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The Effect of Limestone Filler on the Properties of Self Compacting Concrete

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ABSTRACT

The effect of fly ash and limestone powder in concrete on the properties of self compacting fresh concrete mixture and hardened concrete was studied. It has been found that the self compacting concrete mixtures with limestone flour used as a filler show lower fluidity and higher air content as compared to those with fly ash.

The concretes with limestone filler reveal higher water permeability and lower freeze-thaw resistance as well as the resistance to the attack of de-icers as compared to the concretes with fly ash. The rheological properties of the mixture and hardened material are improved at higher fineness of this additive.

From the observations of microstructure of concrete samples one can conclude that the lower resistance of self compacting concrete with limestone filler is the consequence of the higher capillary porosity and the presence of large crystals of hydrated phases.

INTRODUCTION

The compositions of the mixtures for the self compacting concretes differ from the compositions of the other, traditional concretes. The main difference consists in the higher ratio of fine fraction, that is the fraction ≤ 0,125 mm[1]. The fine fractions together with the optimum water content and superplasticizer produce a stable suspension with higher viscosity; this suspension flows spontaneously keeping the aggregate grains without segregation[2,3]. The microfillers, such as fly ash, finely ground limestone, ground granulated blast furnace slag, metakaolinite and the other fine additives are the components of this fine fraction. The properties of concrete mixture and matured concrete are significantly affected by the type of microfiller in cement [4-6].

Fly is readily used in the self compacting concrete technology. As it has been reported by the other authors, the increase of the fly ash content in the mixture results in better workability at fairly good strength development and freeze – thaw resistance. In the presence of the limestone filler the stability of the mixture is affected and the segregation is not observed[6]; however this additive is not used on such a large scale in SCC technology as the fly ash. This may be the consequence of lower durability parameters for hardened materials, such as freeze – thaw resistance and the other properties dealing with durability[7].

In this work the effect of fly ash and limestone filler on the properties of self compacting fresh concrete mixture and hardened concrete was studied.

MATERIALS

The laboratory made portland cement was used; this cement was produced by co-grinding of industrial clinker with 5% gypsum (by mass of cement) to the Blaine specific surface of 320 m^2/kg. The concrete mixtures were produced basing on the blends containing 40% (by volume) of fly ash and limestone flour of different fineness. The phase composition of the clinker was as follows: C_3S – 65,3 %, C_2S – 15,3 %, C_3A - 9,5 %, C_4AF – 6,5 % (by mass).

The concrete mix design was carried out following the Japanese method proposed by Okamura and Ozawa[1].

The characteristics of fillers (chemical composition and specific surface according to Blaine method) is given in Table 1.The composition of concrete mixtures is given in Table 2.

Table 1. Chemical composition of microfillers and Blaine specific surface.

Component	Limestone powder		Fly ash
	L-1	L-2	FA
SiO_2	1,35	0,13	52,77
Fe_2O_3	0,50	0,20	7,32
Al_2O_3	0,46	0,20	24,98
CaO	51,60	52,38	3,42
MgO	2,24	1,68	2,73
Blaine specific surface [m^2/kg]	390	565	305

Table 2. Composition of concrete mixtures.

Component/ parameter	Unit	SCC-1	SCC-2	SCC-3
Factor ßp	–	0,83	1,02	1,01
Cement	kg/m^3	394	377	383
Water	kg/m^3	155	177	176
Fly ash	kg/m^3	263	-	-
Limestone flour	kg/m^3	-	252	258
w/c+d		0,24	0,28	0,27
Superplasticizer	% mas.	2,0	2,4	2,2

The grain size compositions of fillers, as measured using the laser granulometric microanalyzer, are plotted in Fig. 1.

