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# THE INFLUENCE OF DIFFERENT CURING CONDITIONS ON HVFAC RHEOLOGICAL AND MECHANICAL PROPERTIES

***Abstract:***

This research was conducted in order to evaluate the influence of different curing conditions on rheological and mechanical properties of high volume fly ash concrete (HVFAC) in comparison with the ordinary Portland cement concrete (OPC). Four types of concrete were made: two HVFAC and two OPC designed to have the same consistency and 28-day compressive strength for samples cured in water. Also, three different curing regimes were chosen: standard water curing (W), standard laboratory air curing (L) and curing in standard laboratory conditions using curing compound based on the polyolefin emulsion (C). The main objectives were to evaluate the influence of these curing regimes on the compressive strength, flexural tensile strength, modulus of elasticity development over time, water permeability of concrete and concrete shrinkage. The use of curing compound improved previously mentioned properties in some extent compared with the samples cured in standard air conditions.

*Keywords: high-volume fly ash concrete, curing conditions, curing compound, mechanical properties, shrinkage*

# УТИЦАЈ РАЗЛИЧИТИХ ВРТСА НЕГЕ НА РЕОЛОШКЕ И МЕХАНИЧКЕ КАРАКТЕРИСТИКЕ БЕТОНА СА ВЕЛИКИМ САДРЖАЈЕМ ЛЕТЕЋЕГ ПЕПЕЛА

***Сажетак:***

Истраживање је имало за циљ да се испита утицај различитих услова неге на реолошка и механичка својства бетона направљеног са високим садржајем летећег пепела (HVFAC) у поређењу са класичним бетоном на бази цемента (OPC). Направљене су четири врсте бетона: два HVFAC и два ОПЦ који су пројектоване тако да имају исте конзистенције и 28-дневне чврстоће при притиску за узорке негované у води. Такође, примењена су три различита режима неге бетона: стандардна нега у води (W), стандардно неговање на ваздуху у лабораторији (L) и неговање у стандардним лабораторијским условима након премазивања узорака средством на бази полиолефинске емулзије (C). Главни циљ је био да се утврди утицај оваквих режима неге на развој чврстоће при притиску, чврстоће при затезању услед савијања, модула еластичности током времена, водонепропустљивост бетона и скупљања. Примена средства за негу је побољшала предметна својства у поређењу са узорцима негованим на ваздуху.

*Кључне ријечи:: бетон са великим садржајем летећег пепела, услови неге, премази за негу, механичка својства, скупљање*

# 1. INTRODUCTION

The immense impact of the construction industry on the environment is mainly caused by the extremely large use of raw materials, energy consumption and waste production. The use of natural stone aggregates and large carbon dioxide ($CO_2$) footprint from cement production are the main problems of non-sustainable concrete production. The main focus of current research done in the material science field is oriented towards finding new alternatives to conventional construction materials by using waste and recycled materials. In order to preserve natural resources and make concrete more environmentally friendly, supplementary cementitious materials (SCMs) are being increasingly used. SCMs are usually by-products obtained from different industries that possess pozzolanic activity potential. Among different SCMs, fly ash (FA) is available in local countries, it can be used without additional treatment, it has a relatively low price, and, above all, large quantities deposited in the landfills grow rapidly. The important benefit from the utilization of FA as a cement replacement is the reduction of $CO_2$ emissions from the Portland cement production. Approximately one ton of $CO_2$ is released for each ton of the Portland cement clinker [1]. A positive environmental effect of using FA in concrete is also obtained through the decrease of the amount of FA deposited in landfills and through the use of the waste material instead of natural resources for concrete production. It is for these reasons that today there is a general trend of replacing higher amounts of Portland cement in concrete.

Concretes made with high volumes of FA (HVFAC) have been researched science 1985 [2], [3]. HVFAC is usually defined as the concrete with more than 50% of fly ash in the total amount of cementitious materials. A large amount of research has been done regarding the physical and mechanical properties of HVFAC and in addition, work was also done on the evaluation of its material properties through the standards for cement concrete [4]. Furthermore, the connection between HVFAC material properties testing and conditions in practical use must be evaluated for its safe application.

