

Based on the results of the fracture tests, the moment of critical weakness of the particles bonds can be find. The results also show that the resonance method and compressive strength test are not sensitive enough to reflect the actual changes in the porosity and quality of the particles bonds in the internal structure of the materials.

A correlation between the particular characteristics is found based on the experimental data. The relatively strong correlation is observed between the compressive strength and fracture toughness which show that the compressive strength and effective fracture toughness increase with decreasing *w/c*. A similar correlation is found between the compressive strength, dynamic elastic modulus and shrinkage.

The relations between other characteristics is affected by the quality of particle bonds which leads to more complex approach to investigate and evaluate this problem.

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