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Cement and Concrete Research 36 (2006) 879–885
Cement and Concrete Research

Influence of heat curing on the pore structure and compressive strength of self-compacting concrete (SCC)

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Received 20 May 2005; accepted 8 December 2005

Abstract

The mechanical properties of self-compacting concrete (SCC) are well understood. But there are no scientific investigations available on the influence of a heat treatment on the properties of SCC. To evaluate the influence on the compressive strength, SCCs as powder type, combination type and viscosity-agent type in the strength classes between C20/25 and C70/85 were designed and exposed to heat treatment with different maximum temperatures. It has been found that there is an influence of the composition of the concrete, especially the $(w/c)_{eq}$ ratio, on the compressive strength after heat treatment. The reason for the substantial loss of strength in some cases compared to the strength of the concrete, which was stored under standard conditions, is a change of the pore size distribution. An empirical formula is presented to calculate the influence of the heat treatment on the compressive strength of the SCCs.

Keywords: Self-compacting concrete (SCC); Heat treatment; Compressive strength; Pore size distribution

1. Introduction

The presented results are part of an extensive investigation in the influence of a heat treatment on the mechanical properties of SCC [1]. The motivation for this investigation was the fact that in the first version of the guideline for self-compacting concrete of the German Association of Structural Concrete (DAfStb) the heat treatment of SCC was not allowed. Based on results of the investigation in [1] the second edition of this guideline for self-compacting concrete of the DAfStb included the heat treatment of SCC [2].

2. Experimental

To investigate the influence of a heat treatment on the compressive strength of SCC different self-compacting concretes between the strength classes C20/25 and C70/85 were designed as combination type, powder type and viscosity-agent type. The composition of the investigated concretes is shown in Tables 1 and 2. In order to distinguish the different concretes the following nomenclature was introduced: The first letter stands for the type of SCC: K for combination type, M for powder type and S for viscosity-agent type. This letter is followed by the designed strength class (measured on cubes with an edge length of 150 mm). The next number denotes the maximum heat curing temperature of the concrete in Centigrade. All concretes were treated in such a way that they reached a maturity which corresponds to a storage of 3 days at 20 °C.

The $(w/c)_{eq}$-ratio used in Tables 1 and 2 is calculated as follows (see Eq. (1)):

$$(w/c)_{eq} = \frac{w}{c + 0.4f} \tag{1}$$

with: w = water content [kg/m^3], c = cement content [kg/m^3], 0.4 = equivalence factor according to DIN 1045-2, edition 2001-07, f = fly-ash content (accountable $f \leq 0.33c$) [kg/m^3].

The water content of the superplasticizer was taken into account. The superplastiziser is on the basis of polycarboxylate ester with a solid content of 35% (Woerment FM/BV 375). The viscosity agent used was Woermann underwater compound (ST). This viscosity agent is not water-soluble and shows a high

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0008-8846/$ - see front matter
doi:10.1016/j.cemconres.2005.12.004

Table 1
Composition of the combination type SCCs

Component		K25	K45	K65	K85
Cement type [a]		CEM II/ A-LL 32.5R	CEM II/ A-LL 32.5R	CEM II/ A-LL 42.5R	CEM II/ A-LL 42.5R
Cement content	[kg/m^3]	240	300	350	500
Water	[kg/m^3]	170	166	170	185
Limestone powder	[kg/m^3]	316	104	79	0
Flyash	[kg/m^3]	0	99	119	129
Sand 0/4 mm	[kg/m^3]	746	775	751	705
Gravel 4/16 mm	[kg/m^3]	878	900	873	819
Powder content [b]	[kg/m^3]	569	516	560	643
Superplasticizer content	[mass% of cement]	1.25	1.35	1.35	1.60
Viscosity agent content	[mass% of cement]	0.20	0.10	0.10	0.10
$(w/c)_{eq}$	[–]	0.71	0.49	0.43	0.34

[a] Powder is equal to the sum of cement, limestone powder, fly-ash, and particles ≤0.125 mm of aggregates.
[b] According to EN 197-1.

potential of swelling. It is based on natural and synthetic polymers. The lime stone powder Calcit MS14 came from Schön und Hippelein and has a $CaCO_3$ content higher than 99%. Fly-ash from one power plant was used.