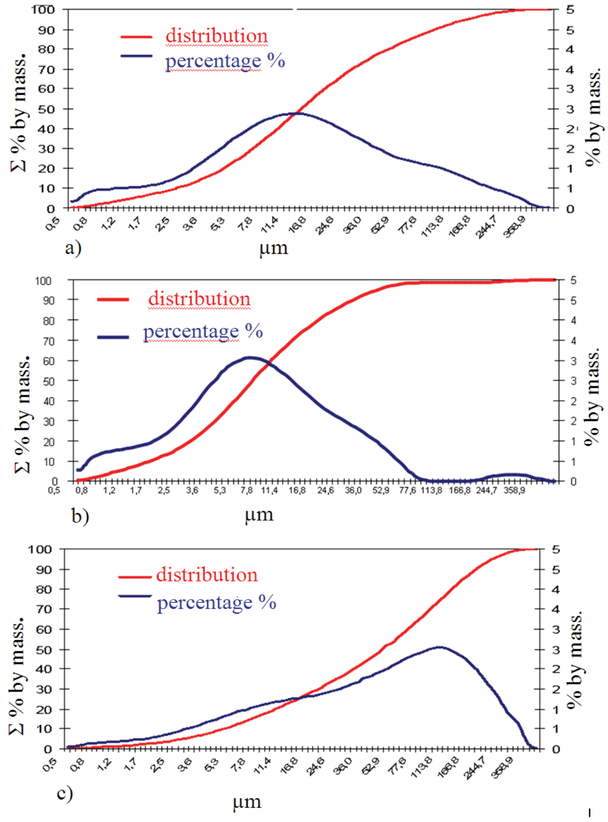

Figure 1. The grain size compositions of microfillers and cumulative grain size distribution curves: a) limestone powder L-1, b) limestone powder L-2, c) fly ash FA.

Based upon the grain size measurements one can notice that the limestone fillers differ significantly. The L-2 filler reveals significantly higher content of very fine particles (< 2,5 μm) than the additive L-1.

The following sample notations were used: SCC -1 mixture with fly ash; SCC-2 and SCC-3 mixtures with limestone flour of lower and higher fineness.

METHODS

The SCC mixtures were examined according to the FFB procedure (Flieβmaβ - Flieβzeit – Blocker – Test)[8].

In this test the fluidity of concrete mixture is evaluated from the flow diameter; the viscosity is characterized by the time needed to form a cake with 500 mm diameter, after the mixture outflow from the conical container (t_{500}). These parameters are controlled after 5, 30, 60 min. from the batch processing with water, with and without the J ring.

The air content in the concrete mixtures was determined according to the PN-EN 12350-7 standard: Testing fresh concrete. Part 7. Air content. Pressure methods.

The rheological parameters of the self compacting concretes were measured using the rotation rheometer ConTec Viscometer 4. The yield stress (τ_0) and plastic viscosity (μ) were determined from the rotation moment at different rotational speed of measuring device. The results were collected for increasing and decreasing rotational speeds in the "SCC Standard system", in the range from 0,4 to 0,08 rotations/s.

The permeability of concrete samples was determined as a depth of water penetration, according to the PN-EN 12390-8 standard: Testing hardened concrete. Part 8. Depth of penetration of water under pressure.

The freeze and thaw resistance was tested in the presence of de-icer (NaCl) according to the Swedish Standard 13 72 44 „Concrete testing – Hardened concrete – Frost resistance".

RESULTS

Properties of concrete mixtures

The results of the examinations of concrete mixtures and rheological tests are shown in Tables 3 and 4, respectively. On the basis on these data one can find that the self compacting concrete mixtures with limestone powder need more superplasticizer and water to attain the required parameters of SCC material; they show lower diameter of outflow cake, longer time of flowing than the SCC with fly ash and easily loose their fluidity with time.

Table 3. The properties of SCC mixtures.

Parameter		**Unit**	**SCC-1**	**SCC-2**	**SCC-3**
factor ßp		–	0,83	1,02	1,01
Diameter of outflow after:	5 min	mm	780	730	780
	30 min	mm	775	625	755
	60 min	mm	770	535	720
Time of flow t_{500} after:	5 min	S	4,5	4,5	3
	30 min	S	6	6,5	4
	60 min	S	7	12,0	5
Test of flow with J ring after 5 min.	Diameter of outflow	mm	770	710	770
	t_{500}	S	5	5	3
	h1)	mm	0	0	0
Air content		% obj.	0,9	1,6	1,4

Table. 4. The yield stress τ_0 [Pa] and plastic viscosity η_p [Pa·s] values for the self compacting mixtures.

Rheological parameters	**Type of mixture**		
	SCC-1	**SCC-2**	**SCC-3**
Yield stress [Pa]	30	-	-
Plastic viscosity [Pa s]	60,2	200,8	120,3

Rheological properties research of concrete mixtures shows (Table 4) that mixtures contained limestone powder SCC-2 and SCC-3 have higher plastic viscosity than SCC-1. However the plastic viscosity of the SCC-3 mixture with the finer limestone additive is lower.

