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The Effect of Heat Treatment on the Properties of Ultra High Strength Concrete

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Abstract. **The influence of heat treatment during curing process of ultra high strength concrete (UHSC) was researched. Four different heat treatment temperatures ranging from 50 to 200^{0}C were studied and compared to the reference temperature regime (20^{0}C). Two series of heat treatment were applied: (a) at the early age of UHSC (3 days) and (b) after 27 days of standard curing regime in water at 20^{0}C. Concrete compressive strength was tested at the early age (4 days) and at the age of 28 days. The water absorption and water penetration under pressure were tested for heat treated and untreated UHSC specimens. SEM and XRD investigations of the studied samples were performed. UHSC with the strength of 123 MPa at the age of 28 days was tested at the standard curing conditions. Results indicate that early age curing at elevated temperature increases early compressive strength from 123 to 189% while at the age of 28 days the compressive strength was only 95 to 117% from reference and depends on the heat treatment regime. The heat treatment of UHSC at the age of 27 days was beneficial with regard to the strength development. Heat-treated UHSC provided compressive strength gain from 112 to 124% from reference. The water absorption for all UHSC specimens was from 2.6 to 3.2 wt.% and it was not affected by the heat treatment. The calcite was detected with XRD in heat treated UHSC samples which indicates the carbonization of Portlandite. This could explain the strength gain of heat-treated samples and the reason for slow compressive strength increase in the case of early heat treatment application. SEM images reveal dense structure and unreacted silica fume particles. The early heat treatment initiated high early strength but the strength of concrete reduced at the age of 28 days comparing to the early strength; therefore late heat application was beneficial for strength gain of the UHSC.**

Keywords: **curing conditions, heat treatment, ultra high performance concrete.**

I INTRODUCTION

In the concrete industry ultra high strength concrete (UHSC) is a result of logical evolution process of traditional concrete and high strength concrete (HSC). UHSC is characterised with high binder content and reduced aggregate size and water to cement (or cement paste) ratio below 0.25 which allows obtaining material without capillary porosity. The compressive strength of UHSC exceeds 150 MPa [1]. To obtain UHSC effective micro and nano fillers such as pozzolans or supplementary cementitious materials have to be used. Incorporation of the above mentioned fillers which contain amorphous SiO_2 additives (i.e. micro and nano silica) in the mixture composition of concrete reduces the amount of Portlandite $Ca(OH)_2$ and $CaCO_3$ due to the pozzolanic reaction in hardened cement paste; therefore the amount of C-S-H increases which promotes strength gain of concrete [2]. Also SiO_2 and Al_2O_3 rich additives like zeolites can increase the amount of the C-S-H gel in concrete and reduce Portlandite by almost 50% at the concrete age of 3 and 28 days [3]. Pozzolanic reactions are slow thus for favourable reactions in UHSC heat treatment can be applied and the potential of supplementary cementitious materials containing high amount of amorphous SiO_2 can be used in the concrete cured at elevated temperatures. Fine glass (<25 μm) incorporated in concrete provides significant pozzolanic reactions in elevated temperatures even at early age [4]. The heat treatment of cement matrix leads to mineral composition change in the structure. Increased temperature during hardening intensifies the formation of CAH_{10} and C_2AH_8 at the temperature 60 and 80°C, while at 110°C there is dense C_3AH_6 structure observed [5].

Gallucci concluded that concrete cured at elevated temperatures up to 60 °C provides lower final strength caused by the C-S-H packing in nanoscale due to lose of bound water; therefore cement paste is coarser and more porous [6]. Derabla and Benmalek has published a research where self-compacting concrete with 50

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MPa strength has been heat treated at 60°C for 24 h. This approach turned out to be cost effective in early age of concrete while in long term the compressive strength loss was observed comparing to reference concrete [7].

Yan and Cui have reported that HSC cured at elevated temperatures restraints the compressive strength development, if only Portland cement is used in mixture composition, while using pozzolanic materials the compressive strength of concrete increases two times at early age and continues to grow in long term [8]. Other research has tested effect of elevated temperature to high volume fly ash concrete and it was concluded that exposure to elevated temperature up to 300°C increased the compressive strength of concrete while treatment at higher temperature decreased the strength of concrete [9], [10].