The SCCs were treated with different maximum temperatures according to the guideline of the DAfStb on the heat treatment of conventional concrete [3]. All concretes should have a maturity which should be equal to a storage of 3 days at 20 °C. After the heat treatment the compressive strength of the SCCs was measured according to DIN EN 12390-3 on cubes with an edge length of 150 mm and compared to the compressive strength of the same concrete when stored for 3 days at 20 °C. The detailed description of the different heat curing regimes is given in Table 3.

The maturity of the concretes was calculated according to de Vree (CEMIJ-method) [4] with the real concrete temperatures.

Table 2
Composition of the powder type and viscosity agent type SCCs

Component		M25	M45	M65	M85	S25
Cement type		CEM II/A-LL 32.5R	CEM II/A-LL 32.5R	CEM II/A-LL 42.5R	CEM II/A-LL 42.5R	CEM II/A-LL 32.5R
Cement content	[kg/m^3]	240	300	350	500	240
Water	[kg/m^3]	168	166	170	183	192
Limestone powder	[kg/m^3]	338	134	66	0	145
Fly ash	[kg/m^3]	0	99	119	137	0
Sand 0/4 mm	[kg/m^3]	752	763	751	705	815
Gravel 4/16 mm	[kg/m^3]	856	887	873	819	928
Powder content	[kg/m^3]	594	545	548	650	402
Superplasticizer content	[mass% of cement]	1.25	1.25	1.35	1.45	1.50
Viscosity agent content	[mass% of cement]	0.00	0.00	0.00	0.00	0.45
$(w/c)_{eq}$	[–]	0.70	0.49	0.43	0.34	0.80

Table 3
Different heat curing regimes used

		Maximum curing temperature T_{max}		
		40 °C	60 °C	80 °C
Precuring temperature	[°C]	30	30	30
Duration of precuring at 30 °C	[h]	3	3	1
Rate of heating	[K/h]	10	10	10
Duration at T_{max}	[h]	14	5	3
Rate of cooling	[K/h]	10	10	10
Curing at 20 °C until testing	[h]	30.6	27.4	4.1
Total curing time	[days]	2.1	1.8	0.8

The temperature of the concretes was measured by means of a thermocouple which was embedded in a separate cube in the fresh state of the concrete. This cube was heat cured like the cubes for the compressive strength tests. The aspired maturity of all concretes equaled to 1822 °Ch. The compressive tests were performed when the concretes reached this maturity.

3. Results and discussion

3.1. Fresh concrete

In Table 4 the results of fresh concrete tests of SCC are summarized.

All tested concretes showed no blocking and no sedimentation of aggregates. The fact that sometimes the spread with J-ring is larger than without J-ring can be explained by the inherent scatter.

3.2. Hardened concrete

Fig. 1 shows the compressive strength of the combination type SCCs versus the maximum heat curing temperature of the different concretes. The concrete K25 shows a decreasing compressive strength with increasing temperature. The same can be seen for K45 until a heat curing temperature of 60 °C. A further increase of temperature leads to an increase in the compressive strength too. The high strength concretes K65 and K85 are not negatively influenced by temperature.

For an easier judgement of the influence of the curing temperature on the compressive strength the compressive strength at the different maximum curing temperatures is related to the value of each concrete at 20 °C. This is presented in Fig. 2 for the combination type SCCs. As can be seen in Fig. 2 the concretes K25 and K45 show an identical loss of relative strength up to 60 °C of about 22% compared to the treatment of the concretes with 20 °C. For the SCC K25 a further loss of strength can be seen with further rising temperature. The loss of strength at 80 °C related to the compressive strength at 20 °C is nearly 30% while the compressive strength of K45 at 80 °C is higher compared to the value at 60 °C.