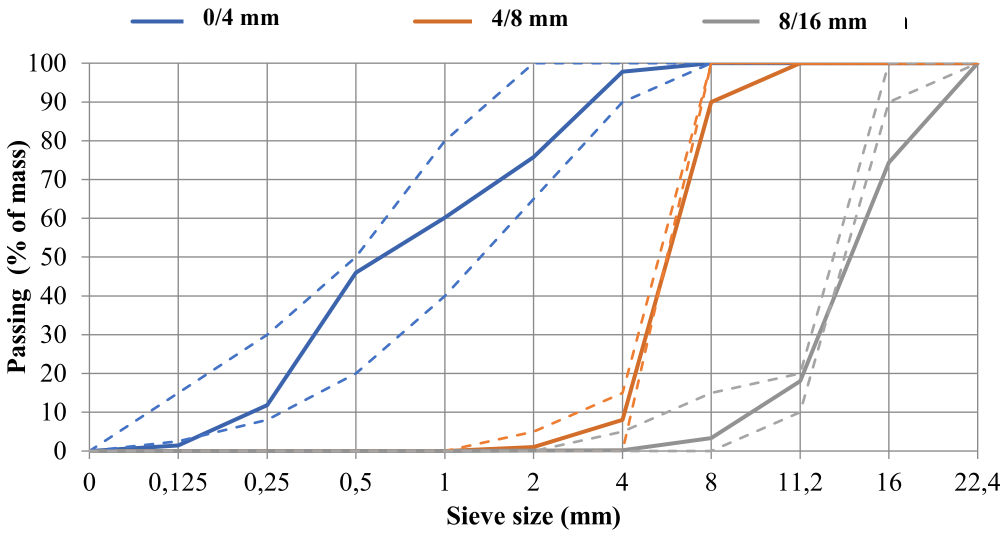
Pozzolanic material is usually defined as the material which will, in the presence of moisture, chemically react with calcium hydroxide $Ca(OH)_2$ at ordinary temperatures to form compounds possessing cementitious properties. At normal temperatures, the pozzolanic reaction is slower than the hydration of cement, so longer curing is needed for the full potential of FA to be reached [5]. It is generally recommended that HVFAC is moist cured for at least 7 days [6]. Adequate duration of moist curing helps the successful development of hydration and pozzolanic reaction, and increased curing temperatures can improve early age strengths [7]. However, results from the literature show that increased curing temperatures or steam curing, although helping the early age strength, can have adverse effect on the 28-day compressive strengths [8], [9]. In order to resolve the discrepancy of current results from literature, more studies are needed to determine the influence of different curing regimes on HVFAC properties. Furthermore, need for increased curing time and humidity recommended for HVFAC can prolong the construction time, so the possibility and efficiency of using current curing compounds in HVFAC should be evaluated.

This paper presents the research conducted in order to evaluate the influence of different curing conditions on HVFAC mechanical properties in comparison with the conventional cement concrete. Four types of concrete were made and tested: two HVFAC and two ordinary Portland cement concretes (OPC) designed to have the similar workability and 28-day compressive strength for samples cured in water. Three curing conditions were chosen for the analysis: standard water curing (W), standard laboratory air curing (L) and curing in standard laboratory conditions using curing compound based on the polyolefin emulsion to coat the samples (C). The main objectives were to evaluate the influence of these curing regimes on the compressive strength, flexural tensile strength, modulus of elasticity development over time, water permeability as well as the concrete shrinkage for both OPC and HVFAC samples.

# 2. MATERIALS AND CURING PROCEDURE

Both types of concrete, OPC and HVFAC, were made with the same component materials (aggregate, cement and water) and the HVFAC mixture was designed to have 50% and 57% of FA in total cementitious materials mass.

All concrete mixtures were made using tap water and river aggregate obtained from "Elita-Cop" separated into three fractions (0/4 mm, 4/8 mm and 8/16 mm) using standard sieving method. Prior to sieving and mixing, the aggregate was dried in the oven until the constant mass was reached. The sieve analysis of used aggregate is presented in Figure 1. The density of used aggregate was 2673 kg/m$^3$, 2578 kg/m$^3$ and 2602 kg/m$^3$ for fractions 0/4 mm, 4/8 mm and 8/16 mm, respectively.




*Dashed lines represent minimum and maximum passing for each size group according to [12]

Figure 1. *Sieve analysis of river aggregate*

Portland composite cement CEM II (class PC 20M (S-L) 42.5R) produced by "Lafarge", Beočin was used for preparation of both concrete types. This type of cement contains additions (ground slag and limestone) of up to 20% of the total mass, and there is no FA in the composition of cement. The specific gravity of cement was 3040 kg/m$^3$.

