PROPRIETĂȚILE MECANICE ALE BETONULUI ULTRACOMPACTANT DE FOARTE MARE PERFORMANȚĂ CU DIFERIȚI ADITIVI MINERALI MECHANICAL PROPERTIES OF ULTRA HIGH PERFORMANCE SELF COMPACTING CONCRETE WITH DIFFERENT MINERAL ADMIXTURES

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Modul de obținere și microstructura betonului autocompactant de înaltă performanță au fost intens studiate în ultimul timp. Proprietățile mecanice (rezistența la compresiune și rezistența la încovoiere) au fost investigate în diferite condiții de întărire (standard și cu abur). Silicea ultrafină are caracteristicile prin care dobândește abilitatea de constituient al compozitelor de înaltă rezistentă, pe bază de ciment. Utilizarea din ce în ce mai frecventă a betonului autocompactant de înaltă performanță justifică necesitatea substituirii cimentului cu adaosuri active. În acest articol este pusă în discuție posibilitatea realizării betonului autocompactant de înaltă performanță cu materiale disponibile în Serbia, pe baza activităților experimentale a autorilor. Sunt prezentate trei serii de probe cu adaosuri reactive diferite (silicea ultrafină a fost substituită cu metacaolin în proporție de 20% și 40% și cu cenușă zburătoare în proporție de 20%). Amestecurile au avut consistența specifică betonului autocompactant. Rezistența la compresiune la 28 de zile a variat între 165 și 195 MPa, pentru probele tratate termic și între 135 și 150 MPa pentru probele cu întărire standard. De asemenea, sunt prezentate micrografiile SEM ale fazelor C-S-H formate în condițiile întăririi aceelerate, cu abur la presiunea normală și în autoclavă.

Preparation and microstructure of ultra high performance concrete (UHPC) have been intensively analyzed in the last few years. The mechanical properties (compressive strength and flexural strength) were investigated under different curing conditions (standard and steam curing). Silica fumes have characteristics that make them necessary in cement composites with ultra strength properties. The search for substitute products thus appears important if the use of UHPC is to become more widespread in the concrete industry. Possibilities of making ultra high performance self compacting concrete (UHPSCC) with materials available in Serbia, based on experimental work are discussed in this paper. Three series of samples were made with different types of fine reactive additives (silica fume was replaced with metakaolin at 20% and 40% and with fine fly ash at 20%). The produced mixes had self compacted consistency. The 28th day compressive strength varied between 165 and 195 MPa for the heat treated specimens and between 135 and 150 MPa for the ones that had not been heat treated. Furthermore, this paper presents SEM micrographs of C-S-H phase formed after steam curing and super-heated steam under preassure.

Keywords: UHPSCC, Fine reactive additive, Steam curing regime, Super-heated steam under preassure

1. Introduction

Modern research in concrete technology pays attention to the development of materials with improved mechanical properties and increased durability [1]. The use of cementitious composites with high mechanical properties and improved ductility allows building reinforced concrete structures with reduced cross-section dimensions and increased earthquake resistance. Reduced cross-section dimensions, improved durability and extended lifecycle of structure save natural resources and protect the environment.

Ultra high performance concrete (UHPC) is a modern composite material which was the outcome of the techniques of improving the microstructure and it is an interesting subject for researchers from all over the world. UHPC has extremely good mechanical properties (compressive strength greater than 150 MPa) and compact microstructure due to the high content of cement in it, very low water/binder ratio, reduced C_aO/S_iO_2 ratio in

cement matrix because of the high content of silica fume and better particle size distribution of aggregates and quartz powder [2]. Most cement manufacturers marketed different kinds of UHPC like BSI (special industrial concrete developed by Eiffage), different kinds of Ductal developed by Lafarge, BCV developed by Vinci group [3].

The world's first engineering structure designed with UHPC was the Sherbrooke footbridge in Sherbrooke, Quebec, built in 1997. Seonyu Pedestrian Bridge in South Korea was built 2002. There have been many constructions of UHPC for highway bridges in the United States, performed by the FHWA (Federal High-Way Administration). In France, recommendations for the use of UHPC reinforced with steel fibers have been issued in 2002. The n°34 overpass on the A51 highway with single span 47.4m long and The Bridge of St Pierre La Cour in Mayenne region in France were built in 2005 [4]. The Jakway Park Bridge in Buchanan County in Iowa is the first North American highway bridge built with UHPC.