There are negative aspects which must be considered in order to apply heat treatment to the UHSC. In some cases significant damages of surfaces were observed for specimens cured at elevated temperatures. It is explained by excessive deformation of the exposed surface layer of concrete [11]. This can cause microcracking of the surface; therefore reduction of mechanical properties and durability of concrete can be observed.

Another problem of HSC exposure to elevated temperatures is related to explosive spalling, when sudden and destructive breaking of surface layer occurs during heating of concrete [12]. This problem has been associated with dense structure of the HSC and UHSC and this leads to low permeability of the material. The evaporation of free water from the structure of such concrete is limited, which builds up inner pressure, and when the tensile strength of concrete is reached, the spalling of concrete surface destroys the structure of the material. The pore pressure begins at 105°C, which corresponds to the water evaporation temperature, and the peak of pressure is reached at 220°C, when either explosive spalling occurs or pore pressure attenuates [13]. This is important finding for understanding the properties of UHCP. It is recommended not to exceed curing temperature above 200°C to avoid the potential pressure building up to critical and reduce the risk of explosive spalling.

In current research two series of heat treatment regimes in curing processes of UHCP were investigated and 200°C was selected as the highest heat treatment temperature.

II MATERIALS AND METHODS

Ultra high strength concrete (UHSC) mixture design was created using CEM I 42.5 N with Blaine fineness of 3787 cm^2/g. The maximal grain size of UHSC filler was 2.5 mm. Four fractions of fine sands were used: 0.3/2.5 mm, 0/1.0 mm, 0/0.3 mm and quartz powder to ensure compact structure of the UHCP. Elkem silica fume grade 971 (Fig. 1) and NanoSilica 999 (Fig. 2) were used as microfillers.

The mixture composition of UHSC is given in Table 1. The amount of cement was 800 kg/m^3, the W/C was 0.25, and water to cement paste (cement and silica fumes) ratio W/(C+P) was 0.22. To ensure workability of such low W/C UHCP mixture superplasticizer Sikament 56 was used (2.5% from weight of cement). Micro silica fume was added 12.5% from the mass of cement (100 kg/m^3) and nanosilica – 2.5% (20 kg/m^3) respectively.

The mixing procedure of UHCP was the following: all dry components except NanoSilica were mixed together for 90 s to obtain homogenous mixture of dry components.

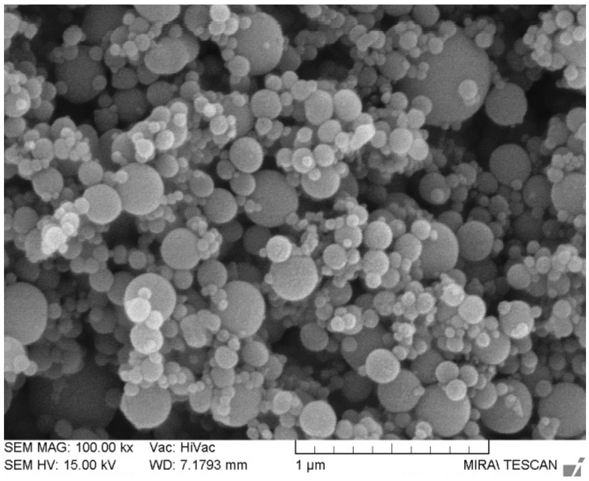

Fig. 1. Scanning electron microscope image of Elkem micro silica fume grade 971.

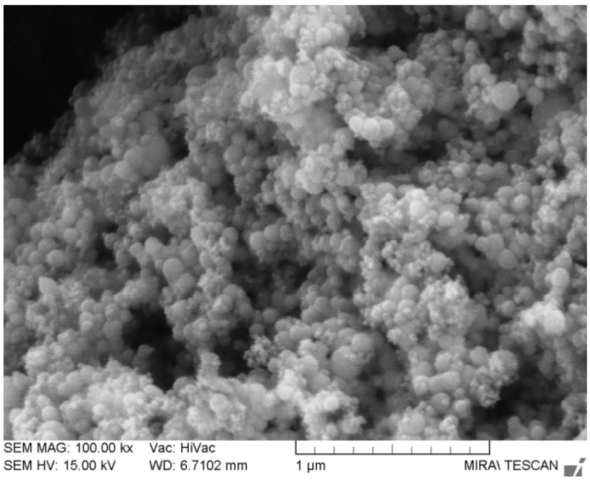

Fig. 2. Scanning electron microscope image of Elkem nano silica fume NanoSilica 999.