FA was obtained from the "Nikola Tesla B" power plant in Obrenovac, Serbia. The average specific gravity of FA determined using the pycnometer method was 2075 kg/m$^3$. The chemical composition of FA is presented in Table 1. In the last row of Table 1, the maximum allowed values of certain substances according to EN 450-1: 2012 [10] are presented. As it can be seen, the total quantity of $SiO_2 + Al_2O_3 + Fe_2O_3$ is higher than 70%. The quantity of particles smaller than 45µm is higher than 12%. It can be concluded that the FA used in this research met the requirements of EN 450-1:2012 for the use of FA in concrete, and according to ASTM-C618 [11] provisions could be classified as class F.

Table 1. *Chemical composition of FA (% of mass)*

| $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | $TiO_2$ | $CaO$ | $MgO$ | $Na_2O$ | $K_2O$ | $P_2O_6$ | $SO_3$ | $MnO$ | *LOI* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| *64.14* | *19.22* | *4.35* | *0.16* | *8.32* | *0.01* | *0.36* | *0.66* | *0.17* | *0.86* | *0.03* | *4.68* |
| - | - | - | - | - | *max 4* | *max 5* | - | *max 5* | *max 3* | - | *max 6* |

The proportioning of the concrete mixtures was based on the absolute volume method. The mixing procedure began with mixing cement, FA, sand and coarse aggregate in a mixing pan for one min, then adding water during the next 30 s, and the mixing continued for approximately 5 min. The mixture was used to cast 10 cm concrete cubes for compressive strength testing, 15 × 30 cm cylinders for testing the modulus of elasticity, 15 cm cubes for testing water permeability and 12 × 36 prisms for testing of shrinkage. All samples were demoulded after 24 hours. The values of the various properties reported in this paper represent the mean value of three measurements.

After mixing, all specimens were cast in steel moulds and the concrete was compacted using a vibrating table. The first group of specimens was demoulded after 24 hours and placed in water thank (W-samples). The second group (L-samples) was cured in standard laboratory air conditions (T=20±2°C, RH=60±5°C). Curing of the third group of samples was done using the curing compound based on the polyolefin emulsion (C-samples). Namely, immediately after casting into moulds, the uncovered concrete surface of the C-specimens was sprayed with the liquid curing compound for preventing water loss in concrete. After the demoulding, all other sample sides were wrapped with plastic foil as shown in Figure 2.

Figure 2. *Sample treatment after casting (left) and C-samples treatment after demoulding (right)*

As the target values, workability class S2 and compressive strength class C25/30 (MB30) were adopted for both OPC and HVFAC mixtures. Concrete samples from each concrete mixture were additionally divided into three categories according to the applied curing procedure: OPC_1-W, OPC_1-L, OPC_1-C, HVFAC_1-W, HVFAC_1-L and HVFAC_1-C. The concrete mix designs of OPC and HVFAC mixtures are presented in Table 2.

Table 2. *Concrete mix design*

| *Mixture* | *Water* | *Aggregate* | | | *Cement* | *FA* | *W/CM** |
|---|---|---|---|---|---|---|---|
| | $m_v$ | *[0/4]* | *[4/8]* | *[8/16]* | $m_c$ | $m_{fa}$ | $\omega$ |
| | *[kg/m³]* | *[kg/m³]* | *[kg/m³]* | *[kg/m³]* | *[kg/m³]* | *[kg/m³]* | *[-]* |
| *OPC_1* | 175 | 835 | 557 | 464 | 285 | 0 | 0.614 |
| *OPC_2* | 175 | 821 | 548 | 456 | 320 | 0 | 0.547 |
| *HVFAC_1* | 183 | 876 | 525 | 350 | 150 | 200 | 0.523 |
| *HVFAC_2* | 195 | 838 | 503 | 335 | 200 | 200 | 0.488 |

* *Water-to-cementitious materials ratio*

## 3. RESULTS AND DISCUSION

### 3.1. Workability

Concrete workability was tested using the Abrams cone method according to SRPS EN 12350-2: 2010 [13]. The average values of measured slump values are presented in Figure 3. The obtained values for the OPC and HVFAC mixtures corresponded to workability class S1 (OPC_1 and OPC_2) or S1-S2 (HVFAC_1 and HVFAC2). From the engineering point of view it can be considered that the workability of these four concretes is of the same class.

| Mixture notation | Slump Δh [cm] |
|---|---|
| *OPC_1* | 3.3 |
| *OPC_1* | 3.9 |
| *HVFAC_1* | 4.1 |
| *HVFAC_1* | 4.8 |

Figure 3. *Abrams cone slump test (left) and slump test results (right)*

### 3.2. Density, compressive and flexural tensile strength

Density of hardened concrete samples (Table 3) was performed according to the SRPS EN 12390-7: 2019 [14]. As expected, HVFAC had lower density compared to OPC, regardless of cement and FA amount. However, there was no significant difference in density for samples cured in the same way. The only exception was HVFAC_2 water-cured sample that had a slightly lower density (7.5% compared to HVFAC_1-W sample).