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The bridge opened to traffic on November 2008.

2. Literature review

2.1 UHPSCC properties

The properties of concrete such as strength or durability significantly depend on the properties of the cement paste, and the bond behavior of cement paste and aggregates. The characteristics of the cement paste and the bond behavior between aggregate and cement paste depend on many factors such as effective water/cement ratio, reactivity of cement and mineral admixture, and particle size distribution of the powders [5].

The mix compositions of UHPC are characterized by high cement, superplasticizer and mineral admixtures content. Compared to normal concrete, significant improvements of UHPC include the particle size homogeneity, porosity and microstructures. The UHPC mixture improves homogeneity by using no coarse aggregate (particle size of sand is between 150 and 600 μ m) [6].

In general, water demand of cementitious composites depends on the specific surface area and the packing density [7]. Quartz powder used in generally differs from quartz sand in UHPC particle size and mineral composition and often lead to a higher water demand and reduced workability of concrete. The water demand of powders and the finest particles in concrete are significant parameters for the design of concrete. It is composed of a layer of adsorbed water molecules around the particles and an additional amount needed to fill the intergranular voids of the Therefore, the appropriate powder system. determination of the amount of water, needed to cover all particles with a water layer of a certain thickness is important. Besides the degree of hydration as well as cement type and amount, the water content is mainly responsible for the percentage of capillary pores, which is a direct indicator for the durability of concrete and mortars.

The fines fraction of the aggregate is acknowledged to improve the packing density of concrete when used in right proportions. DeLarrard and Sedran maximized the packing density of the cementitious materials to reduce the water/binder ratio and improved the strength and durability of the composites [7]. Blending cement with appropriate proportions of fly ash and silica fume can increase the packing density of the cementitious materials and increase the flowability of the cement paste formed. To measure the packing density of cementitious materials, Wong and Kwan [8] have developed a method, called the wet packing method, which mixes the cementitious materials with water, measures the solid concentration of the cement paste formed at varying water/binder ratio and determines the packing density of the cementitious materials as

the maximum solid concentration achieved. [9]. Very fine recycled glass powder can be used as mineral admixture [10] and can have a positive influence on a denser microstructure in concrete.

The binder content of UHPC is 4 times higher when compared to normal concrete, which leads to more than 10 times higher admixture The application content. of modern superplasticizers allows high water reduction, good workability of fresh concrete and achieving extraordinary properties mechanical of composite. The spread-flow test (or sometimes referred to as paste line test or mini-slump flow test) according to Okamura and Ozawa [11] appears to be the classical method for the determination of the water demand of powder materials involved in SCC.

In order to ensure the required selfcompacting properties of UHPC, it is essential to choose the right amount and type of superplasticizer and optimized mixing procedure. The addition of steel fibers increases the ductility of concrete, which is most important for very brittle materials. Some researches show that the ductility of UHPC is about 250 times higher than the ductility of conventional concrete.

In constructions made using such concrete, mixing times longer than 4 min for SCC and over 12 min for UHPC were not uncommon. The output of UHPC and SCC at the production plant is below normal vibrated concrete which is a decisive economic factor. Long mixing times are essentially due to the special composition of these kinds of concrete where very low w/c ratios (0.25) and high amounts of superplasticizer and fine mineral additions are used. The effect of concrete composition and tool speed on the time needed to mix UHPC and SCC has been investigated in the research of Mazanec Lowke and Schießl [12]. The water-binder ratio, powder grading and the amount of superplasticizer in a fine-grained UHPC varied systematically. The necessary mixing time (stabilisation time) was determined from the power supplied to the mixing tool during the mixing process. The effect of tool speed was investigated using an UHPC mix. Doubling the tool speed from 1.4 to 2.9 m/s reduced the stabilisation time by approximately 11%. The speeds which was four times higher led to a reduction of 16%. The effect of tool speed is included in the empirical model which thus permits the calculation of the stabilisation time of a UHPC mix for the given composition, particle size distribution of the constituents and tool speed.

The research of hybrid beams and parts of structures is receiving considerable attention. They optimize the share of UHPC in construction and try to economically justify the use of this type of composite. Potential use is in the production of special precast prestressed concrete elements.