The higher air content in the mixtures with limestone can be explained by the higher viscosity; this can be derived from the time of flow test (t_{500}) and the results of the measurements of rheological properties.

The properties of the self compacting concrete mixture are improved by the fineness of limestone. The mixture is then better flowable at lower percentage of superplasticizer and better de-aired.

The properties of hardened concrete

The results of compressive strength tests are given in Table 5, the permeability is shown in Table 6. As one can see, the SCC-1 concrete with the fly ash filler shows very low water permeability as compared to the values for the concretes produced with limestone; the higher fineness of limestone brings about the reduction of water permeability.

Table 5. Compressive strength of concretes.

Concrete	Compressive strength [MPa] at age				
	1 d	3 d	7 d	28 d	90 d
SCC-1	8,13	21,26	33,04	43,15	63,07
SCC-2	13,18	36,61	44,38	54,20	63,75
SCC-3	15,03	38,44	47,32	54,12	64,13

Table 6. Permeability of concretes.

Concrete	Depth of water penetration in concrete [mm]
SCC-1	33
SCC-2	105
SCC-3	80

The freeze – thaw resistance was evaluated basing on the mass of salt scales, on the samples cured following the procedure given in the Swedish Standard 13 72 44 „Concrete testing – Hardened concrete – Frost resistance”. The results are presented in Table 7.

Table 7. The freeze – thaw resistance of concretes.

Sample	Mean mass of scales (kg/m^2) vs. number of cycles:							
	7	14	21	28	35	42	49	56
	in NaCl							
SCC-1	0,001	0,001	0,001	0,004	0,005	0,006	0,008	0,009
SCC-2	0,67	2,64	3,00	-	-	-	-	-
SCC-3	0,30	1,03	2,60	-	-	-	-	-

According to the specification given in the standard cited above, only the SCC-1 sample with the fly ash microfiller exhibits good frost resistance, having the mass loss as low as 0,012 kg/m^2 after the 56 cycles of freezing and thawing. The samples with limestone filler loose their resistance to frost after 7 cycles.

Porosity of cement matrix

The results of the porosity measurements produced with help of the BET and mercury porosimetry are shown in in Fig. 2 and Table 8. The substantial differences between the porosities of cement matrix as a function of microfiller has been thus exhibited.

The cement matrix in concretes with limestone microfiller (SCC-2, SCC-3) reveals higher volume fraction of mesopores in the range 20-200 nm and lower content of that in the range 2-20 nm, as compared to the material produced with fly ash. The mean pore diameter in the limestone containing matrix is about three times higher than the value obtained for the material with fly ash. The higher water permeability and lower freeze – thaw resistance and resistance to the attack of de-icers in case of concrete with limestone filler can be thus explained.

It has been found also that the higher fineness of limestone filler brings about the reduction of mesopores from the range 20-200 μm. What is more – the concrete with the fly ash microfiller reveals over four times higher porosity in the range of macropores (200-20000 μm), as compared to the limestone containing materials.

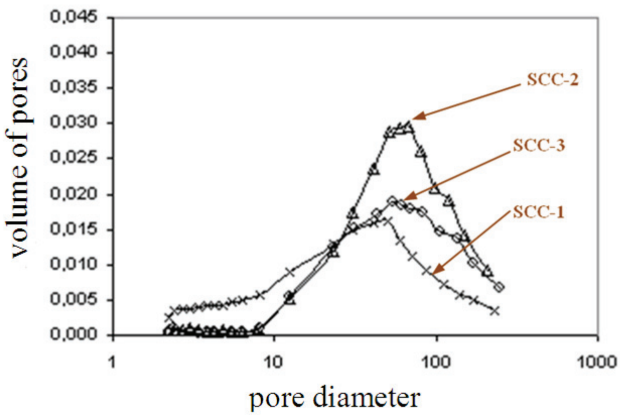

Figure 2. Distribution of mesopores.

