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Influence of Silica Fume on Ultrahigh Performance Concrete

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Abstract**—**Silica fume, also known as microsilica (MS) or condensed silica fume is a by-product of the production of silicon metal or ferrosilicon alloys. Silica fume is one of the most effective pozzolanic additives which could be used for ultrahigh performance and other types of concrete. Despite the fact, however is not entirely clear, which amount of silica fume is most optimal for UHPC. Main objective of this experiment was to find optimal amount of silica fume for UHPC with and without thermal treatment, when different amount of quartz powder is substituted by silica fume. In this work were investigated four different composition of UHPC with different amount of silica fume. Silica fume were added 0, 10, 15 and 20% of cement (by weight) to UHPC mixture. Optimal amount of silica fume was determined by slump, viscosity, qualitative and quantitative XRD analysis and compression strength tests methods.

Keywords**—**Compressive strength, silica fume, ultrahigh performance concrete, XRD.

I. Introduction

ULTRAHIGH performance concrete (UHPC) is one of the most innovative construction materials in nowadays. It has superior mechanical and durability properties, which allows withstanding the neediest requirements.

In Lithuania UHPC is known as concrete which has compressive strength over 100 MPa [1]. By adding pozzolanic additives compressive strength can be easily increased up to 150-200 MPa. Such high compressive strength may be achieved by the following measures: elimination of coarse aggregate makes the mixture more homogeneous [2]; higher content of fine aggregates improves the granular composition of the mixture [3]; components of the mixture are selected with similar modulus of elasticity to achieve a more uniform compressive deformation of concrete [4]; properties of concrete matrix are improved by adding pozzolanic additives; W/C ratio reduced by adding high range water reduces (superplasticizers); thermal treatment of specimens also improves the concrete's tensile stress-strain behavior and the addition of steel or polypropylene fibers reduces brittle failure fracture of concrete.

Pozzolanic materials are very important in production of UHPC. The most widely and effective pozzolanic material is silica fume. Silica fume, also known as microsilica (MS) or condensed silica fume is a by-product of the production of silicon metal or ferrosilicon alloys [5], [6]. Although silica fume is relatively old additive, however the amount as supplementary materials in concrete mixture is not fully understood. Jianxin Ma developed Ultra High Performance Self Compacting Concrete and founded, that optimal amount of silica fume should be more than 25% (by weight of cement), to get the densest granular mixture [7]. Kennouche S. researched self-compacting concrete and founded that best workability results could be achieved when silica fume is added at 15% to cement content [8]. Badr El-Din Ezzat Hegazy made bricks for extremely aggressive environment and founded, that best physical and mechanical properties could be achieved when mixture consist 25% (by weight of cement) of silica fume [9]. A. A. Elsayed in his experiment with slag cement founded, that high amounts of silica fume should be avoided because mixtures slump intend to decrease, but resistance to water penetration intend to increase [10]. Carsten Geisenhaunsluke founded, that optimal amount of silica fume, conform to lowest viscosity of the mixture [11]. Later Michael Schmidt confirmed that theory, and added that maximum packing density correlates with minimum viscosity of concrete mixture [12]. Ivailo Terzijski founded, that when micro filler is substituted by the same amount silica fume (by volume), the slump almost remains constant [13]. Melanie Shink [14] and Jennifer C. Scheydt [15] also found similar results. M. K. Maroliya in his research on Reactive Powder concrete founded by qualitative XRD analysis method, that optimal amount of silica fume depends on applied curing regime [16]. Detlef Heinz noticed that even if thermal regime was applied, silica fume still reacts with portlandite creating additional C-S-H phases [17]. According to literature review it is not entirely clear, which amount of silica fume is optimal. It looks like optimal amount of silica fume depends on type of concrete, particle size distribution, required workability, physical and mechanical properties. However is not entirely clear by qualitative XRD analysis, how exactly portlandite remains unreacted. The objective of this work is to find optimal amount of silica fume for UHPC with and without thermal treatment, when different amount of quartz poeder is substituted by silica fume. For investigation slump, viscosity, qualitative and quantitative XRD analysis and compression strength tests methods were applied.

