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Cement hydration in the presence of high filler contents

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Abstract

To realise self-compacting concrete, high filler contents are often used, and in order to avoid problems with excessive heat development during hardening, inert filler materials can be used. In this research two different filler types, limestone and quartzite, are considered in combination with different Portland cements. Although the filler material has often been considered to be inert, experimental results show that it does influence the hydration processes. On the one hand the reaction rate is influenced due to a modified nucleation possibility, and on the other hand, in some cases, the reaction mechanisms are altered, with a new hydration peak occurring. Based on isothermal conduction calorimetry on different cement-filler systems, an existing hydration model for blended cement is modified for these systems. Within the degree of hydration based hydration model, the cement/powder ratio seems to be an important parameter for the cement-filler systems. The model accurately predicts the heat of hydration during the hardening process.

Keywords: Self-compacting concrete; Filler; Hydration reaction; Modeling

1. Introduction

To realise a concrete that is self-compacting, two apparently incompatible properties have to be combined into one concrete: a high flowability and a high segregation resistance. This is made possible by the use of superplasticizers and viscosity enhancing agents combined with high concentrations of fine particles [1,2]. As a consequence, the high content of powder materials (cement and fillers) can lead to the development of a high heat of hydration, with rather large thermal stresses in the hardening concrete and early age thermal cracking as a possible consequence.

In the literature, the incorporation of cement replacement materials is sometimes recommended [3], in order to limit the heat generation. Non-reactive materials like stone dust could further reduce the heat of hydration. However, very few data concerning the development of the heat of hydration in self-compacting concrete (SCC) are given.

An extended research program concerning SCC is ongoing, with one of the research topics dealing with the heat generation in SCC during hardening. Isothermal and adiabatic hydration tests are carried out in order to evaluate the heat generation of self-compacting concrete. The results of these tests are compared with results obtained for traditional concrete. The applicability to SCC of an existing degree of hydration based hydration model is investigated.

2. Experimental determination of the heat of hydration

2.1. Isothermal hydration tests

To evaluate the heat generation in isothermal conditions, conduction calorimetry is used. This test is carried out on small samples of cement paste, thus excluding any influences of aggregate particles. To keep the temperature of the sample constant, the heat developed by the hydration reactions of the cement paste is liberated to the environment through a heat flux meter. The electrical potential measured

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Table 1
Composition of the powder mixes used in the isothermal tests

	Mix 1	Mix 2	Mix 3	Mix 4
Cement (g)	7.5	4.5	4.5	2.5
Filler (g)	–	3	3	5
Water (g)	3.75	2.25	3.75	3.75
w/c	0.5	0.5	0.83	1.5
w/p[a]	0.5	0.3	0.5	0.5
c/p	1.0	0.6	0.6	0.33

[a] Powder p = cement c + filler f.

Table 2
Chemical properties of the cement

	CEM I 42.5 R %	CEM I 52.5 %	CEM I 52.5 HSR LA %	Limestone filler %	Quartzite filler %
CaO	61.53	63.95	64.23	–	0.02
SiO_2	19.59	20.29	20.80	0.80	99.5
Al_2O_3	4.99	4.52	3.55	0.17	0.20
Fe_2O_3	2.98	2.35	3.94	0.10	0.03
MgO	0.78	2.22	2.40	0.50	–
K_2O	0.87	0.94	0.50	–	0.04
Na_2O	0.36	0.20	0.17	–	–
SO_3	3.29	3.35	2.74	–	–
Cl^-	0.080	0.015	0.014	0.002	–
$CaCO_3$	–	–	–	98.00	–
C_3S	58.2	59.0	60.6	–	–
C_2S	12.7	12.6	16.6	–	–
C_3A	8.19	8.01	2.75	–	–
C_4AF	9.1	9.4	13.1	–	–
Blaine (m^2/kg)	281	286	418	526	360

at the clamping-screws of the heat flux meter is directly proportional to the heat production rate q (expressed in Joule per gram of cement per hour, J/gh). An extensive description of the conduction method can be found in several national codes, e.g. the Belgian Standard NBN B12-213.

In order to be able to make a thorough study of the heat generation, isothermal hydration tests are carried out on pure cement as well as on mixtures of cement and filler. Two different fillers (a limestone filler originating from Marquise, France and a quartzite filler originating from Mol, Belgium) are combined with different Portland cements (CEM I 42.5 R, CEM I 52.5 and CEM I 52.5 HSR LA). In this first stage of the research, these tests are carried out using no chemical admixtures such as superplasticizers and viscosity enhancing agents.

Table 1 gives the powder mixtures as used in the tests. Four different compositions are considered, each of which is repeated for the different cement-filler combinations, and at three different temperature levels (10 °C, 20 °C and 35 °C). This table also shows some of the characteristics of the mixtures; such as w/c (water/cement ratio), w/p (water/powder ratio) and c/p (cement/powder ratio). In this, the powder content is the sum of both cement and filler materials.