The scatter of the compressive strength of high strength concretes K65 and K85 up to a temperature of 60 °C is within the range of dispersion which is known for compressive strength tests. At 80 °C both high strength concretes however show a compressive strength which is not within the normal

Table 4
Results of fresh concrete tests

		K25	K45	K65	K85	M25	M45	M65	M85	S25
V-funnel time	[s]	10.5	13.0	18.0	14.0	11.0	12.0	15.5	12.0	5.0
Spread	[mm]	750	720	690	780	780	740	745	770	720
Spread with J-ring	[mm]	750	725	690	765	785	730	750	730	685
t_{500}	[s]	5.0	6.0	10.0	8.0	6.0	7.0	6.0	8.0	4.0

scatter. The compressive strength is clearly higher compared to a permanent storage at 20 °C. As can be seen from Figs. 1 and 2 there is no negative influence of elevated temperatures on the high strength combination type SCCs.

The results of the compressive strength tests of the powder type and the viscosity-agent type concretes are shown in Fig. 3. It is interesting that there is an equal behaviour of the high strength SCCs and the normal strength SCCs of the powder type. The compressive strength of the viscosity-agent type S25 is steadily decreasing with rising heat curing temperature.

Fig. 4 demonstrates the change of the compressive strength of the powder type and the viscosity-agent type SCCs compared to the values of the permanent storage of these concretes at 20 °C. It can be seen, that there is a different behaviour compared to the combination type SCCs (see Fig. 2). All powder type SCCs are adversely affected by temperature up to 60 °C. A further increase of curing temperature up to 80 °C leads to an improvement of compressive strength. The high strength powder type SCCs even have a higher compressive strength at 80 °C than at 20 °C. This behaviour is equal to the combination type SCCs. The loss of strength of M65 and M85 up to 60 °C can not be explained by the scatter of the measurement. There must be a change in the structure of the concretes due to the heat treatment that causes the loss of strength. The behaviour of S25 is similar to K25. The compressive strength of this concrete declines permanently with increasing curing temperature and reaches a loss of strength compared to storage at 20 °C of about 30%.

Generally SCCs with a high $(w/c)_{eq}$-ratio are not appropriate for a heat treatment as they show a substantial loss of strength compared to the storage at 20°C. Concerning the high strength concretes with a low $(w/c)_{eq}$-ratio the combination type SCCs as well as the powder type SCCs are suitable for heat curing. But the tested combination type SCCs show the most favourable behaviour.

In Fig. 5 the total porosities of selected SCCs at different curing temperatures with a maturity equivalent to a storage of 3 days at 20 °C are presented. The total porosity was calculated according to the following Eq. (2):

$$p_{\mathrm{tot}} = \left(1 - \frac{\rho_{\mathrm{b}}}{\rho}\right) \times 100\% \quad [\mathrm{vol.\%}] \tag{2}$$

where: p_{tot}=total porosity [vol.%], ρ=specific gravity [g/cm^3], ρ_{b}=bulk density [g/cm^3].

The specific gravity of the concretes was calculated with the mass and the volume of a fine ground sample of each concrete. The volume of the ground powder was measured by means of a helium pycnometer.

Except for M25 all concretes had a higher total porosity at 20 °C than at the elevated temperatures. This means that there is no higher total porosity in the hardened concretes due to the elevated temperatures of the heat curing which could have been a possible explanation for the partially substantial loss of strength. The reason for the difference in compressive strength at the elevated temperatures compared to the storage at 20 °C lies in the pore size distribution of the concretes. For the investigation of the pore size distribution the SCCs with the highest and the lowest $(w/c)_{eq}$-ratio of each type was chosen because between those concretes were the biggest differences in the temperature behaviour (see Figs. 1–4). These concretes were investigated after the treatment with 20 °C, 60 °C and 80 °C. The pore size distribution was investigated with a mercury intrusion porosimeter (MIP) with a maximum pressure of 200 MPa. That means the smallest possible pore radius that was