II. Materials Used for the Research

A. Cement

Portland cement CEM I 52.5 R was used in experiment. Main properties: paste of normal consistency– 29.3%; soundness (Le Chatielier) – 1.0mm; initial setting time –

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145min; compressive strength (after 2/28 days) – 38.6/65.3 MPa. Particle size distribution is shown in Fig. 1.

TABLE I
CHEMICAL COMPOSITION OF PORTLAND CEMENT AND SILICA FUME

Components	Quantity, %	
	CEM I 52,5 R	Silica fume
SiO_2	20.61	92.08
TiO_2	-	-
Al_2O_3	5.45	1.16
Fe_2O_3	3.36	1.24
MnO	-	-
MgO	3.84	0.80
CaO	63.42	1.07
SO_3	0.80	1.27
Na_2O	0.20	1.13
K_2O	1.00	0.67
P_2O_5	-	-
$Na_2O_{eq.}$	0.86	1.57
Loss of ignition	1.00	-

B. Silica Fume

Silica fume, also known as microsilica (MS) or condensed silica fume is a by-product of the production of silicon metal or ferrosilicon alloys. Main properties: density – 2120kg/m^3, bulk density (free-flow/compacted) – 255/329 kg/m^3, hygroscopicity 158%, natural fall angle 54º. Particle size distribution is shown in Fig. 2.

TABLE II
MIXING PROCEDURE OF UHPC

Time, sec.	Mixing procedure
60	Homogenization of silica fume, cement, quartz powder and quartz sand
30	Addition 100% of water and 50% superplasticizer
60	Homogenization
120	Pause
30	Addition of the remaining superplasticizer
60	Homogenization

C. Quartz Sand

In experiment quartz sand was used. Main properties: fraction: 0/2; density 2670kg/m^3, bulk density 1600kg/m^3, impurities ≤ 0.5%.

D. Quartz Powder

In experiment quartz powder was used. Main properties: density 2671kg/m^3; bulk density – 900kg/m^3; specific surface (according to Blaine) – 4423cm^2/g.

E. Chemical Admixture

In experiment was used superplasticizer based on polycarboxylic ether (PCE) polymers. Main properties: appearance: dark brown liquid, specific gravity (20°C) – 1.010÷1.070g/cm^3, alkali content – 2.5%, chloride content – 0.1%.

III. METHODS

A. Mixing, Sample Preparation and Curing

Fresh concrete mixes were prepared in modified laboratory mixer (mixing procedure given in Table II). Mixtures were prepared from dry aggregates. Cement and aggregates were dosed by weight, water and chemical admixtures were added by volume (Table III). Cylinders (d=50mm, h=50mm) were formed for the research to determine concrete properties. Homogeneous mixes were cast in moulds and kept for 24 hours at 20°C/95 RH (without compaction). After 24 hours some specimens were kept in water and for some specimens thermal treatment was applied (Table IV).

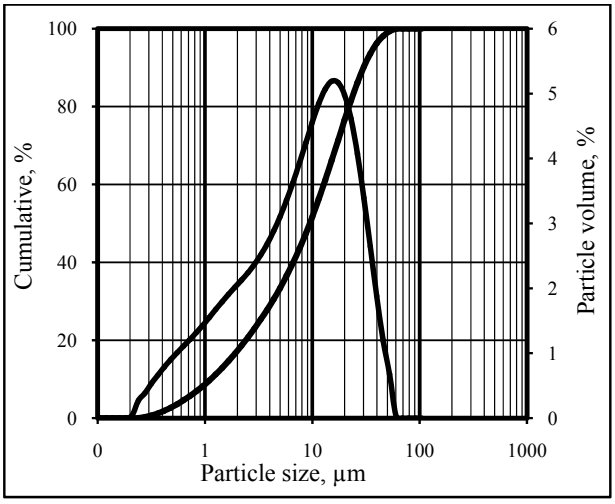

Fig. 1 Particle size distribution of cement

B. Slump and Viscosity

Slump was measured according to EN 1015-3 Standard (without compaction) [18]. Dynamic viscosity was measured by falling ball method (modified Stokes method) described in [19].

