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EKSPERIMENTALNO ISPITIVANJE OSNOVNIH SVOJSTAVA BETONA ZA 3D ŠTAMPU

Rezime: *Predmet rada je eksperimentalno ispitivanje svežeg betona koji će se koristiti prilikom 3D štampe. Razmatrane su različite vrste mešavina sa i bez hemijskih dodataka (superplastifikator i kontrolor hidratacije) pri različitom vodovezivnom faktoru. U svežem stanju ispitivana je zapreminska masa, vreme vezivanja i konzinstencija metodom rasprostiranja u toku vremena. Granična nosivost na pritisak i savijanje određenja je na uzorcima kocke i prizme pri različitoj starosti. U zaključku rada pokazan je uticaj hemijskih dodataka na ispitana svojstva. Mešavina sa vodovezivnim faktorom 0.45 i učešćem od 13.8% kontrolora hidratacije postiže optimalne karaktristike betona za 3D štampu.*

Ključne reči: *beton, 3D štampa, zapreminska masa, vreme vezivanja, konzistencija, granična nosivost*

EXPERIMENTAL INVESTIGATION OF BASIC CONCRETE PROPERTIES FOR 3D PRINTING TECHNOLOGY

Summary: *The aim of this paper is an experimental investigation of fresh concrete properties of the mixture designed for 3D printing. Different mixtures with and without chemical additions (superplasticizer and hydration controller) with variable water-binder factor were considered. Density, time setting and workability flow/slump tests were determined in the fresh state. Ultimate capacities for compressive and flexural strength were analysed on cube and prism specimens at different age. The effect of chemical additions on properties was shown in the conclusion of this paper. The mixture with the waterbinder ratio 0.45 and amount of 13.8% of hydration controller had obtained the optimal properties for 3D printing.*

Key words: *concrete, 3D printing, density, time setting, workability, ultimate capacity*

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1. INTRODUCTION

The 21 century has brought with it the comprehensive implementation of modern digital processes in all branches of the economy. One successful example of this is the use of three-dimensional (3D) printing technology. 3D printing technology was first conceived in the 80s, as a result of the need for simplifying the process of creating complex elements of various shapes and sizes. Complicated tools and equipment were replaced by sophisticated technologies such as 3D printing. This led to significant savings in both time and resources [1].

The basic idea is to create an element by applying material in layers according to the desired path, which is generated on the basis of a 3D computer model. Fresh material from the reloading silo is pumped through the hose to the printhead through which it is extruded. The movement of the printhead follows the path defined by the G-code obtained on the basis of a 3D computer model of the element to be printed. This process is often known in the literature as "Additive manufacturing" (AM-gradual manufacturing). Initially, it was applied to materials such as metals, polymers, ceramics, textiles and other composite materials. [2]

The beginning of the application of 3D printing technology in concrete structures is associated with Behrokh Khoshnevis from the University of Southern California. Khoshnevis has patented one of the basic methods for 3D printing concrete - "Contour Crafting" (CC) [2]. Other common concrete printing methods include: Fused Deposition Modeling (FDM), Ink printing, Spraying method. [2]

Although it is called 3D printed concrete, the material currently being printed has an aggregate particle of less than 3 mm, and accordingly the term mortar is often used in the literature instead. In addition to the fine aggregate, which is often divided in two or three fractions, a mandatory component is cement (different types of CEM I / CEM II have been used so far), as well as a large number of different chemical additives. [3]–[8]

The advantages of 3D concrete printing technology are as follows: the possibility of higher construction speed, the absence of formwork, material savings, the need for a smaller number of workers, safer working conditions, and the possibility for sustainable development. While reviewing the literature, the following shortcomings were observed: higher initial investment, need for qualified labour, insufficient knowledge of the quality of materials, lack of regulations and standards for the design and construction of these structures, details during execution etc. [2], [9]–[11].

Despite previously mentioned shortcomings, the application of these technologies in the construction yielded significant results. Various constructions were made by using 3D printed concrete, such as: family houses, multi-story buildings, pedestrian bridges, as well as individual elements in the form of pillars, walls, but also non-constructive elements such as sculptures, planters, outdoor furniture and the like [2], [4], [10].

Within this work, the emphasis is placed on the properties of concrete mixtures used for printing. The aim was to made a mixture with optimal setting time of approximately 2h and plastic consistency. It was assumed that it would provide adequate printability properties. In order to reach these targeted values, the amount of water and chemical additions were varied while the amount of other components remain unchanged.