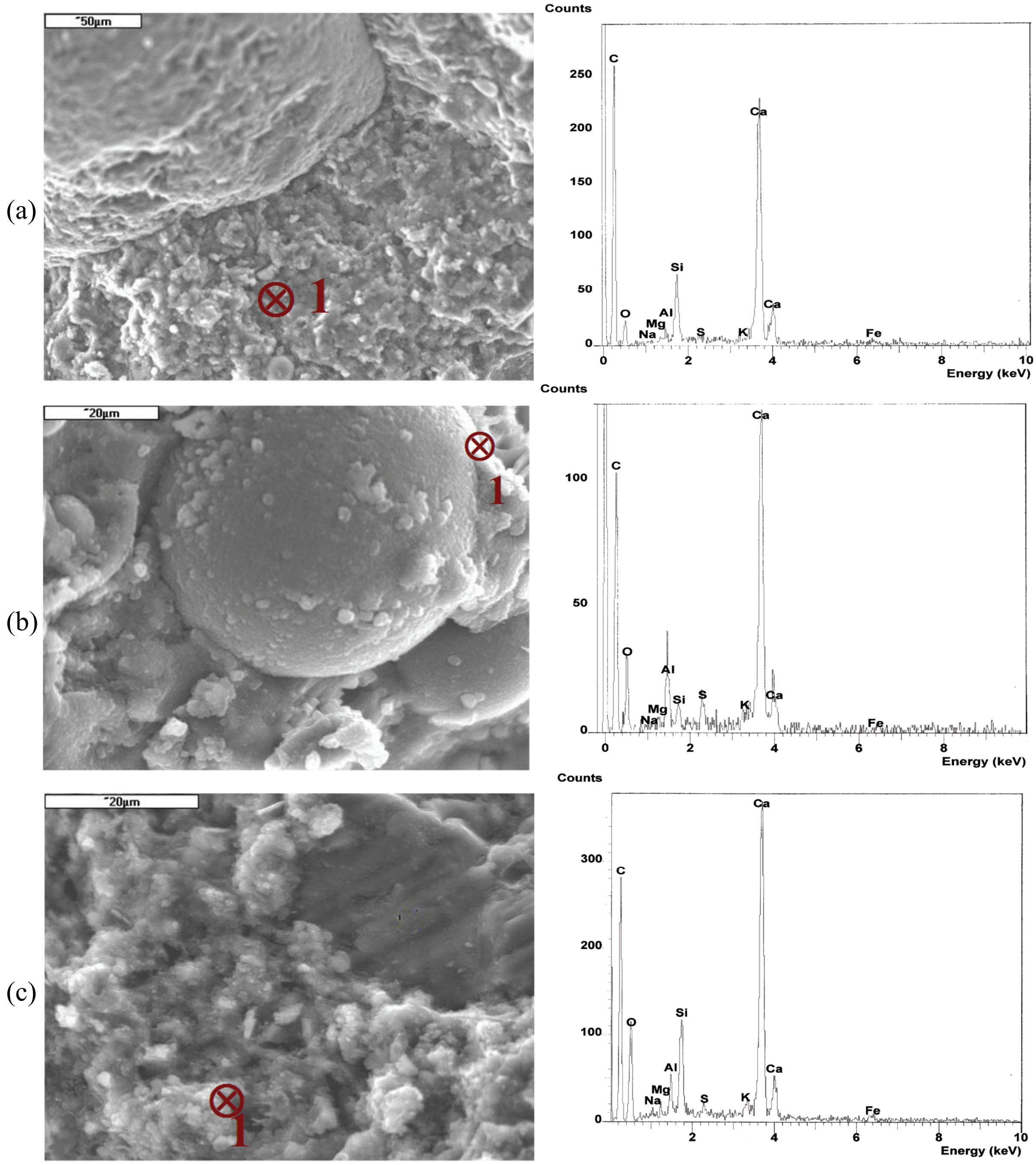

Figure 3. Microstructure and EDAX spectrum of the matrix in illustrated spot 1 for (a) SCC-1 cement matrix after 28 days maturing. See the C-S-H and the sand grain adherent well to the paste, (b) cement matrix in the fly ash containing SCC-1 after 28 days maturing. See the C-S-H and $Ca(OH)_2$ crystals , and (c) cement matrix in the limestone L-2 containing SCC-3. See the $CaCO_3$ residual crystals together with C-S-H.

Table 8. Results of porosity tests on SCC samples.