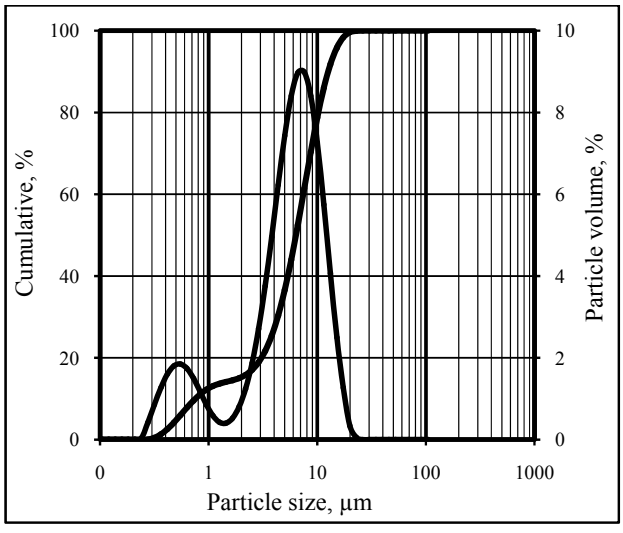

Fig. 2 Particle size distribution of silica fume

TABLE III
COMPOSITIONS OF ULTRAHIGH PERFORMANCE CONCRETE

Composition	Amount of silica fume, %	Water, l	Cement, kg/m^3	W/C	Micro filler, kg/m^3		Quartz sand, kg/m^3	Super plasticizer, l
					Silica fume	Quartz powder		
SF0	0	224	735	0.30	-	511	962	36.75
SF10	10				74	438		
SF15	15				110	401		
SF20	20				147	364		

C. Sample Preparation for XRD Analysis

Hardened cement pastes with 0%, 10%, 15% and 20% of silica fume were used for XRD analysis. The XRD measurements were performed with a XRD 3003 TT diffractometer of GE Sensing & Inspection Technologies GmbH with θ-θ configuration und CuKα radiation (λ=1.54 Å). The angular range was from 5 to 70 ° 2 Theta with a step width of 0.02 ° and a measuring time of 6 sec / step. For XRD quantitative phase analysis using the Rietveld refinement the samples were mixed with 20 wt. %ZnO (a standard material widely used in XRD analysis) as an internal standard and stored in argon atmosphere until measurement. This permits the estimation of the amount of non-crystalline phases by the Rietveld fitting procedure.

D. Compressive Strength

Compressive strength was performed after 28 days according to EN 12390-4 Standard [20]. Compressive strength was measured from 6 cylinders (d=50mm; h=50mm) as average value.

TABLE IV
ACTIVATION METHOD OF UHPC

Notation	Activation method
1D-T20	Without heat treatment, after demoulding specimens were stored for 27 days in 20°C water
7D-20T-1D-T80	After demoulding specimens were left for 7 days in 20°C water and then stored for 24 hours in 80°C water and rest of the time were left in 20°C water

IV. RESULTS AND DISCUSSIONS

According to the methods described before, there were created four composition of UHPC with different amount of silica fume (Table III). The objective of this work is to find optimal amount of silica fume for UHPC with and without thermal treatment, when different amount of quartz powder is substituted by silica fume. For investigation slump, viscosity, qualitative and quantitative XRD analysis and compression strength tests methods were applied.