2. BASIC CONCRETE PROPERTIES FOR 3D PRINTING TECHNOLOGY

Basic properties of concrete intended for 3D printed concrete can be selected into the following groups:

1. Properties of fresh concrete mix before the printing process - essentially, they fully correspond to the properties of ordinary concrete: bulk density, setting time and consistency. Values of these properties can be a good indication of whether the fresh concrete mix is suitable for 3D printing, i.e. “printable”.

2. Properties that are typical for 3D printed concrete and which are quantified during the printing process: a) "flowability" (or in some references "pumpability") – the property that allows the fresh mixture to move through the hose to the printer's nozzle, b) "extrudability" – the property of passing the fresh mixture through the printer's nozzle without impairing the quality of the layer being printed, c) "buildability" – the property which refers to ability of printed layer to withstand the weight of the layers above without excessive deformations [3], [4], [10]. All together, these properties define the general one- “printability”.

Apart from properties in these two groups, rheological properties (shrinkage and creep) are also important in the case of 3D printed concrete, especially due to the higher amount of cement compared to the ordinary one.

There are still no standardized methods for testing of concrete intended for 3D printing. Based on a review of the literature, it seems that the test methods applied to standard mortars have been used to test these properties.

Testing the bulk density of 3D printed concrete is significant for its hardened state. It has been shown that high quality printed samples have higher density compared with the samples casted in a moulds [12]. These conclusions were based on 10-cm cubic samples cut from printed boards (35x35x12 cm in size) for the case of printed concrete.

The consistency of the fresh concrete mixture- “flowability” is quantified using the same methods that are applied in determining the consistency of mortar and concrete mixtures: slump/flow tests, the flow table test, V-funnel test, squeeze flow test and the rheometer test [3]. Chen et al. [6] investigated the effect of viscosity modifying admixture (VMA) additives on the “printability” of concrete mixtures with added calcined clay. The spreading diameter of the fresh concrete mixture was determined using the spreading method on a shaking table according to ASTM C1437–15. The mixture with the best printing properties had a spreading diameter of 140 mm after 10 minutes and 115 mm after 120 minutes. The reference mixture with a spreading diameter of 300 mm was assessed as not adequate for printing [6].

The setting time of the fresh concrete mixture determines the property called "open time", which is defined as a maximum period of time during which the fresh concrete mixture can be pumped through the printer head. This property is determined directly during the printing process, however, it can also be determined indirectly by measuring the consistency using the slump/flow test or the rheometer penetration test, which is commonly applied to mortar mixes [3].

Basic mechanical properties such as compressive strength and flexural strength are determined on specimens defined according to standards which are considered reliable when applied to traditional concrete. In some studies, strength values were compared between samples obtained by 3D printing and casting in moulds [3,13]. For samples obtained by 3D printing, direction of force action has significant influence on the measured values of mechanical properties [3,13]. The force can be applied vertically, longitudinally and horizontally in relation to the direction of layering [3]. Contrary to this, Hirsch et al. [13] showed that in the case of compressive strength testing, no significant differences in printed and cast samples were observed, while in the case of flexural strength the difference was noticeable.

3. EXPERIMENTAL WORK

3.1. Experimental setup and programme

The experimental tests carried out can generally be divided into two groups. Tests in the fresh state included the determination of the density, setting time and workability of the analysed concrete mixtures. The second group of tests was focused on the mechanical properties of concrete mixtures, such as compressive strength and flexural strength. The samples used in the test were 10-cm cubes and prisms with dimensions of 4x4x16 cm. The age of samples testing was 7 and 28 days. The mentioned mechanical characteristics were tested on samples that were not obtained by printing, but were cast in a mould.

3.2. Mix design

The testing samples were made from a mixture that is a product of the company NaturaECO®. Basic components such as cement, microsilica, quartz sand in three fractions and polypropylene fibers are mixed in the dry state. In this way, a ready-made "premix" was formed, which was delivered in 30 kg bags.

When making a fresh concrete mixture, the dry "premix" is mixed with water and chemical additives, with an approximate mixing time of five minutes. By varying the amount of water, as well as the amount of various chemical additives, a total of nine different concrete mixtures were formed for the purposes of this research, which are marked with the letters A-I. The chemical additives used in the mixtures were superplasticizer MGlenium ACE 770 and the hydration controller Master ROC SA 167. The detailed composition of all test mixtures is shown below in Table 1. It should be noted that the test mixtures have two values for the water binder factor 0.45 and 0.50.