Parameter	SCC-1	SCC-2	SCC-3
Density[a] (g/cm^3)	2,5033 ±0,0014	2,5229 ±0,0015	2,5590 ±0,0015
Apparent density (g/cm^3)	2,2684 ±0,0165	2,3278 ±0,0165	2,3865 ±0,0110
Specific surface, S_{BET} (m^2/g)	4,94	2,54	2,13
Porosity (cm^3/g)			
- **Micropores** (< 2 nm)	0,049	0,032	0,028
- **Mesopores** (2-200 nm)	0	0	0
- volume (cm^3/g) including	0,018	0,025	0,018
2-20 nm	0,006	0,002	0,003
20-200 nm	0,012	0,023	0,015
Ø mesopores[b] (nm)	14	38	39
- **Macropores** (200-20000 nm)			
- volume (cm^3/g) including	0,031	0,007	0,010
200-2000 nm	0,019	0,004	-
2000-20000 nm	0,012	0,003	-
Total porosity (%)	10,0	7,7	6,7
Internal porosity (%)	2,8	2,0	-

[a] Determined by helium method.
[b] Mean diameter of mesopores.

As it has been found in the studies of microstructure by SEM method, in the 28 days maturing cement matrix in SCC-1 with fly ash microfiller there is the C-S-H as a main product together with the calcium hydroxide and ettringite (Fig. 3, see next page). One can observe a very good adhesion between the matrix and the fine aggregate.

The microstructure of cement matrix in the self compacting concretes with limestone microfiller is less compact than the microstructure in the material with fly ash. There numerous $Ca(OH)_2$ and unreacted $CaCO_3$ large crystals are observed (Figs. 3, 4, and 5).

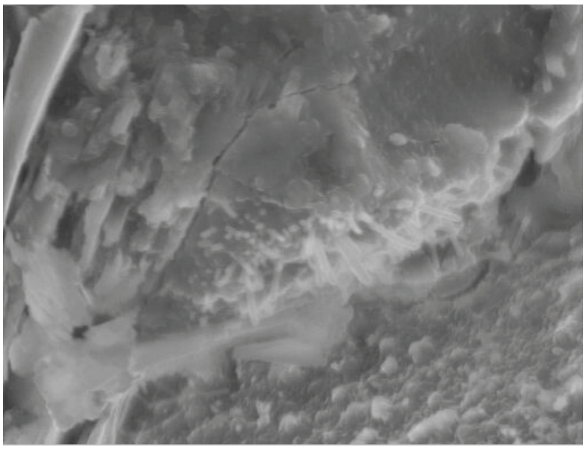

Figure 4. Microstructure of cement matrix in the limestone L-1 containing SCC-2. See the sand grain with $Ca(OH)_2$ and ettringite crystals.

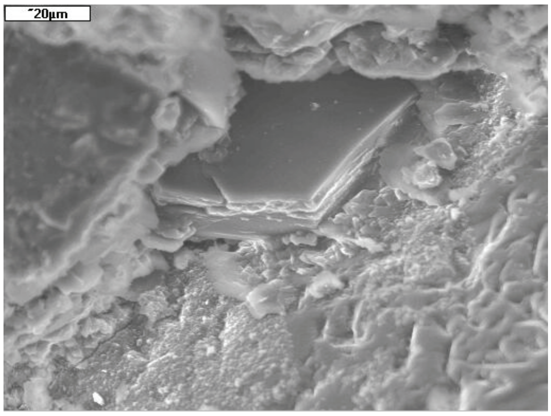

Figure 5. Microstructure of cement matrix in the limestone L-2containing SCC-1.See the sand grain with $Ca(OH)_2$ crystals.

As one can conclude from the studies of microstructure of cement matrix in self compacting concretes, the application of fly ash is the more convenient way, as the durability of concrete is concerned. The concrete with the fly ash filler is better compacted and shows lower porosity in the range of capillary pores.

CONCLUSIONS

The self compacting concrete mixtures with the limestone microfiller need more superplasticizer and water to achieve the parameters of self compacting mixture. They reveal also a lower diameter, longer time of flow and they lose earlier the fluidity. This means that the values of rheological parameters, that is the yield stress and plastic viscosity, are higher than for the mixtures with the fly ash microfiller. The fluidity of self compacting concrete mixture with limestone filler grows with the fineness of this additive.

The self compacting concretes with the limestone filler show higher water permeability and lower freeze – thaw resistance in the presence of de-icers than the concretes with the fly ash additive. These parameters can be improved by the higher fineness of limestone flour.

The shortage of freeze – thaw resistance and the resistance to the attack of de-icers in case of the limestone containing self compacting concretes is the consequence of the microstructure of cement matrix.

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