Interesting fact was noticed, that when silica fume is added to UHPC composition from 0% to 20% (by weight) slump all the time remained almost constant and substitution degree of quartz powder to silica fume did not affect the slump of UHPC mixture (Fig. 3 and Table VI). However the viscosity of UHPC mixture was affected dramatically. Viscosity decreased about 2 times from 44 Pa·s to 20 Pa·s. Highest viscosity was obtained in composition without silica fume (composition SF0). The best results were obtained (composition SF15), when quartz powder is substituted by 15% of silica fumes (Fig. 3 and Table VI). The lowest viscosity probably relates with best particle size distribution and maximum packing density of UHPC. According to literature review this value should get best compressive strength results.

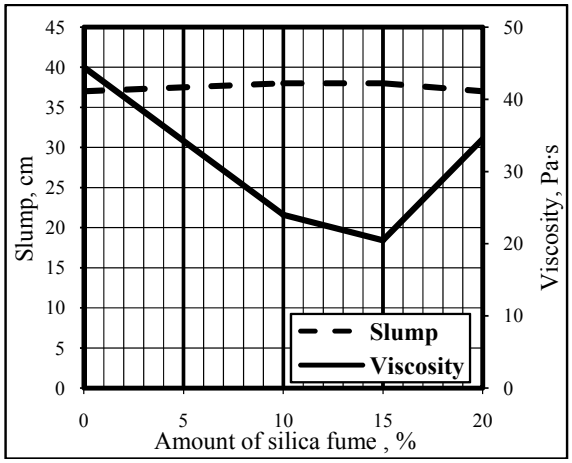

Fig. 3 Influence of silica fume on slump and viscosity of UHPC

As was expected density of UHPC with and without heat treatment, when different amount of quartz powder was substituted by silica fume, remained almost constant and it was about 2400 kg/m^3 (Fig. 4 and Table VI). The lowest density was obtained in composition SF0 without silica fume, and the highest density was obtained in composition SF15 when 15% of quartz powders were substituted by silica fume. These results correlate with highest and lowest viscosity.