Mixture	A	B	C	D	E	F	G	H	I
„Premix“ (g)	15612	15612	15612	15612	15612	15612	15612	15612	15612
Water (g)	2376	2640	2376	2640	2376	2640	2640	2376	2376
MGlenium ACE 770 (g)	-	-	40	40	40	20	20	40	40
Master ROC SA 167 (g)	-	-	-	-	8	-	8	46.4	13.8

Table 1. Mix designs of 3D printed concrete mix

3.3. Test results and analysis

3.3.1. Density of concrete

The density of all test mixtures was determined in the fresh state, immediately after casting in the moulds and in the hardened state, after 24 hours. The bulk density test was performed according to the SRPS EN 12390-7:2019 [14] on 10-cm cube samples. The results are shown in Figure 1.

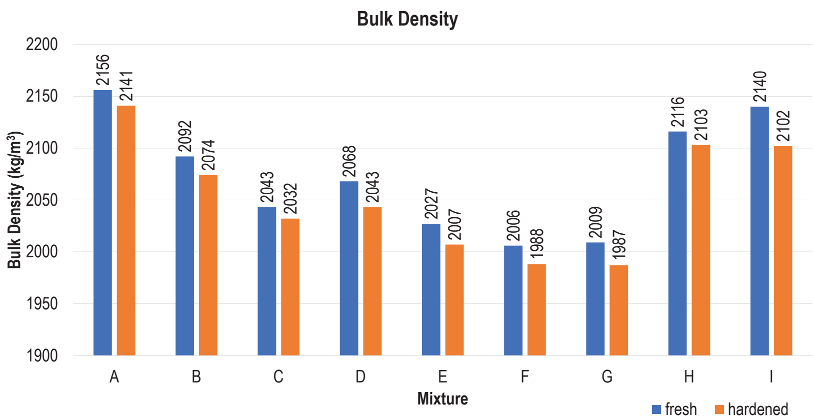

Figure 1. Value of density of the concrete in fresh and hardened state

It can be seen that for all mixtures the density in the hardened state is lower than in the fresh state, which is a consequence of the evaporation of water. The values vary by mixture in a range of only a few percentage. The volumetric mass of all mixtures in the fresh state was above 2000 kg/m^3, while in the hardened state was above 1980 kg/m^3. This values are within the range of 1800-2200 kg/m^3 that corresponds to the results obtained from ordinary mortars.

3.3.2. Time setting and workability

As part of this research, the test methods that were used to determine the setting time and consistency were taken from the following standards: SRPS EN 1015-3:2008 [15] and SRPS EN 1015-9 :2008 [16]. The consistency of the fresh concrete mixture was determined on a shaking table, where the diameter of mixture spreading was measured in two perpendicular directions, after 15 impacts. The setting time was determined as the time in which the value of spreading diameter decreased by 30 mm. The test procedure is shown in Figure 2, and the test results in Figure 3.

a) b)

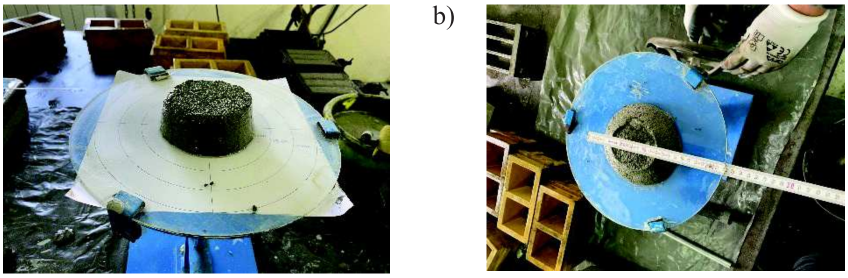

Figure 2. Flow table test: a) sample before testing, b) measurement of diameter

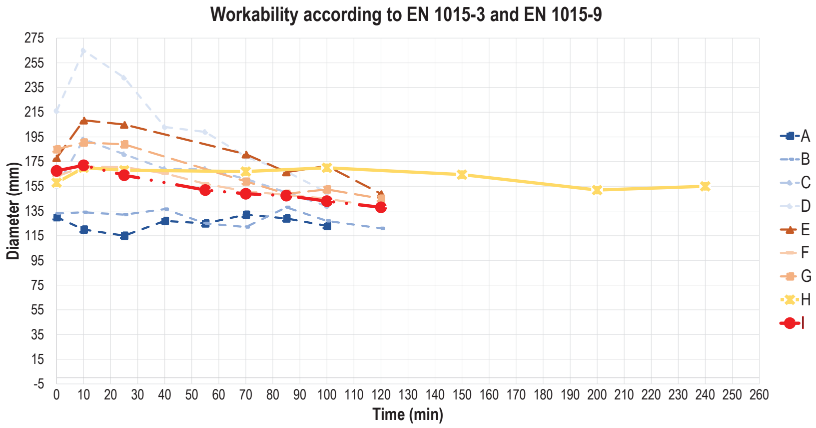

Figure 3. Flowability test results of concrete mixture

Based on Figure 3, it can be concluded that the amount of hydration controller in the fresh concrete mix has an influence on the consistency over time. The aforementioned additive has the role of delaying the beginning of the setting time and ensuring the same characteristics of the concrete mixture over time.