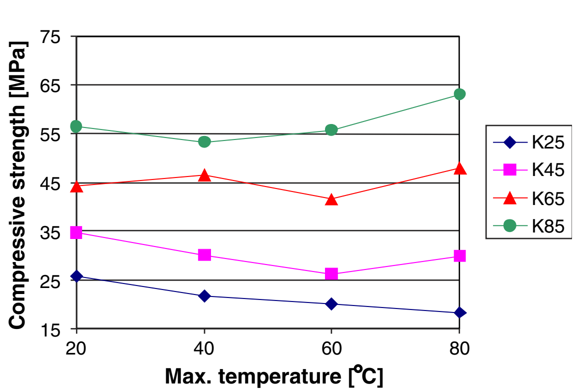


Fig. 1. Compressive strength vs. the maximum curing temperature for the combination type SCCs.

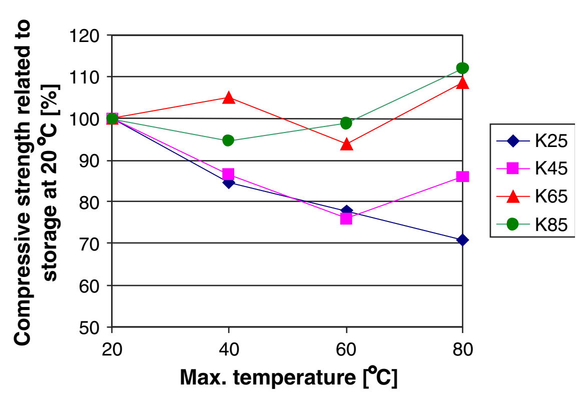


Fig. 2. Relative compressive strength related to storage at 20 °C vs. maximum curing temperature for the combination type SCCs.

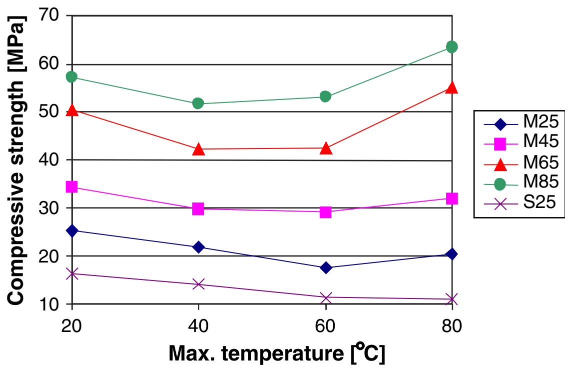


Fig. 3. Compressive strength vs. maximum curing temperature for the powder type and viscosity-agent type SCCs.

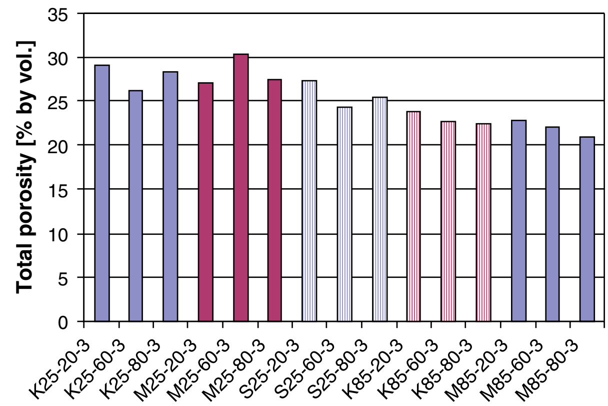


Fig. 5. Total porosity of different SCCs cured at 20 °C, 60 °C and 80 °C.

measured amounted approximately to 3.75 nm (assumption: all pores have cylindrical shape). For the MIP investigations prisms with the dimensions $40 \times 40 \times 160$ mm^3 were manufactured by using the same mix composition used for the compressive strength tests. Only the coarse aggregates larger than 4 mm were omitted. These prisms were heat-cured like the specimens for the compressive strength tests. After reaching the aspired maturity, the prisms were crushed and dried at 105 °C to stop hydration as fast as possible. The pore size distribution was measured on fragments selected from the center of the prisms.