Compressive strength was determined at the ages of 7, 28 and 90 days. The test was conducted in accordance with SRPS EN 12390-3:2010 [15]. Figure 4 shows the development of compressive strength of both OPC and HVFAC mixtures cured in different regimes. At the age of 28 days all samples cured in water had similar compressive strength, which corresponded to the concrete strength class C25/30. An exception was mixture HVFAC_1, that had slightly lower compressive strength compared to other samples (approximately 15%).

*Table 3 – Hardened sample density*

| **Density, γ (kg/m³)** | | | |
|---|---|---|---|
| **Notation** | **Age (days)** | | |
| | 7 | 28 | 90 |
| OPC_1-L | 2320 | 2277 | 2270 |
| OPC_1-W | 2370 | 2366 | 2397 |
| OPC_1-C | 2304 | 2292 | 2295 |
| OPC_2-L | 2322 | 2275 | 2293 |
| OPC_2-W | 2368 | 2357 | 2394 |
| OPC_2-C | 2380 | 2348 | 2310 |
| HVFAC_1-L | 2169 | 2188 | 2197 |
| HVFAC_1-W | 2331 | 2337 | 2298 |
| HVFAC_1-C | 2263 | 2239 | 2202 |
| HVFAC_2-L | 2251 | 2359 | 2164 |
| HVFAC_2-W | 2308 | 2162 | 2337 |
| HVFAC_2-C | 2256 | 2216 | 2195 |

In the first 7 days, OPC samples had higher compressive strength increase compared with the HVFAC, while from 7 to 90 days HVFAC showed higher increase in compressive strength. This can be explained with the fact that the pozzolanic reaction needs time and takes place after the beginning of hydration, approximately at the age of 7–14 days or later [16], [17]. The pozzolanic reaction was more pronounced from 7 to 28 days compared to period from 28 to 90 days, as a consequence of small amount of used Portland cement (150 kg/m$^3$ and 200 kg/m$^3$). The extent of the pozzolanic reaction in HVFAC depends on the available $Ca(OH)_2$ and water content while lower increase in compressive strength of HVFAC mixture can be explained with high FA amount and, possibly, not enough Portland cement. It can also be noticed that the influence of different curing conditions was less pronounced at early ages than in later ages, for both concrete types and especially for the OPC mixtures. This can be explained with the fact that all samples were stored in moulds for the first 24 hours and had enough water for early age strength development.

It can be seen from Figure 4. that the application of curing compound based on the polyolefin emulsion did not result in the preservation of the concrete compressive strength compared to water-cured samples, regardless of the concrete type. The L-samples and C-samples had lower compressive strength compared with the water cured samples (up to 27%). However, the use of compound based on the polyolefin emulsion reduced the compressive strength up to 10% compared to water-cured samples.

In the absence of necessary moisture content, the compressive strength decrease in the case of HVFAC samples was more pronounced compared to the OPC samples. On the other hand, all samples cured with curing compound had higher compressive strength compared to air cured samples. The influence of different curing conditions on the compressive strength analysed in this study was similar for both OPC and HVFAC mixtures. In the case of OPC samples treated with curing compound compressive strength was up to 13% higher compared to air cured samples, while in case of HVFAC samples this increase was up to 22%.

Concrete flexural tensile strength was measured using three-point bending test (Figure 5). The measured flexural tensile strength values at age of 450 days are shown in Figure 6. Flexural tensile strength follows the trends described for compressive strength. OPC samples had higher flexural tensile strength compared to HVFAC samples, for specimens cured in a same way. Differences between OPC and HVFAC samples were up to 45%, which was significantly higher than in the case of compressive strength. The highest values of flexural strength were noticed for water cured samples, while the lowest for air cured samples.