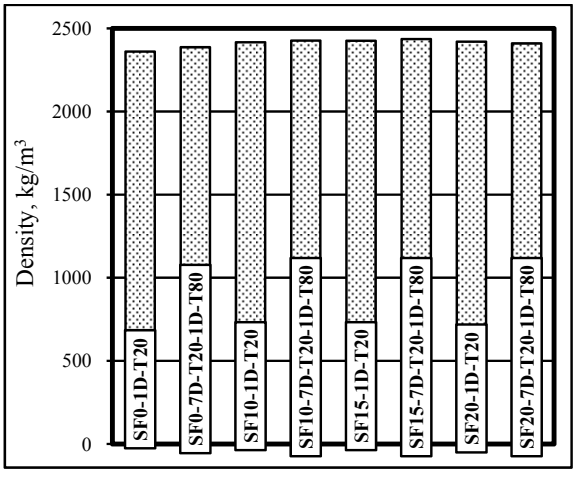

Fig. 4 Density of UHPC with and without heat treatment

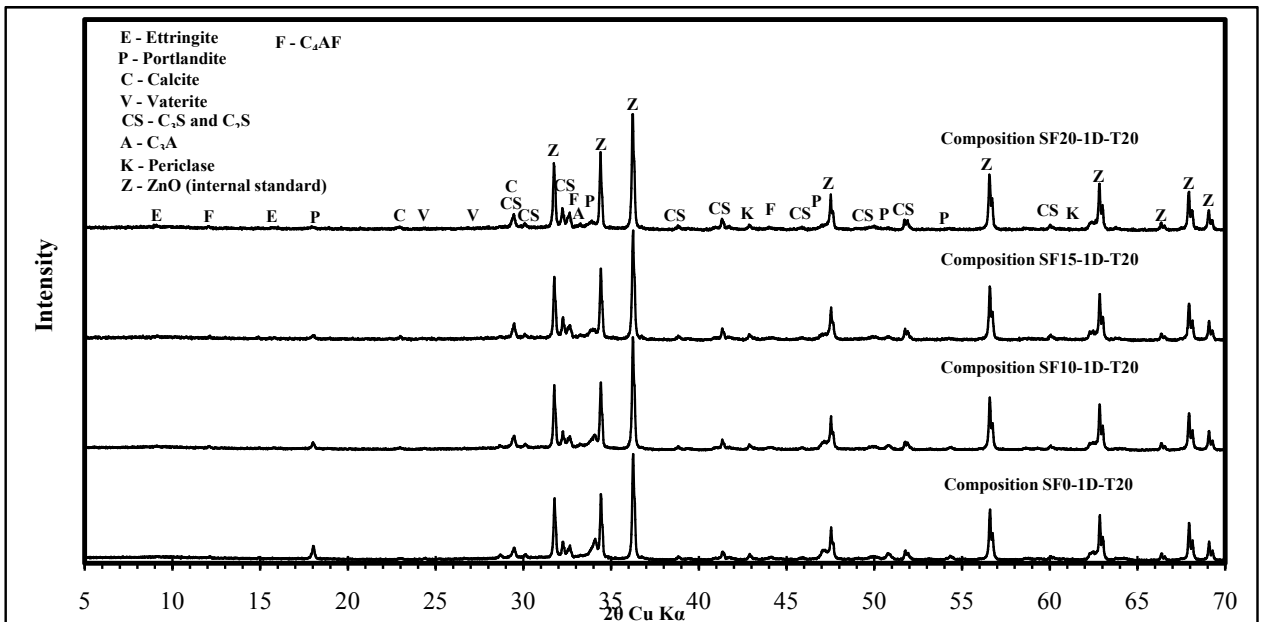

Fig. 5 XRD patterns of hardened cement pastes cured for 28 days at 20 °C temperatures

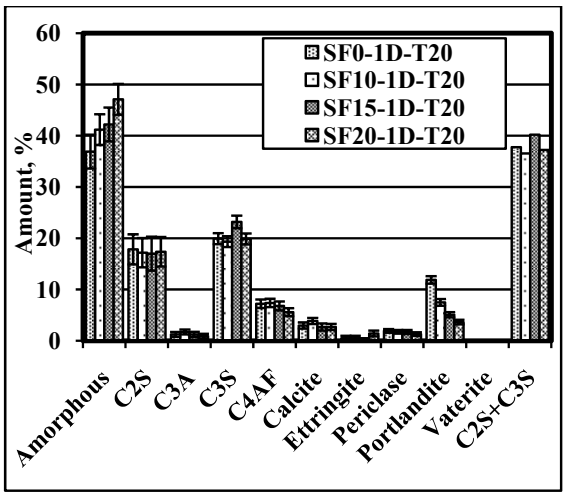

Fig. 6 Mineralogical composition of the binder for UHPC when heat treatment is not applied

Fig. 5 illustrates the XRD patterns of four hardened cement pastes with different amount of silica fume, when heat treatment was not applied. CH phase was found at 17.9°, 33.9°, 46.8°, 50.2° and 53.8°. Evidently, the crystalline phase of CH was decreased with increased amount of silica fume. C_2S and C_3S phases were found at 29.3°, 29.7°, 31.9°, 38.3°, 41.3°, 45.3°, 49.4°, 51.2° and 59.6°. It was noticed, what with increased amount of silica fume also decreased intensities of C_2S and C_3S phases. Decreased C_2S and C_2S peaks probably related with better solubility of clinker phase. Decreased CH peaks probably related with the consumption by pozzolanic reaction of silica fume.

Figs. 6 and 7 illustrate XRD quantitative analysis of four hardened cement pastes with different amount of silica fume. Experiment results revealed, that the clinker phases in the specimens (SF0, SF10, SF15, SF20) continued to react with water forming amorphous phases and portlandite.

Silica fume reacted with portlandite to form additional C-S-H phases. When heat treatment was not applied (Fig. 6 and Table V) and silica fume was increased up to 20% portlandite phase decreased (by weight) from 11.9% (composition SF0 without silica fume) to 3.7% (composition SF20 with 20% of silica fume) and amorphous phase increased from 36.9% (composition SF0 without silica fume) to 47.1% (composition SF4 with 20% of silica fume).