The results of the mercury intrusion porosimetry measurements for K25 cured with different temperatures is shown in Fig. 6. It can be seen that with rising curing temperature the amount of bigger pores is rising too. The mean pore radius is getting bigger with rising temperature as well and is about 6 times higher after the treatment with 80 °C compared to storage at 20 °C. The major fraction of the pores after heat treatment are medium size capillary pores (meso-capillary pores). An increasing part of pores of this size influences the compressive strength. The total porosity of K25-80-3 and K25-60-3 however is lower than of K25-20-3 (see Fig. 5).

The behaviour of the SCC K85 is similar (see Fig. 7). There is a change in the pore structure between the specimen that were cured at 20 °C and the specimen that were treated with elevated temperatures. In opposition to K25 there is no difference in the pore size distribution of K85 after treatment with 60 °C and 80 °C. The change in the pore size distribution between 20 °C and the elevated temperatures is within the range of micro-capillary pores. Pores of this size don't have great influence on the compressive strength. This correlates very well with the results of the compressive tests of this concrete (see Figs. 1 and 2).

The influence of the elevated curing temperatures on the pore structure of SCC M25 (Fig. 8) is comparable to K25. There is a shift of the porosity to coarser pores, but the difference between 60 °C and 80 °C is very small. The mean pore radius of the heat-treated concretes is about three times higher compared to standard storage. These findings correlate very good with the compressive test results as well (see Figs. 3 and 4).

The high strength powder type SCC M85 (Fig. 9) behaves analogue to the high strength combination type K85. There is a difference between normal storage and curing with elevated temperatures but the range of this change is not influencing the compressive strength as the major part of the pores is still in the size of very small capillary pores.

The viscosity-agent type S25 (Fig. 10) shows the same behaviour depending on the heat curing temperature like the other SCCs of the strength class C20/25. The greatest difference is in the mean pore radius of S25. After standard treatment with 20 °C this concrete has the largest mean pore radius of all concretes tested. The cause for the large mean pore radius at all

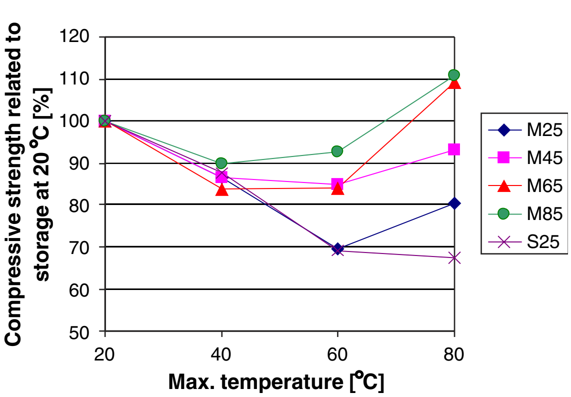


Fig. 4. Change of compressive strength related to storage at 20 °C vs. maximum curing temperature for the powder type and viscosity-agent type SCCs.

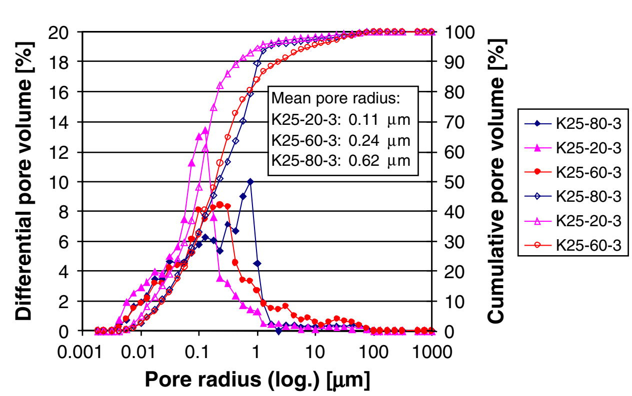


Fig. 6. Pore size distribution of K25 at different maximum curing temperatures.