The setting times for all the test mixtures are also shown below in Table 2. Mixture H, which has a high amount of hydration controller, showed no changes in the consistency during the period in which the spread diameter measurement was performed.

Mixture	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
First diameter (mm)	120	134	193	265	209	171	191	170	170
End diameter (mm)	123	121	161	203	167	138	159	155	138
Setting time (min)	>100	>120	70	40	85	120	70	>240	120

Table 2. Setting time for analysed concrete mixture

As shown in Table 2 no final values for setting time were measured for mixtures A, B and H. For mixtures A and B, based on Figure 3, it can be concluded that hardening would start in the near future. A substantial amount of hydration controller was used for Mixture H. The amount used corresponds to half of the maximum dosage specified by the manufacturer's specification. This is enough to delay the start of bonding by 48 hours, according to the manufacturer's recommendations.

3.3.3. Compressive strength

In addition to affecting the load-bearing capacity of the structure itself, concrete strength significantly affects the quality of printing. It is necessary to obtain an adequate increase in strength over time, that will allow the lower layers to withstand the load from the layers above without excessive deformations.

The compressive strength was tested according to the standard SRPS EN 12390-3:2014 [17]. The test was performed on a 2000 kN–capacity Matest press on 10-cm cube samples A comparative analysis was carried out by comparing the strengths of concrete mixtures at different ages (7 and 28 days). The results are shown in Figure 4.

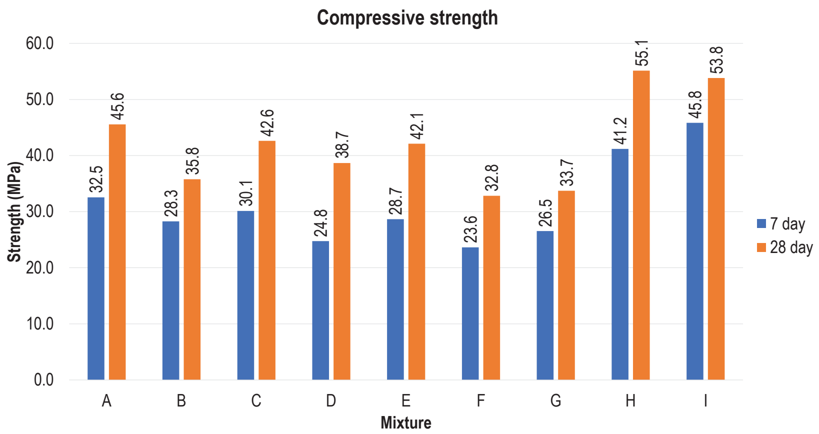

Figure 4. Test results for concrete compressive strength

The values vary depending on the mixture, and based on Figure 4, it can be concluded that the mixtures with higher water-to-binder ratio have lower compressive strength values, as expected. Also, the highest values were obtained for mixtures H and I, in which the presence of chemical additives is significant, especially hydration controllers. An increase in strength over time was observed for all mixes.

Based on the average strength value of three samples at the age of 28 days according to the standard SRPS EN 206:2011 [18], Table 3 shows the mean concrete strength and classes (C) for all test mixtures.

Mixture	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
$f_{cm,cube,10}$ ***(MPa)***	45.6	35.8	42.6	38.7	42.1	32.8	33.7	55.1	53.8
f_{cm} ***(MPa)***	34.7	27.2	32.4	29.4	32.0	24.9	25.6	41.9	40.9
f_{ck} ***(MPa)***	26.7	19.2	24.4	21.4	24.0	16.9	17.6	33.9	32.9
Class (C)	C25/30	C16/20	C20/25	C20/25	C20/25	C16/20	C16/20	C30/37	C30/37

Table 3. Concrete classes for mixtures

The highest obtained strength classes C30/37 correspond to mixtures H and I, which have lower water-to-binder ratio of 0.45 and a significant amounts of chemical additives, compared to other mixtures.

3.3.4. Flexural strength

The last set of tests was focused on the analysis of the flexural strength. Flexural strength was tested according to SRPS EN 1015-11:2008 [19]. The age of the samples during testing was 28 days.