Fig. 1 shows the pore size distribution of the different cements and fillers, and Table 2 summarizes the chemical properties of these materials. The mineral compositions of the three cements, as calculated by means of Bogue's formulas [4,5], are also given.

2.2. Adiabatic hydration tests

The heat production rate q (expressed in Joule per gram of cement per hour, J/gh) and the total heat production Q (expressed in Joule per gram of cement, J/g) can be calculated by measuring the temperature rise of a perfectly insulated concrete as a function of time during an adiabatic hydration test. A suitable test set-up was developed by De Schutter and Taerwe [6]: around a cylindrical concrete specimen (∅ 280 mm, height 400 mm) a water ring is created. By means of a differential thermostat, connected with a heating element, the water ring is kept at the same temperature as the concrete. In this way the heat loss is kept

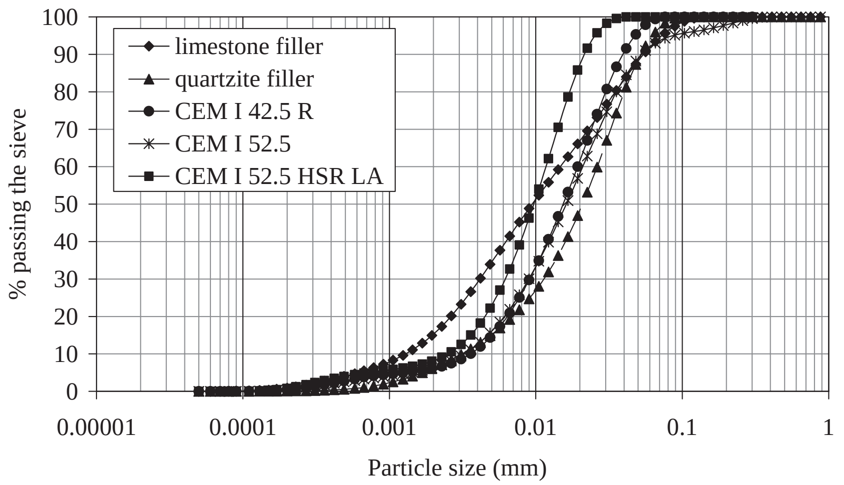

Fig. 1. Particle size distribution cement and fillers.

Table 3
Composition and properties of the mixes used in the adiabatic tests

		SCC 1	SCC 2	TC 1	TC 2
Coarse aggregate 4/14	kg/m^3	698	698	1280	1280
Sand 0/4	kg/m^3	853	853	670	670
CEM I 42.5 R	kg/m^3	360		300	
CEM I 52.5	kg/m^3		360		300
Limestone filler	kg/m^3	240	240		
Water	kg/m^3	165	165	150	150
Superplasticizer	l/m^3	2.3	2.2		
Slump flow	mm	795	810		
V-funnel	s	7.94	9.44		
U-test	mm	SL*	SL*		
Air content	%	1.8	1.2		
Density	kg/m^3	2330	2360		
Compressive strength	N/mm^2	63.8	60.7	52.2	64.7
E-modulus	N/mm^2	35,320	38,540		

SL*: self-levelling.

at a minimum value, and the concrete is kept in adiabatic conditions.

In this experimental program, the heat of hydration is determined on two different compositions of self-compacting concrete (Table 3). The composition is the same for both mixes, having the cement type as a parameter. The cements considered are CEM I 42.5 R (SCC 1) and CEM I 52.5 (SCC 2), originating from the same batches as the cements used in the isothermal tests. The filler used is the same limestone powder as was used in the isothermal tests. Each mix is characterised by means of slump flow, V-funnel, U-test, air content and density, 28-day compressive strength and the Young's modulus (Table 3).

In an earlier study, adiabatic hydration tests were performed on a traditional concrete (TC) consisting of 300 kg cement, 150 kg water, 670 kg sand and 1280 kg gravel per m^3 [7]. These tests were carried out with CEM I 42.5 R and CEM I 52.5. The 28-days strength of the TC was about 52 N/mm^2 for TC-1 (based on CEM I 42.5 R) and about 65 N/mm^2 for TC-2 (based on CEM I 52.5). Young's modulus was within the range of 35 000 to 38 000 N/mm^2 at 28 days.

3. Results of the hydration tests

3.1. Isothermal hydration tests

Figs. 2–5 show the heat production rate q J/gh as a function of time t, as obtained experimentally in the isothermal hydration tests at 20 °C (after elimination of the first 'wetting peak' [6,8]). Similar curves are obtained for the tests at 10 °C and 35 °C. A more interesting parameter than time is the degree of hydration, α defined as the cement fraction that has reacted. Due to difficulties in experimentally determining α, this study has used the degree of reaction, r, defined as the fraction of the heat of hydration that has been released at any point during testing [6,8–10]:

$$r(t) = \frac{Q(t)}{Q_{\max}} = \frac{1}{Q_{\max}} \int_0^t q(t)\mathrm{d}t. \tag{1}$$

In order to study the effect of the filler on the heat development of the hydrating cement, some numerical results are summarized in Tables 4 and 5. Table 4 gives the maximum heat production rate $q_{\max}$ J/gh for all different mixes at the three testing temperatures. Table 5 gives the total heat of hydration $Q_{\max}$ at the end of the test.