Then NanoSilica as water suspension (with ratio 1:2) and 75% of water were added to the mixture of dry components and mixed together for 120 s. In the last stage superplasticizer with the rest of water (25%) were added and mixed until homogenous and workable mixture was obtained. The total mixing time of UHCP was 6 minutes. 3 batches were prepared with 7 l of UHCP in each batch. The UHCP was cast in 18 cubical moulds with dimensions 5x5x5 cm, 4 cubical moulds with dimensions 10x10x10 cm and 9 prismatic moulds with dimension of 4x4x16 cm. After casting of UHCP in moulds samples were vibrated for 10 s.

Table I

Mixture composition of UHCP

Mixture composition	**kg/m^3**
Cement CEM I 42,5 N	800
Sand 0.3/2.5 mm	510
Sand 0/1.0 mm	480
Sand 0/0.3 mm	100
Quartz powder	100
Micro silica fume	100
Nano silica fume	20
Superplasticizer	20
Water	200
W/C	0.25
W/(C+P)	0.22

Samples were remoulded after 3 days of hardening and cured at standard conditions in water (±20^{o}C) or early heat treatment regime was applied. Four heat treatment conditions at temperature 50, 100, 150 and 200°C were tested for UHCP. The heat treatment was applied after remoulding the samples (on 3rd day) or at the age of 27 days after standard curing conditions (Table 2). The temperature increase during heating was 10 °C/min, samples were treated at the maximal temperature for 4 h and then cooled to room temperature.

Table II

Heat treatment regimes applied to UHCP

Sample ID	Concrete age, d	Heat treatment, °C
Ref	-	-
HT3-50	3	50
HT3-100	3	100
HT3-150	3	150
HT3-200	3	200
HT27-50	27	50
HT27-100	27	100
HT27-150	27	150
HT27-200	27	200

The compressive strength of concrete was tested according to LVS EN 12390-3. Compressive strength was determined for the specimens sized 50x50x50 mm. Depth of penetration of water was tested according to LVS EN 12390-8. Concrete specimens with dimensions 100x100x100 mm were tested under pressure of 500 KPa for 72+2 h. Water absorption was determined according to EN 1097-6. Scanning electron microscope (SEM) (Tescan Mira/LMU) was used for microstructural research and the mineralogical composition was determined with XRD (PAN analytical X'Pert PRO).

III RESULTS AND DISCUSSION

The density of obtained UHSC was 2.32 g/cm^3 and after heat treatment at temperature 200°C it decreased to 2.25 g/cm^3 which can be attributed to the evaporation of free water from the structure of UHSC. The SEM image of microstructure for reference sample (Ref) is given in Fig. 3 and for HT3-200 in Fig. 4. In both images dense microstructure of specimens was observed and the unreacted silica fume particles were detected. The significant difference of microstructure after heat treatment cannot be observed by SEM images; however, microstructure of the heat treated samples looks more porous compared to the reference sample. It could be explain by dehydration of C-S-H gel during heat application.

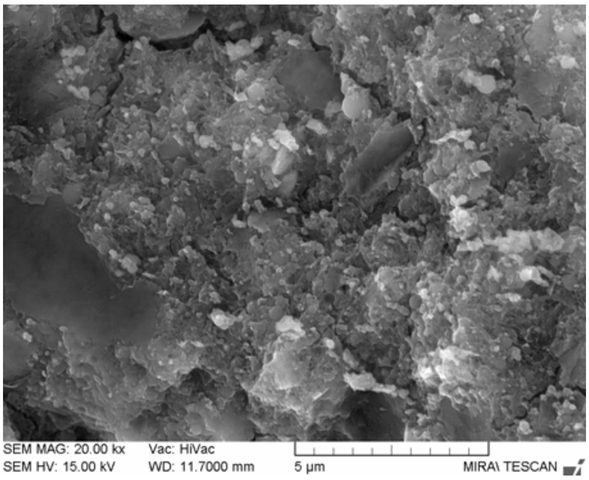

Fig. 3. The microstructure of UHSC cured at standard conditions at the age of 4 days (Ref).