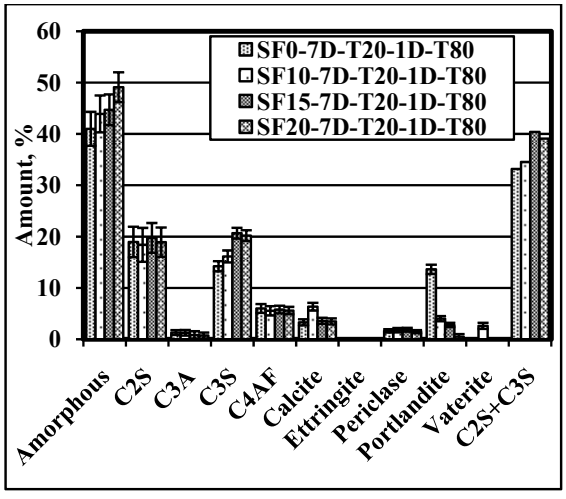

Fig. 7 Mineralogical composition of the binder for UHPC when heat treatment is applied

When heat treatment was applied (Fig. 7 and Table V) and silica fume was increased up to 20% portlandite phase

decreased (by weight) from 13.6% (composition SF0 without silica fume) to 0.5% (composition SF20 with 20% of silica fume) and amorphous phase increased from 40.9% (composition SF0 without silica fume) to 49.1% (composition SF4 with 20% of silica fume).

Interesting fact was noticed, that highest portlandite consumption by silica fume was in composition SF20, with 20% (by weight) of silica fume however the best compressive strength result was obtained in composition (SF15) with 15% (by weight) of silica fume. When heat treatment was not applied compressive strength increased about 34 % from 92 MPa (composition SF0 without silica fume) to 124 MPa (composition SF15 with 15% of silica fume).

TABLE V
MINERALOGICAL COMPOSITION OF THE BINDER FOR UHPC WITH AND WITHOUT HEAT TREATMENT

Compositions		Phases									
		Amorphous	C_2S	C_3A	C_3S	C_4AF	Calcite	Ettringite	Periclase	Portlandite	Vaterite
SF0-1D-T20	wt. %	36.9	17.8	1.3	19.9	7.2	3.0	0.5	2.0	11.9	0.0
	σ, %	3.3	2.9	0.5	1.1	0.8	0.6	0.5	0.4	0.7	0.0
SF0-7D-T20-1D-T80	wt. %	40.9	19.0	1.3	14.2	6.1	3.3	0.0	1.6	13.6	0.0
	σ, %	3.3	3.0	0.5	1.0	0.9	0.6	0.0	0.3	0.9	0.0
SF10-1D-T20	wt. %	41.2	17.2	1.7	19.4	7.4	3.9	0.5	1.8	7.5	0.0
	σ, %	3.0	2.9	0.5	1.1	0.8	0.6	0.5	0.4	0.7	0.0
SF10-7D-T20-1D-T80	wt. %	43.9	18.4	1.3	16.1	5.6	6.4	0.0	1.8	4.0	2.6
	σ, %	3.6	3.3	0.5	1.2	0.9	0.8	0.0	0.4	0.5	0.6
SF15-1D-T20	wt. %	42.2	17.0	1.3	23.2	6.8	2.7	0.0	1.7	5.1	0.0
	σ, %	3.3	3.3	0.5	1.2	0.9	0.7	0.5	0.4	0.5	0.0
SF15-7D-T20-1D-T80	wt. %	44.7	19.7	0.8	20.7	5.8	3.6	0.0	1.9	2.8	0.0
	σ, %	3.0	2.9	0.7	1.1	0.8	0.6	0.0	0.4	0.4	0.0
SF20-1D-T20	wt. %	47.1	17.4	0.9	19.9	5.6	2.7	1.4	1.3	3.7	0.0
	σ, %	3.0	2.9	0.5	1.1	0.8	0.6	0.6	0.4	0.5	0.0
SF20-7D-T20-1D-T80	wt. %	49.1	18.9	0.7	20.2	5.6	3.5	0.0	1.5	0.5	0.0
	σ, %	2.9	2.9	0.6	1.1	0.7	0.6	0.0	0.3	0.5	0.0

When heat treatment was applied compressive strength increased about 48 % from 93 MPa (composition SF0 without silica fume) to 138 MPa (composition SF15 with 15 % of silica fume). These results correlate with lowest viscosity and highest density of UHPC.