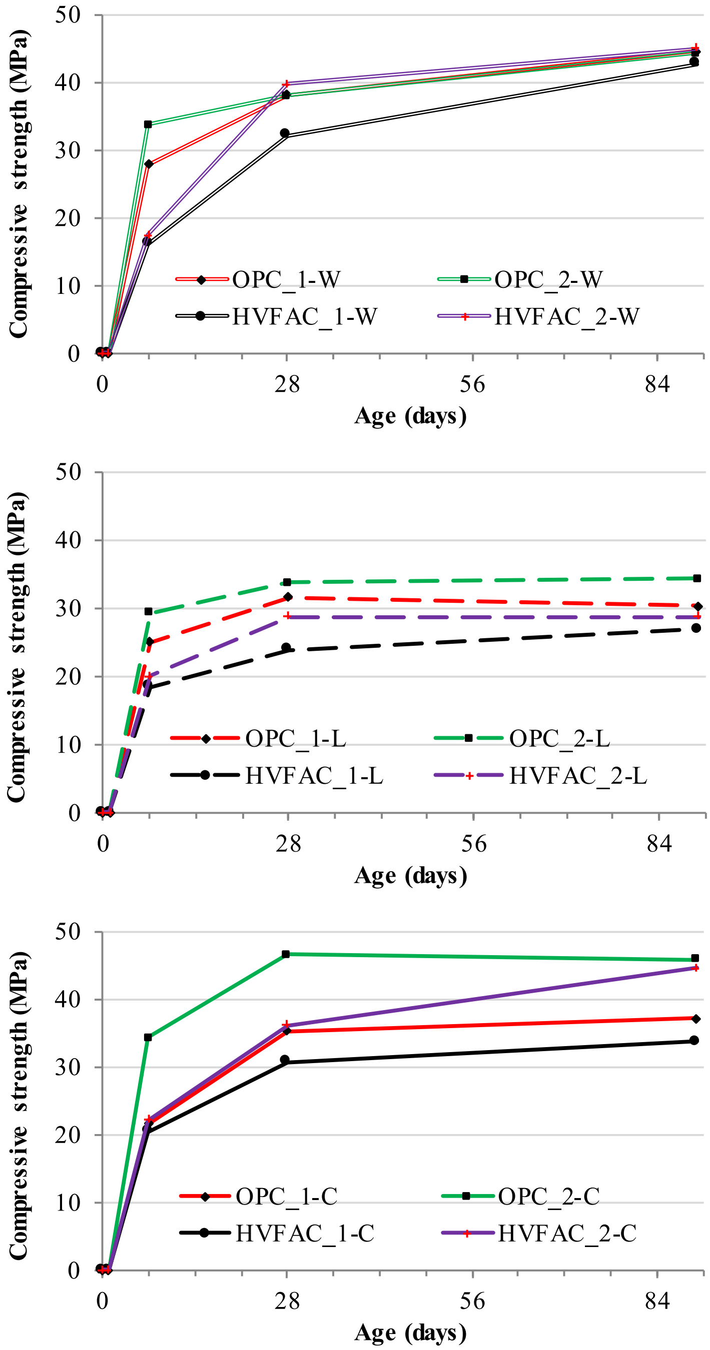



Figure 4. *Compressive strength of differently cured OPC and HVFAC mixtures*

Figure 5. *Three point bending test*

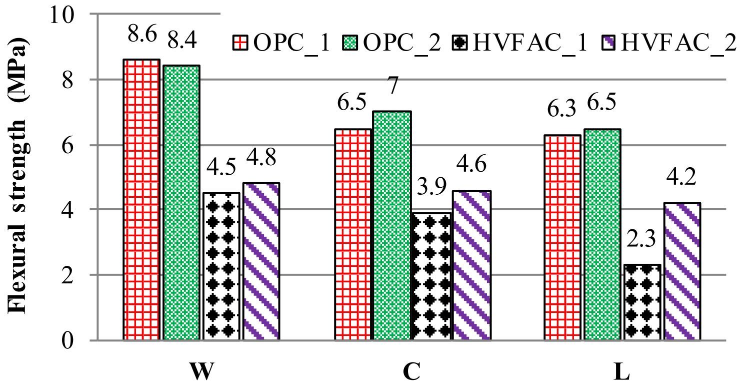



Figure 6. *Flexural tensile strength of differently cured OPC and HVFAC mixtures*

No significant difference between air cured and compound cured samples can be noticed-the differences were up to 9%. The only significant difference was in the case of HVFAC_1 - up to 41%.

**3.3. Modulus of elasticity**

The modulus of elasticity was tested at the age of 3, 7 and 28 days (Figure 7). The test was conducted in accordance with SRPS EN 12390-13:2015 [18]. Figure 8 shows the effect of different curing conditions on the modulus of elasticity.