The flexural strength values for all tested mixtures are shown in Figure 5. It is shown that the water-to-binder ratio has an influence on the values. Lower strength values were obtained for mixtures with higher water-to-binder ratios. The obtained values for all mixtures were higher compared with the ones usually obtained for ordinary cement concrete. However, it should be noted that the bending tension test was used, which as a rule results in higher values compared to all other tension tests.

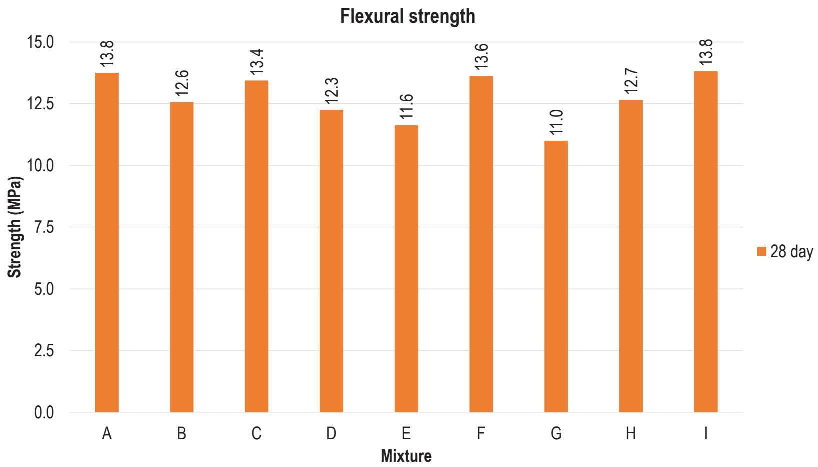

Figure 5. Test results for concrete flexural strength

4. DISCUSSION AND CONCLUSIONS

Structures and elements that have been successfully built in 3D printing technology are the motivation for further development of this technology. There is an increase in number of professional and scientific research organizations that work to eliminate shortcomings, improve and popularize the entire process.

Based on results from the conducted laboratory tests, the following conclusions can be drawn:

- There are still no standardized methods that are exclusively used for concrete intended for 3D printing. The material properties of concrete should be considered separately from the properties that are characteristic for the printing process (buildability, extrudability, open time,...) to get some input information about concrete properties with regard to printability. Also, there are no explicit connection between these properties and the properties of the printed concrete.

- Examination of the density in the fresh and hardened state showed that the values between the mixtures in both conditions differ by several percentage points depending on the amount of components. In the fresh state, the value of the volumetric mass was in the range of 2006-2156 kg/m^3, while in the hardened state it was 1987-2141 kg/m^3. Based on the value in the hardened state, which for all mixtures was in the range of 1800-2200 kg/m^3, it can be concluded that all considered mixtures belong to the mortar category [3].
- The influence of chemical additives, especially hydration controllers, is especially pronounced. It was shown that mixtures with a larger amount of this chemical additive had a delayed starting point for setting, as in the case of mixture H. The final setting time was not measured for mixtures A and B, while for the remaining six mixtures, setting time was obtained in the range of 40-120 min. The consistency of the test mixtures was quantified through the value of the spreading diameter according to the flow table test.
- Based on the measured values, mixtures A and B have a plastic consistency, while mixtures D and E have a liquid consistency according to the provisions of the SRPS EN standard. All the remaining mixtures have a plastic-liquid consistency, as their spreading diameter value is in the range of 140-200 mm.
- Strength values at the age of 7 days for all mixes were in the range of 23.6-45.8 MPa, while at 28 days it were 32.8-55.1 MPa. It has been shown that those mixtures that have a higher water-to-binder ratio had lower compressive strengths. The highest values of the compressive strengths at 28 days were obtained for mixture H, which had 55.1 MPa. The considered mixtures belong to concrete classes in the range C16/20-C30/37.
- For all mixtures, the obtained flexural strength values at the age of 28 days were in the range of 11.0-13.8 MPa. It was shown that mixtures with higher water-to-binder ratio had lower strength values, except for mixture F, which had higher water-to-binder ratio of 0.5 but also higher strength. The flexural strength values are much higher than accustomed for cement concrete.
- As it is a preliminary test of mixtures that are intended for printing, of all the considered mixtures, mixture I has the greatest potential (setting time about 2h, plastic consistency and class C30/37). It has been shown that it has an adequate density, setting time and mechanical properties.

For further popularization and expansion of the application of 3D printing technology in modern design, it is necessary to work on solving the previously mentioned shortcomings and open questions. In this context, the primary task is to define the relevant standards for testing of basic properties of this kind of concrete in its fresh and hardened state and this process is under development by RILEM organization with two technical committees (TC PFC and TC ADC).

5. ACKNOWLEDGEMENTS

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