2.2. Microstructure and phase development in UHPC

The fine additives used in cement systems are being classified into reactive and the so-called "inert" substances. The "inert" additives in normal processing conditions (fine guartz, limestone, dolomite etc.) are thought to improve strength only through a physical filling effect: they fill up the void spaces that remain between the coarser particles. The reactive additives (i.e. pozzolans) act through a double mechanism: they tend to physically fill the void space between the larger particles, which is otherwise occupied by water that is not free to contribute to fluidity [13]. Additionally, with time they engage in chemical reactions to produce additional material, combine some of the water in their reaction products and reduce in this way the porosity of matrix and interface transition zone [14].

The compressive strengths of all the blended cements are lower than that of the plain cement paste, during the early hydration (1–3 days). In addition, the strength decreased with increasing the pozzolan content. The reductions in strength as a result of pozzolan substitution are more significant at the low water/binder ratio [15].

Korpa et al. reported a quantitative comparison between the phase development in normal and ultra high performance cementitious systems during 28 days of hydration [16]. It was shown there that qualitatively, there is no great difference between the main phases obtained in both systems. the phase development in ultra high performance cementitious formulation is quantitatively and kinetically different from that in normal concrete formulation. This was related to the different components employed and their associatedreactions. For both formulations the most remarkable changes concerning the phase contents are recorded between the first and the second hydration day and up to the seventh day. After the seventh day less phase content changes are measured. Because of the insufficient water amount for hydration, nearly 13 wt.% (referring to systemweight) of the main cement phase C_3S remains non hydrated in the UHPC system [16].

2.3. Modeling curing regimes

Hydrothermal curing of precast concrete is a traditional technique that reduces the curing time and accelerates the development of the early mechanical properties. Steam curing of UHPC at high temperatures has from the start been the subject of many research. Different curing regimes with applied temperatures of over 300 °C have been reported in the literature. The effect of hydrothermal treatment of UHPC is seen in the formation of an improved microstructure with crystalline calcium silicate phases (C-S-H phases). High temperature exposure affects the reduction of the pores in composite texture and results in the increase of compressive strength, compared to the

samples cured under ambient conditions.

It was reported from Chan and Chu [17] that the steel fiber-matrix bond characteristics are remarkably improved by adding silica fume in RPC matrix, due to the interfacial-toughening effect upon fiber slip. Massidda et al. [18] studied how the physical and mechanical properties of reactivepowder mortars reinforced with brass-coated steel fibers are affected by super-heated steam under preassure at 180 °C. Generally, super-heated steam under preassure is beneficial to the mechanical properties both in terms of flexural and compressive strength. 3 hours of high pressure steam curing of specimens that had been precured at ambient temperature for 3 days resulted in flexural strength of 30 MPa and compressive strength of 200 MPa [19].

The puzzolanic reaction of fly ash is significantly accelerated, so super-heated steam under preassure might be a useful tool to reduce the high cement and silica fume content in UHPC by using secondary cementitious materials. Pure heat treatment at 1 bar leads to the formation of foshagite and xonotlite and Ca-rich phases like jaffeite. However, portlandite is still present at 200°C in this series but the amount is considerably reduced [2,20].

3. Experimental program

The mix design of UHPC significantly differs from that of normal and high-strength concrete. High cement content (usually more than 850 kg/m³) together with the mineral additives content means a significantly higher proportion of binder material. Mechanical properties of UHPC greatly depend on the physical and mechanical properties of cement.

Portland cement (CEM I) of the strength class 42.5 R was used in this study and the chemical, physical and mechanical properties of this cement are shown in Table 1. Although it is declared as cement with the strength class 42.5. properties it can because of its be classified into high performance cement suitable for making composites with exceptionnal mechanical properties. The use of pozzolan is also necessary. It will fill the pores in the cement paste and contributes to the strength of composite by forming the products of pozzolanic reaction with the free calcium hydroxide, the product of primary hydration. Three different types of supplementary cementitious materials were used in this study, i.e. silica fume (SF), а commercial product of company Sika, metakaolin - argical M1000 (MK) and fine ground fly ash (GFA). Their chemical and physical properties are also shown in Table 1.

Quartz powder with average particle size of 50 μ m and quartz sand with Dmax = 0.5 mm were used as aggregate. A modified polycarboxilates

based superplasticizer was used to allow high water reduction and to obtain high early strength.