TABLE VI
PHISICAL AND MECHANICAL PROPERTIES OF UHPC

Parameter	Composition			
	SF0	SF10	SF15	SF20
Slump, cm	37.0	38.0	38.0	37.0
Viscosity, Pa·s	44	24	20	35
Density, kg/m3 (without heat treatment)	2361	2417	2426	2421
Density, kg/m3 (with heat treatment 7D-T20-1D-T80)	2387	2427	2436	2410
Compressive strength, MPa (without heat treatment)	92	113	124	107
Compressive strength, Mpa (with heat treatment 7D-T20-1D-T80)	93	124	138	128

In experiment was noticed what silica fume reacts with calcium hydroxide to produce calcium silicate hydrates (C-S-H), thus compressive strength of concrete increases. Since silica fume has very high surface area, high amounts ≥15% (by weight of cement) of silica fume is not desirable for good workability concrete, because water requirements for normal consistency mixture increases. Another interesting fact was noticed, that thermal treatment in analyzed composition does not significantly increased compressive strength, however amount of portlandite was reduced drastically.

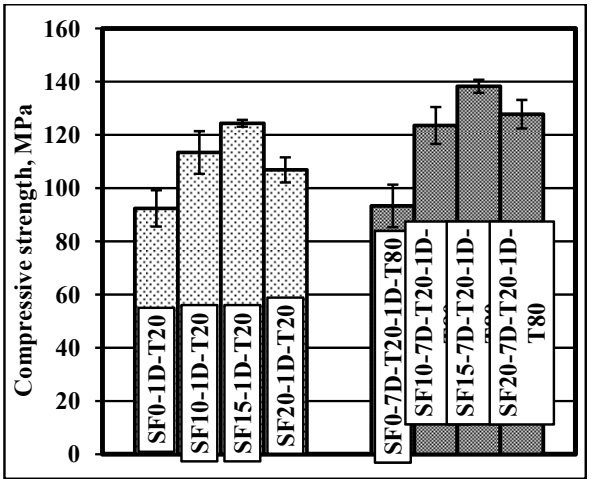

Fig. 8 Compressive strength of UHPC with and without heat treatment

Best viscosity and compressive strength results were achieved in composition SF15, when quartz powder was substituted by silica fume. In experiment were proofed, that high amount of silica fume does not necessary gives best workability and compressive strength results. Higher density mixture also could be achieved by properly choosing aggregates, cement, and thus amount of silica fume could be reduced. This is economically beneficial.

V. Conclusions

Extensive experiment was carried out to determine effect of silica fume on ultrahigh performance concrete for best compressive strength performance when water to cement ratio is 0.30. The following conclusions can be derived from the present investigation:

1) The results of the present investigation indicate that, when quartz powder was replaced from 0% to 20% by silica fume slump all the time remained almost constant and it was about 38cm. Lowest viscosity of ultrahigh performance concrete was achieved, when quartz powder was replaced by 15% of silica fume, and it was 20 Pa·s.
2) Regardless of the selected thermal treatment the lowest amount of portlandite was detected in hardened cement samples with 20% addition of silica fume. When heat treatment was not applied portlandite was detected 3.7% (by weight) and 0.5% (by weight) with heat treatment.
3) Best compressive strength results at 28 days fallows almost the same trend as lowest viscosity of ultrahigh performance concrete mixture. When heat treatment was not applied and quartz powder was replaced by 15% of silica fume compressive strength was obtained 124 MPa and 138 MPa with heat treatment.

Acknowledgment

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