Figure 7. *Measuring of modulus of elasticity*

Similar like in the case of compressive strength, the W-samples had the highest values of the modulus of elasticity at the age of 28 days. For the OPC mixtures, the L-samples and C-samples had lower 7-day modulus of elasticity compared with the W-samples, for 19% and 10%, respectively. These differences were slightly lower at the age of 28 days: 13% and 9% lower modulus of elasticity for L-samples and C-samples compared with the W-samples was obtained, respectively.

This difference was similar for HVFAC samples. The L-samples and C-samples had lower 7-day modulus of elasticity compared with the water-cured samples, for 15% and 2%, respectively. At the age of 28 days, these differences were slightly higher: 23% and 15% lower modulus of elasticity for the L-samples and C-samples compared with the W-samples was obtained, respectively. HVFAC had slower increase of modulus of elasticity in the first seven days compared with OPC samples. After 7 days the increase was more pronounced for HVFAC, especially for the water-cured samples.

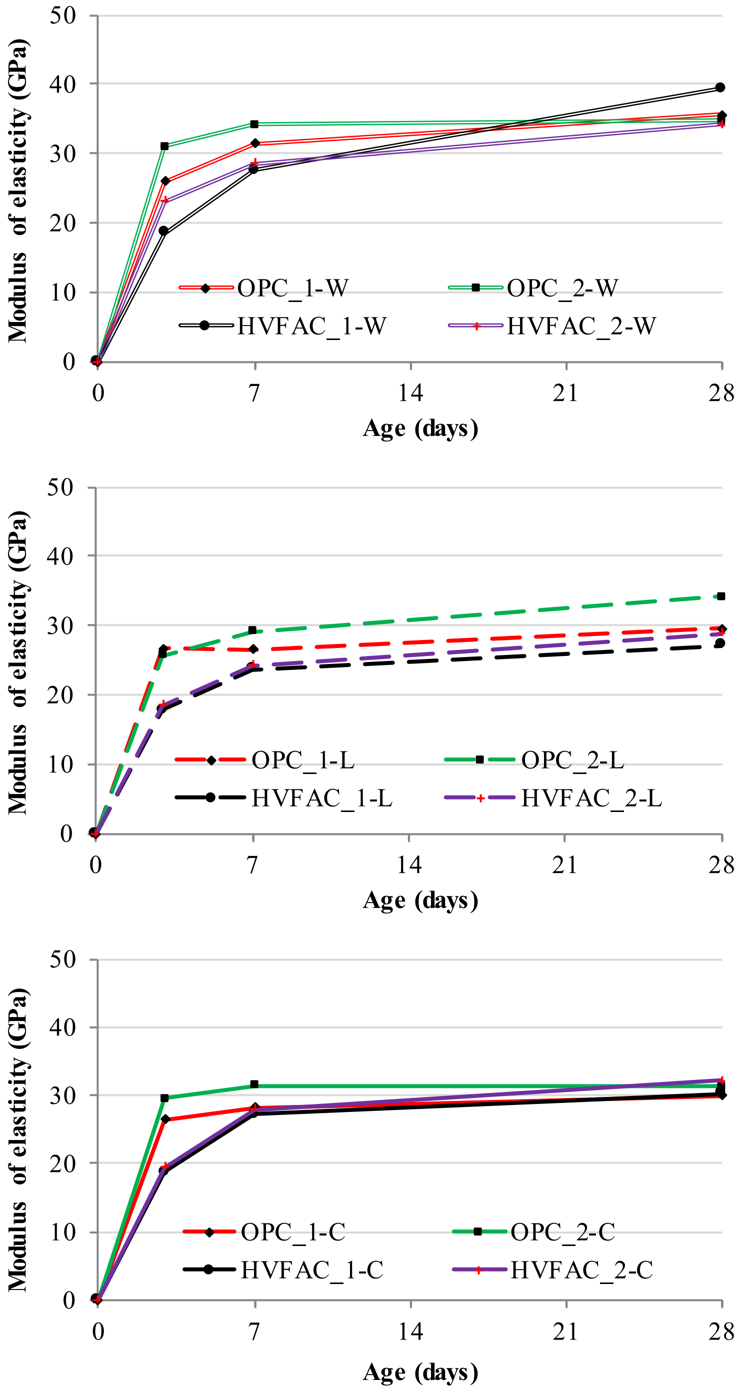



Figure 8. *Modulus of elasticity development of differently cured OPC and HVFAC mixtures*

The values of the modulus of elasticity at age of 28 days were compared with the values calculated by the empirical equation (1), as defined in EN 1992-1-1 [19]:

$$E_{cm}=22\left[\frac{f_{cm}}{10}\right]^{0.3} \qquad (1)$$

where:

$f_{cm}$ (MPa) - 28 days mean compressive strength,

$E_{cm}$ (GPa) - modulus of elasticity at age of 28 days.