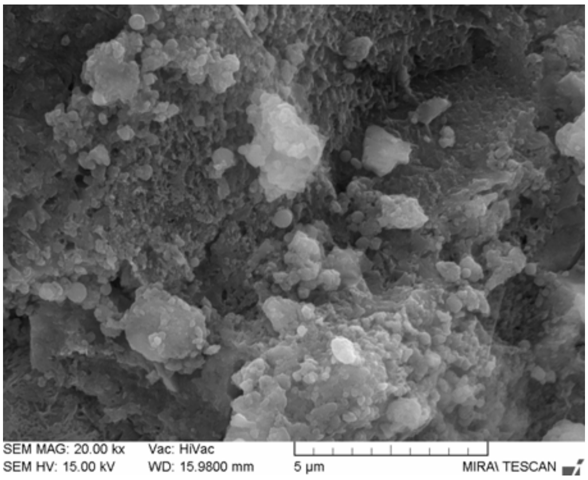

Fig. 4. The microstructure of UHSC cured at 200 °C for 4 h at the age of 4 days (HT3-200).

The results of compressive strength are given in Table 3. The early heat treatment of UHSC was beneficial for early compressive strength. The compressive strength for reference (Ref) UHSC was 77 MPa at the age of 4 days. The heat treatment even at 50°C for 4 hours increased the compressive strength up to 95 MPa but significant strength gain was observed for UHSC treated at temperature 100°C (130 MPa).

The heat treatment at early age in the temperature above 100°C – 143 MPa (150°C) and 145 MPa (200°C) was less effective. However, the early heat treatment did not provide further compressive strength increase during ageing of the concrete. At the age of 28 days compressive strength of reference increased to 123 MPa while for HT3-50 the decrease of compressive strength to only 117 MPa was observed–. HT3-100 provided almost the same compressive strength as Ref at the age of 28 days (124 MPa) while samples cured at 150 and 200°C (HT3-150 and HT3-200) provided compressive strength increase to 137 and 144 MPa respectively.

Table III

Compressive strength results of UHSC

Sample ID	Compressive strength, MPa		Strength index, %	
	4th day	28th day	4th day	28th day
Ref	77	123	100	100
HT3-50	95	117	119	95
HT3-100	130	124	141	101
HT3-150	143	137	146	110
HT3-200	145	144	147	115
HT27-50	-	138	-	111
HT27-100	-	144	-	115
HT27-150	-	150	-	118
HT27-200	-	153	-	120

The heat treatment for UHSC at the age of 27 days was beneficial to the compressive strength increase for all heat treatment regimes. The compressive strength of samples treated at temperature 200 °C in age of 27 days increased up to 153 MPa comparing to the reference samples (123 MPa). This indicates the importance of initial water curing at standard conditions of UHSC and understanding the necessity for heat treatment at proper age of the concrete. The strength index increased to 111 to 120% compared to the reference by applying heat treatment at the UHSC aged 27 days

The relative strength increase of UHSC related to the heat treatment temperature and the age of UHSC, when heat treatment was applied, is given in Fig 5. The initial compressive strength increase at the age of 4 days was significant (119%) even at the heat treatment at 50 °C, while the strength at 28 days decreased to 95% comparing to the reference. The compressive strength increase at the age of 28 days for samples, which were heat treated at the age of 27 days, was almost linear and strength increase was observed for every sample treated in elevated temperature.