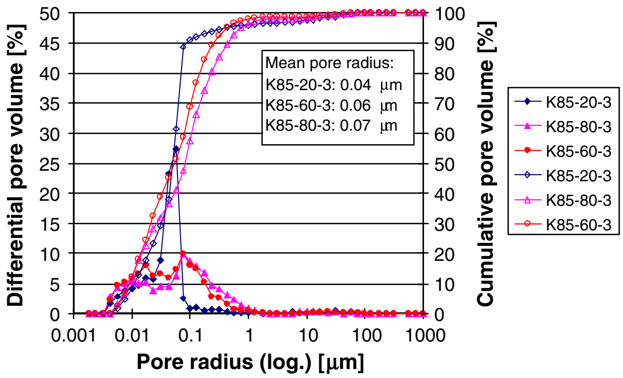


Fig. 7. Pore size distribution of K85 at different maximum curing temperatures.

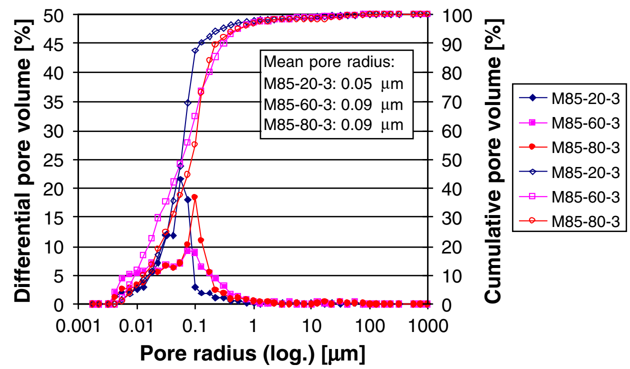


Fig. 9. Pore size distribution of M85 at different maximum curing temperatures.

curing temperatures is the high $(w/c)_{eq}$-ratio of this concrete (see Table 2).

It can be concluded from the results of the compressive strength tests, the total porosities, and the pore size distribution of the investigated concretes, that the reason for the difference of compressive strength of the SCCs after different curing temperature is the change of pore size distribution of the concretes. The changes in the pore size distribution can possibly be explained by the different $(w/c)_{eq}$-ratios of the SCCs. The admixing water of the concretes with a low $(w/c)_{eq}$-ratio will be chemically or physically bound after a very short period of time after mixing. Compared to these concretes, the SCCs with a high $(w/c)_{eq}$-ratio have still a relatively high amount o free water in the matrix when the heat curing of the concrete starts. Due to the increasing temperature, the free water will expand and as the expansion takes place at a time when the setting and hardening of the concretes is not fully finished, the expanded volume of the water is affixed in the matrix as larger pores when room temperature is reached again. The chemically or physically bound water of the concretes with a low $(w/c)_{eq}$-ratio can not expand in the same magnitude as the free water can. This could explain the different behaviour between the high strength and low strength SCCs.

The influence of the change in pore size distribution on the compressive strength shall be expressed by means of an empiric formula. The basis for this formula is the relation presented by Popovics [5,6] to calculate the influence of fresh concrete air pores on the compressive strength (Eq. (3)).

$$\beta_{\mathrm{crel}} = \frac{\beta_{\mathrm{c}}}{\beta_{\mathrm{c0}}} = 10^{-\gamma_{\mathrm{B}} a_{\mathrm{FB}}} \tag{3}$$

with: β_{crel}=relative compressive strength [–], β_{c}=compressive strength with air pores [MPa], β_{c0}=compressive strength without air pores [MPa], γ_{B}=parameter depending on the material, a_{FB}=fresh concrete air pore content [vol.%].