The differences between measured and calculated values range within 5%, for all curing conditions. This means that the empirical procedure defined in EN 1992-1-1 [19] can be applied for OPC and HVFAC cured on a different ways.

### 3.4. Shrinkage

Shrinkage measurement started at age of 7 days. Temperature and humidity conditions in the laboratory during the measurement of shrinkage was recorded and shown in Figure 9.

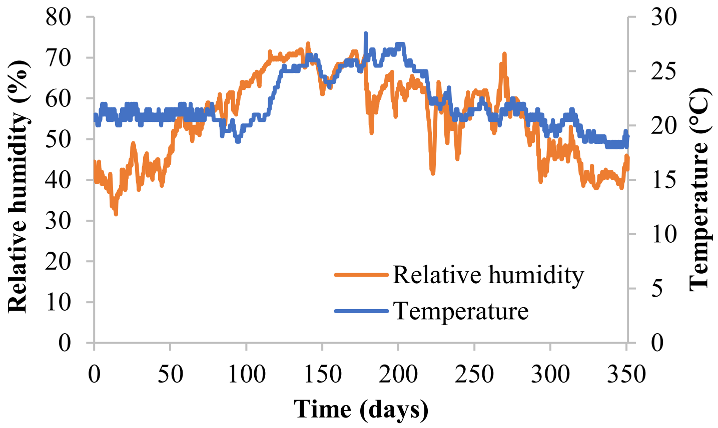



Figure 9. *Temperature and humidity conditions in the laboratory during experiment*

The average value of shrinkage measurements for air-cured samples and compound-cured samples are presented in Figures 10 and 11, respectively. As expected, HVFAC samples had lower shrinkage values compared to OPC because these concretes were made with lower cement amount. HVFAC_1 had the lowest shrinkage as a consequence of lowest cement amount.

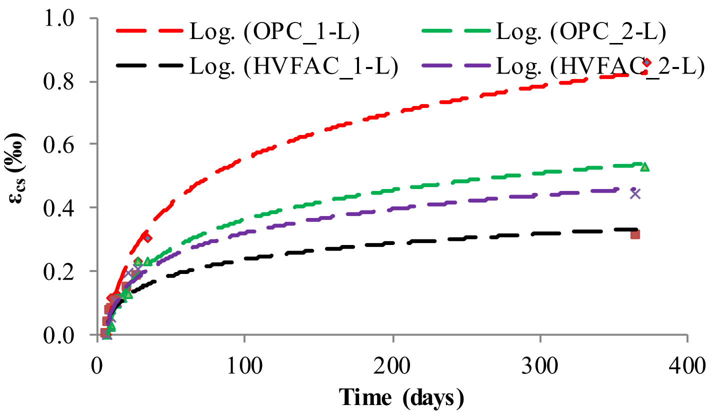



Figure 10. *Shrinkage of air cured OPC and HVFAC samples*

OPC samples cured with compound based on the polyolefin emulsion had lower shrinkage compared to air cured samples. On the other hand, the opposite result was obtained with HVFAC.

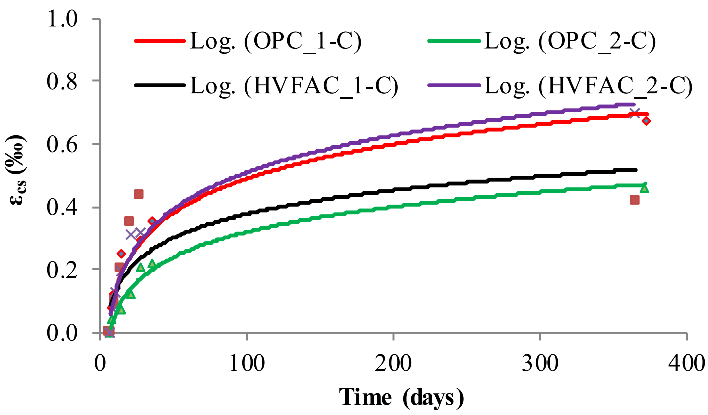



Figure 11. *Shrinkage of OPC and HVFAC samples cured based on the polyolefin emulsion*

### 3.5. Water permeability test

The testing of concrete water permeability was performed according to the SRPS EN 12390-8: 2010 [20]. Tested samples were exposed to water at a pressure of 5 bars for 72 hours in the laboratory conditions (T=20±2°C and RH=60%). After being exposed to water, the samples were broken and the maximum water penetration depth was measured. The obtained results are shown in Figure 12.