Table 1

Properties of cement, silica fume, fine blended fly ash and metakaolin / Proprietățile cimentului, silicei ultrafine, cenuşii şi metacaolinului

	Chemical composition, [%]				
		Compoziția	chimică, [%]		
	Cement	Silica fume	FB fly ash	Metakaolin	
	Ciment	Silice	Cenuşă	Metacaolin	
		ultafină			
S _i O ₂	20.51	92.52	53.59	55.10	
AI_2O_3	65	0.64	25.03	40. 2	
Fe ₂ O ₃	2.80	0.31	6.11	1.40	
CaO	63.41	0.38	7.51	-	
M _g O	1.85	0.44	3.02	-	
Na ₂ O	0.29	0.32	0.52	-	
K ₂ O	0.79	0.87	1.28	-	
SO ₃	2.69	0.22	0.72	-	
C	0.003	-	0.00	-	
L.O.I.	2.8	-	2.65	1.00	
I.R.	0.62	-	-	-	
F.C _a O (%)	0.43	-	0.015	-	
Physical prope Proprietățile fi. Specific gravit Densitatea [kg Initial setting ti Timpul inițial d Final setting ti Timpul final de	erties of cer zice ale cim y [kg/m ³] /m ³] ime [min] de priză me [min] e priză	nent <i>lentului</i>	3100 260 330		
Specific surfac Suprafața spe Cement / Cime	ce [m²/kg] <i>cifică</i> e <i>nt</i> (Blain)		413		
Silica fume / S	Silice ultrafin	aă (BET)	17000		
Fine blended (Blain)	l fly ash	/ Cenuşă	330		
Metakaolin / N	letacaolin (BET)	20000		

Compressive strength of cement [MPa]

Rezistența la compresiune a cimentului

30.3

48 6

62.1

2 days / zile

7 days / zile

28 days / zile

Brass coated steel fibers with 8 mm length and a diameter of 0.15 mm were used. The aspect ratio and tensile strength of the fibers were 53.3 and 2200 MPa, respectively.

materials Commercial available in the domestic market were used for preparing concrete Self compacting properties of fresh mixtures. concrete mixtures were corrected by adjusting the content of the superplasticizer. All mixtures were designed to display a slump flow of 275±10mm on the small cone (Fig. 1.). The mixture ratios were based on several different approaches presented in literature and our own previous research. Test series were produced according to the mixing sequence in Table 4. Based on this, 12 different mixtures including only silica fume as mineral admixture and 4% of steel fibers were prepared and cured under ambient conditions [21]. Mixture which has the highest compressive strength was selected and used in following experiments. The concrete mixture proportions applied in this study are presented in Table 2.



Fig. 1 - Mini slump cone / Miniconul pentru măsurarea tasării [11]

After curing 24 hours under ambient conditions, samples were removed from the mold and subjected to different curing regimes shown in Table 3. Curing regimes were adopted based on the research presented in the literature and our own research.

Mixture proportions of UHPSCC / Compoziția betonului autocompactant cu pulberi reactive

Material / Material	USF	U20MK	U20GFA	U40MK
Cement / Ciment (kg/m ³)	950	950	950	950
Silica fume / <i>Silice ultrafină</i> (kg/m³)	270	216	216	160
Metakaolin / <i>Metacaolin</i> (kg/m ³)	-	55	-	110
Fine blended fly ash / <i>Cenuşă</i> (kg/m³)	-	-	55	-
Quartz powder / <i>Praf de cuarț</i> (kg/m³)	350	350	350	350
0-0.5mm Quartz / <i>Cuar</i> ţ(kg/m³)	550	550	550	550
Water / Apă (kg/m³)	235	215	215	215
Superplasticizer / Superplastifiant (kg/m ³)	55	53	51	53
Steel fiber <i>/ Fibră de oțel</i> (kg/m ³)	310	310	310	310
Water from superplasticizer / Apă din superplastifiant (kg/m ³)	31.3	30.2	29.1	30.2
Flow slump / Scăderea fluxului (mm)	280	277	272	276