This formula shall be used on the clearly smaller capillary pores of concrete. As this equation does not separate between different pore sizes a modification of the formula is necessary to calculate the influence of the heat curing on the compressive strength (Eq. (4)).

$$\beta_{\mathrm{crel}} = \frac{\beta_{\mathrm{c}}}{\beta_{\mathrm{c0}}} = 10^{-((\xi p_1)+(\beta p_2)+(\gamma p_3))} \tag{4}$$

with: β_{crel}=relative compressive strength [–], β_{c}=compressive strength with air pores [MPa], β_{c0}=compressive strength without air pores [MPa], p_1, p_2, p_3=fraction of different capillary pore sizes of total porosity [vol.%], ξ, β, γ=weighting factors concerning the influence of the different pore sizes on the compressive strength [–].

To calculate the influence of the changed pore size distribution the measured pore volume of the concretes was divided into gel porosity with a radius r smaller than 5 nm

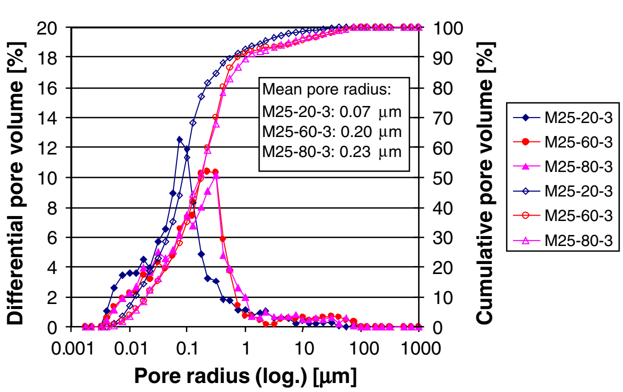


Fig. 8. Pore size distribution of M25 at different maximum curing temperatures.

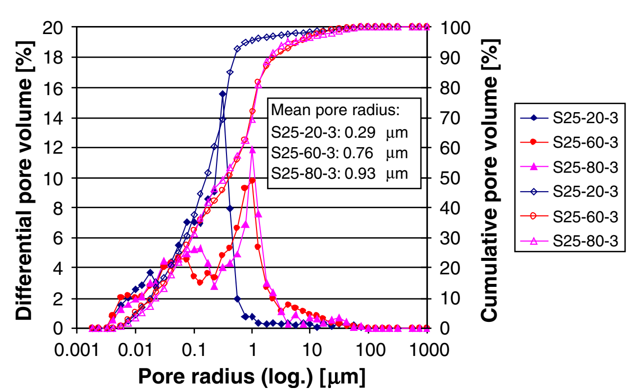


Fig. 10. Pore size distribution of S25 at different maximum curing temperatures.

(according to [7]), micro-capillary pores with $r<0.5$ µm, meso-capillary pores ($r>0.5$ µm) and macro-capillary pores ($r>15$ µm). The gel porosity has a negligible influence on the compressive strength and is therefore unaccounted in the calculation. The fraction of each type of capillary pores can be determined from the cumulative pore volume which is a result of the mercury intrusion porosimetry and the total porosity of each mixture. The basis for the calculation of the change in pore size distribution are the results of each concrete after standard treatment with 20 °C. Then the difference of each capillary pore size after the heat treatment compared to standard storage is determined. To calculate the influence of these changes on the compressive strength Eq. (4) has to be modified. Instead of the total fraction of each capillary pore size the change of each capillary pore size compared to the value of standard treatment is employed (Eq. (5)).

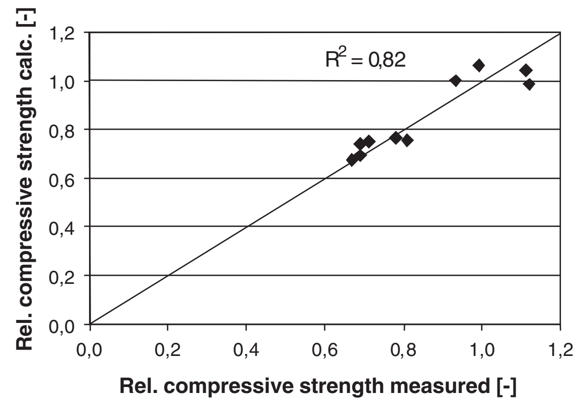
$$\beta_{\mathrm{crel}} = \frac{\beta_{\mathrm{cwb}}}{\beta_{\mathrm{cnorm}}} = 10^{-((\xi\Delta p_1)+(\beta\Delta p_2)+(\gamma\Delta p_3))} \tag{5}$$