According to the presented results, it can be concluded that, in this particular case, the curing conditions had the highest impact on the concrete water permeability. Samples cured in water had low penetration depths (18-25 mm) which correspond to waterproof class V-III (OPC_2, HVFAC_1 and HVFAC_2) and V-II (OPC_1) according to SRPS U.M1.206:2013 [21]. In the case of concretes cured in other two ways, considerably higher water penetrations depths were measured. Samples cured with compound based on polyolefin emulsion had water penetration between 48 and 138 mm and these concretes cannot be classified as water-resistance concretes (except HVFAC_2 which correspond to waterproof class V-I). Even deeper water penetration depths were exhibited in air cured samples (between 128 and 138 mm).

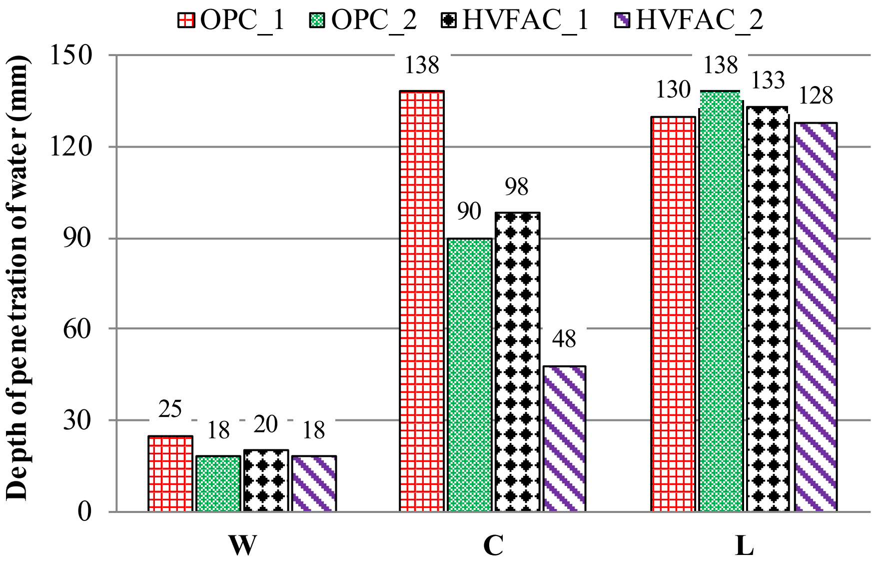



Figure 12. *Maximum depth of water penetration*

The importance of water penetration is reflected from the durability point of view, since the water penetration is directly related to concrete open porosity. This means that in case of air cured or cured with compound based on polyolefin emulsion it can be expected higher chloride penetration or carbonation depth.

## 4. CONCLUSION

In order to support the implementation and promotion of sustainable development and the importance of concrete curing conditions different tests were conducted on the OPC and HVFAC mixtures. Based on the presented results, the following conclusions can be made:

Concrete samples cured in water had the highest compressive strength. For HVFAC water/moist curing is recommended in order to achieve adequate compressive strength.

- For both types of concrete, water-cured samples had significantly higher flexural tensile strength and modulus of elasticity compared with the L- and C-samples.
- HVFAC samples had lowest shrinkage as a consequence of lower cement amount compared to OPC. OPC samples cured with compound based on the polyolefin emulsion had lower shrinkage compared to air cured samples. The efficacy of curing compound was lower in HVFAC samples.
- The curing conditions had the most significant impact on concrete water permeability. The OPC and HVFAC samples cured in water had the lowest water penetration. Samples cured with compound based on polyolefin emulsion had lower water penetration compared to air cured samples. However, in both cases of curing, penetration depths for L- and C-samples

was too high regardless of concrete type and these concretes cannot be classified as water-resistance concretes.

Generally, the use of the curing compound based on the polyolefin emulsion improved concrete properties (except water resistance) compared to air cured.

## ACKNOWLEDGEMENTS

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