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Table 3

Cure programme and sample codes rogramul tratamentului termic şi codul probelor

Programui tratamentului termic și codul probelor			
Cure code	Cure type		
Codul probei	Tipul de tratament		
SC95/1d	24 h 95°C steam cure /		
	întărire cu abur		
SC05/2d	48 h 95°C steam cure /		
3095/20	întărire cu abur		
	48 h 95°C steam cure and		
SC95/7d	5 days heat cure / tratament		
0000/14	termic cu abur la presiune		
	atm și 5 zile autoclavă		
	4h 8 bar autoclave / 4 h		
AC8/4	autoclavă la presiune abur		
	de 8 bar/autoclavă		
	4h 20 bar autoclave /		
AC2 /4	4 h autoclavă la presiunea		
	de 20 bar autoclavă		

Table 4

Mixing sequence / Etapele procesului de amestecare						
Drv	mixina	1	Timp	de	amestecare	2 minutes

uscată	
With water added	4
Timp de amestecare cu apa	
With superplasticizer added / Timp de	5
amestecare cu superplastifiantul	
With fibers added	3
Timp de amestecare cu fibrele	

4. Results and discussion

4.1. Mechanical properties

Previous experimental were shown that compressive strength various between 130 to 160 MPa and flexture strength from 22 MPa to 30 MPa at ambient conditions on 4x4x16 cm specimens [21]. Samples for testing the mechanical properties were prepared in molds 4x4x16 cm without applying pressure on the fresh concrete. The results of the compressive strength and flexural strengths under 3 point flexure and uniaxial compression, in accordance with the EN196-1 standard are presented in Figure 3 and Figure 4, respectively. Samples were tested after heat treatment at different ages in accordance with Table 3. The analysis of these results shows that compressive strengths of various concrete with 20% replacement of silica fume with metakaolin or fly ash are equivalent to or slightly lower than those of various concrete containing only silica fume. The compressive strengths of U20MK and U20GFA mixtures were slightly higher than USF mixture after steam curing. The compressive strength of the autoclaved sample series was higher compared to the steam cured series with SC95/7d cure programme. Samples of USF mixture showed much larger difference between these two values compared to the samples of which contained ground fly ash or mixtures metakaolin.



Fig. 2 - Cross section of tested sample / Secțiune transversală a probei încercate.







Fig. 4 - The influence of different curing regimes on flexure strength / Influența condițiilor de întărire asupra rezistenței la încovoiere

UHPC without fibers is a glass like brittle material with a comparatively high modulus of elasticity of

50 to 70 GPa. The typical tension strength of the pure matrix is about 8 MPa. The results of flexural strength tests showed a clear influence of steel fiber content and different curing regimes. That means if the bending strength of fibered UHP is introduced into the design of structures it has to be considered that the bending strength primarily depends on the kind and the amount of fibers used, and not on the orientation and the distribution of the fibers. Flexure strengths of the various concrete with 20% replacement of silica fume with or fly ash generally metakaolin are higher compared to the concrete containing silica fume only. Relative compressive to flexture strength ratio of USF mixture was higher compared to the other mixture (Fig. 5.) because its flexture strength was lower compared to mixtures with metakaolin or fly ash. The samples of U20GFA mixture showed a smaller increase of relative compressive strength during steam curing (Fig. 6.)



Fig. 5 - Relative compressive vs. flexture strength with different curing regime / Rezistența la compresiune relativă în corelație cu rezistența la încovoiere relativă, în diferite condiții de întărire



Fig 6 - Relative compressive strength ratio of steam cured vs.SC95/7d steam cured sample / Rezistența la compresiune relativă în corelație cu rezistența la 7 zile, SC95/7d, a probelor întărite cu abur

4.2. Microstructure

Cement paste can also be considered as a random composite made of unreacted cement, CSH, CH, capillary pores and other chemical phases. The randomness in cement paste microstructure is measured in micrometers or bellow. The unique features of the UHPC microstructure can be summarized as the interfacial transition zone, which represents a small region next to the particles of guartz aggregate and each of the phases beeing itself a multiphase. Microstructural inhomogeneities can seriously affect the strength and other related mechanical properties because controlled these properties are bv the microstructural extremes, not by the average microstructure.

Optical and scanning electron microscopy (SEM) was used to examine the effect of steam curing and super-heated steam under preassure on the microstructure and mechanical properties of UHPC. The compressive strength of the composites with metakaolin or fly ash was of particular interest on the possibility of substitution of silica fume with these mineral additives. Samples were taken from the inside of the test sample to avoid the possible influence of thermal effects on the surface.