with: β_{crel}=relative compressive strength [–], β_{cwb}=compressive strength after heat treatment [MPa], β_{cnorm}=compressive strength after standard treatment [MPa], Δp_1=difference in micro-capillary porosity after heat treatment compared to standard treatment [vol.%], Δp_2=difference in meso-capillary porosity after heat treatment compared to standard treatment [vol.%], Δp_3=difference in macro-capillary porosity after heat treatment compared to standard treatment [vol.%], ξ, β, γ=weighting factors regarding the influence of each type of capillary pores on the compressive strength [–].

The weighting factors were determined by regression analysis out of the measured compressive strengths of the concretes and the associated pore size distributions. The data basis for the regression analysis is compiled in Table 5.

Table 5
Data basis for the regression analysis to determine the weighting factors for Eq. (5)

Concrete	Relative compressive strength [–]	Difference in micro-capillary porosity [vol.%]	Difference in meso-capillary porosity [vol.%]	Difference in macro-capillary porosity [vol.%]
K25-20-3	1.00	–	–	–
K25-60-3	0.78	−4.45	2.94	0.47
K25-80-3	0.71	−5.09	5.60	−0.01
M25-20-3	1.00	–	–	–
M25-60-3	0.69	3.57	0.64	0.68
M25-80-3	0.81	0.24	1.68	0.56
S25-20-3	1.00	–	–	–
S25-60-3	0.69	−10.32	8.20	−0.06
S25-80-3	0.67	−9.05	8.22	−0.04
K85-20-3	1.00	–	–	–
K85-60-3	0.99	−2.75	−0.26	−0.02
K85-80-3	1.12	−3.12	0.85	−0.03
M85-20-3	1.00	–	–	–
M85-60-3	0.93	−5.05	0.21	0.21
M85-80-3	1.11	−2.86	−0.04	−0.02



Standard deviation [-]	0.067
Coefficient of variation [%]	6.7

Fig. 11. Comparison of measured and calculated relative compressive strength of the SCCs after heat treatment.

The corresponding weighting factors are:

ξ=0.00639	β=0.02828	γ=0.13187

So Eq. (5) can be written as follows (Eq. (6)):

$$\beta_{\mathrm{crel}} = \frac{\beta_{\mathrm{cwb}}}{\beta_{\mathrm{cnorm}}} = 10^{-((0.00639\Delta p_1)+(0.02828\Delta p_2)+(0.13187\Delta p-3))} \tag{6}$$

As can be seen from the numerical values of the weighting factors the influence of the pore sizes on the compressive strength is increasing with the pore radius.

In order to judge the significance of Eq. (6) the measured and the calculated relative compressive strengths of the investigated concretes are compared (see Fig. 11).

Despite the little data basis for the regression analysis there is a good correlation between the calculation with Eq. (6) and the measurement.

4. Summary

The influence of a heat treatment on the pore size distribution and the compressive strength of different SCCs was investigated. The heat curing of the SCCs leads to a change of the pore size distribution to coarser pores but not to a increasing total pore volume of the concrete. The change of the pore size distribution is correlated to the $(w/c)_{\mathrm{eq}}$-ratio of the concretes. With increasing $(w/c)_{\mathrm{eq}}$-ratio the mean pore radius of the concretes is stronger influenced. To calculate the influence of the changed pore structure an empiric formula is presented. The calculated results of this formula show a good correlation with the measurements.

Acknowledgements

The authors thank the Gips-Schüle foundation for the financial support of this investigation.

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