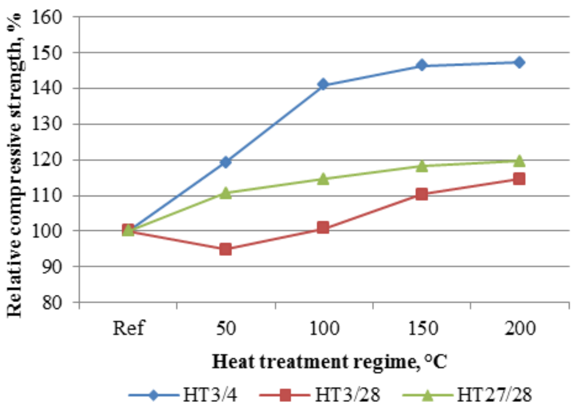

Fig. 5. Relative strength index of heat treated UHSC samples comparing to reference samples with standard curing regime.

The water absorption of UHSC was from 2.2 to 3.5%, and it was not affected by the heat treatment regime. The water penetration under pressure of 5 Bar was not detected for untreated UHSC at the age of 28 days (0 mm) while for heat treated samples HT3-200 the water penetration was 3 mm in average and up to 8 mm in some parts. This could be explained by the crack pattern of UHSC which appeared after heat treatment and thermal stresses, induced by the increased temperature and drying conditions, and was clearly seen after water penetration test (Fig 6).

Fig. 6. The microcrack pattern of split UHSC sample cured at 200 °C for 4 h (HT3-200).

The XRD patterns of reference and HT3-200 are given in Fig. 7 and Fig. 8. The results indicate that UHSC cured at standard conditions has quartz (Q), larnite (L) and plagioclase (P) minerals, while in heat treated sample calcite (C) has been detected, which could indicate the carbonization of free portlandite ($Ca(OH)_2$) and therefore possibly increases the early age strength of UHCP. The reduction of portlandite could restrict the pozzolanic reaction; therefore long term strength gain is limited for heat treated samples.

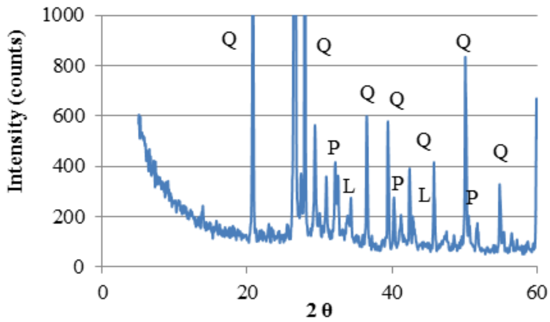

Fig. 7. XRD pattern of UHSC cured at standard conditions (Ref) at the age of 28 days.

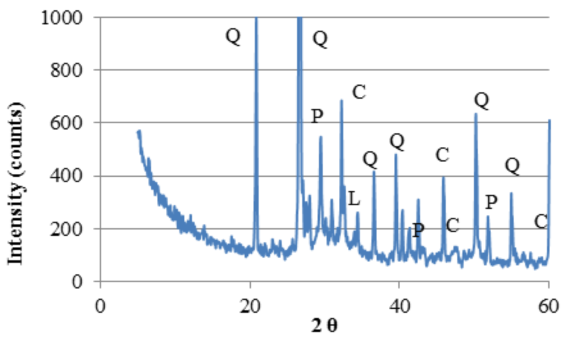

Fig. 8. XRD pattern of UHSC cured at 200 °C for 4 h (HT3-200) at the age of 28 days.

IV CONCLUSIONS

It was concluded that the early heat treatment at the temperatures ranging from 50 to 200°C is effective to gain early age strength of UHSC, while in the long term curing in temperature from 50 to 100°C was not effective and strength reduction was observed comparing to UHSC cured in standard conditions. Early heat treatment was effective in temperature range from 150 to 200°C, when compressive strength at 28 days increased from 110 to 115% comparing with reference concrete. The heat treatment of UHSC at the age of 27 days increased its compressive strength from 111 to 120%, and was beneficial with all heat treatment regimes. The heat treatment caused microcrack pattern of UHSC which reduced resistance to water penetration under pressure. In the mineralogical composition of UHSC after heat treatment calcite $CaCO_3$ was detected, which indicates the carbonization of free $Ca(OH)_2$ and is one of reasons for early age strength gain of UHSC.

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