Fig. 7 - Optical microscope image of pore in steam cured sample – USF mixture / Imagine de microscopie optică, a porilor unei probe întărite cu abur



Fig. 8 - SEM image - Transition zone between quartz grain and C-S-H phase in super-heated steam under preassure sample at 8 bar – U40MK mixture / Imagine SEM a zonei de tranziție între granula de cuarț și faze C-S-H, a probei U40MK, tratate în autoclavă la 8 bari presiune abur.

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The examination revealed an increased porosity in the area of aggregate and steel fibers. The microstructure of cement matrix shown in Figures 7-12 confirm the hypothesis that the texture of steam cured and super-heated steam under preassure concrete showed more homogenous and dense cement paste when compared to the conventional concrete. The highest porosity was detected in non heat-treated speciments [21].



Fig. 9 - SEM image - Transition zone between steel fibre and C-S-H phase in super-heated steam under preassure sample at 8 bar –U20MK mixture / ImagineSEM a zonei de tranziţie între fibra de oţel şi fazele C-S-H, a probei U20MK, tratate în autoclavă la 8 bari presiune abur.



Fig. 11 - SEM image of pore in in CSH phase - steam cured sample - U20GFA Mixture / Imagine SEM a porilor probei U20GFA tratată cu abur la presiune normală

The thermal treatment of C-S-H phases results generally in the development of crystalline phases, but at steam curing regimes sometime were appeared fall of mechanical properties and failures in structure. In this experimental work there were no structure degradation and one reason for that should be high content of steel fibers. The dense microstructure of cement phases extended to aggregate boundaries and the transition zone between quartz grains and C-S-H phase was absent.

5. Conclusion

The test results of the samples with self compacting properties showed satisfactory mechanical properties considering the water/binder ratio that was in the range of 0.20 to 0.21. The substitution of silica fume by 20% of metakaolin or fine ground fly ash in the tested samples had no important effect on the compressive strength under different curing regimes.



Fig. 10 - SEM image- development of crystalline C-S-H phase in super-heated steam under preassure sample – U40MK mixture / Imagine SEM a evoluției fazelor C-S-H către structuri cristaline, aprobei U40MK tratate în autoclavă



Fig. 12 - SEM image - Transition zone between quartz grain and C-S-H phase in super-heated steam under preassure sample - U20M mixture / Imagine SEM a zonei de tranziție între granula de cuarț şi faze C-S-H, a probei U20M tratată în autoclavă.

This was especially evident in the samples cured by autoclave. The decrease of mechanical properties of the test samples with 40% supstitution of silica fume by metakaolin was evident. Samples exposed to AC20/4 curing regime were shown approximately 30% compressive strength increase compared to steam cured samples and more than 40% flexural strength increase between super-heated steam under preassure and heat cured samples on 1 bar.

Amorphous C-S-H phase is formed within the micro structure of the samples cured under ambient conditions, while steam curing and superheated steam under preassure develop more homogenous cement paste micro structure and formes a closed network of crystal fibers.

The main conclusion of this investigation is that the rate of clinker hydration governs the rate of pozzolanic reaction contribution to the strength in the case of high reactivity pozzolanic materials, like silica fume and metakaolin. Crystalline C-S-H phase fills microcracks and small defects in micro structure of UHPC. In addition. the cohesion between cement paste quartz and aggregate is increased in super-heated steam under preassure samples, which is especially evident in guartz filler particles where form a these particles and C-S-H phase transit zone with increased cohesion. The reduction of pore size is also a characteristic of super-heated the steam under preassure samples when compared to the samples cured under ambient conditions.

results Test show a very strong influence of different curing regimes in relation to the development of a more stable structure than amorphous C-S-H phases and to the mechanical properties of UHPC. Comparing the micro structure of the samples cured in water at 20°C and steam cured or super-heated steam under preassure samples, the micro structure is denser with the increase of temperature and applying vapor pressure. This phenomenon can explain increase of the compressive strength.

ACKNOWLEDGEMENTS

The work reported in this paper is a part of the investigation within the research project TR 36017 "Utilization of by-products and recycled waste materials in concrete composites in the scope of sustainable construction development in Serbia: investigation and environmental assessment of possible applications", supported by the Ministry for Science and Technology, Republic of Serbia. This support is gratefully acknowledged.

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