3.2. Adiabatic hydration tests

The experimentally obtained hydration curves are given in Fig. 6. For both cement types, the adiabatic curve of the corresponding TC is also given as a comparison. From the adiabatic curves it can be concluded that the maximum temperature rise is systematically higher for a self-compacting concrete in comparison with traditional concrete. A more detailed comparison can be made when the heat generation is expressed per unit weight of cement. For that purpose, the measurement of the evolution of the

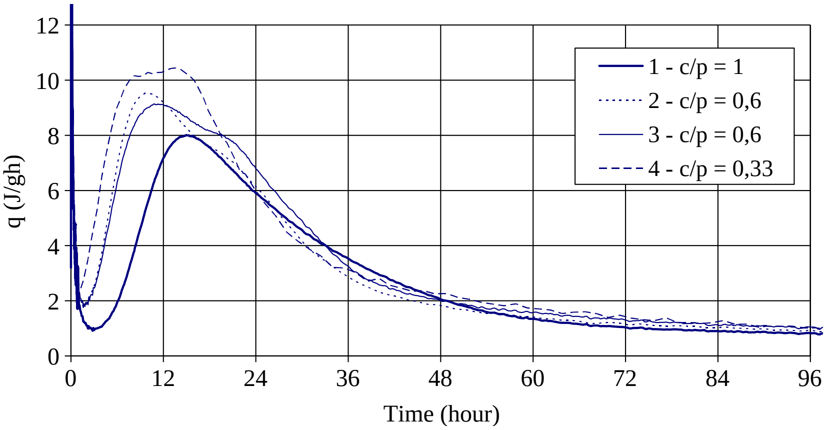

Fig. 2. Heat production rate for mixtures with CEM I 42.5 R and limestone filler at 20 °C.

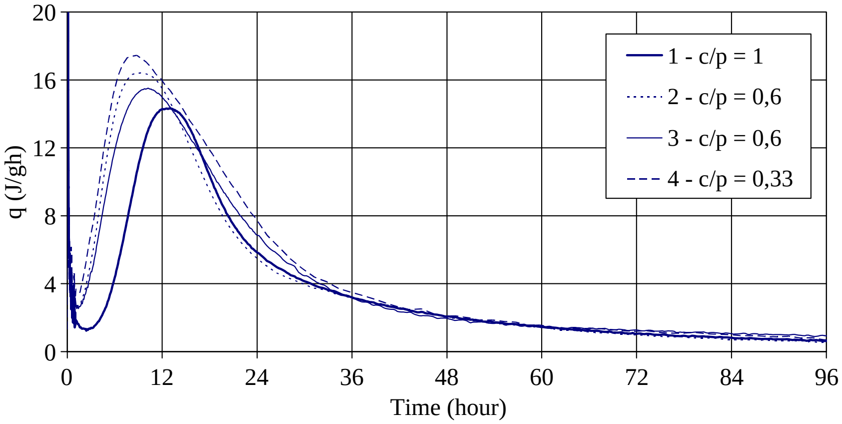

Fig. 3. Heat production rate for mixtures with CEM I 52.5 and limestone filler at 20 °C.

temperature of the concrete is translated to a heat production curve, according to:

$$Q(t) = c_c \frac{\rho}{C}(\theta(t) - \theta_0) \qquad (2)$$

with Q = cumulated heat of hydration (in J/g of cement), c_c = specific heat of concrete (in J/kg °C), θ_0 = start temperature (in °C), ρ = concrete density (in kg/m^3), C = cement content (in kg/m^3). For the compositions considered, the specific heat of the concrete has been approximated by 1000 J/kg °C [8].

Knowing the cumulated heat production $Q(t)$, the heat production rate $q(t)$ in adiabatic conditions can be calculated. In order to be able to compare the different compositions, $q(t)$ can be further transformed into $q_{20}(r)$, with q_{20} = the heat production rate at a temperature of 20 °C and r = degree of reaction defined as $Q/Q_{\max}$ (with $Q_{\max}$ the maximal cumulated heat of hydration at the end of the reaction). The influence of the temperature is modelled by means of an Arrhenius function [11]. More details about this transformation can be found in [8,12]. In this way the results shown in Figs. 7 and 8 are obtained, for the different cement types considered.

4. Discussion

4.1. Reaction mechanism

From the results given in Figs. 2–5, and from the corresponding results at different temperatures, it can be seen that the reaction mechanism of the hydrating cement is in some cases altered by the presence of the filler, for example the CEM I 42.5 R combined with limestone filler. When limestone filler is added to the cement, the induction period is shortened considerably and an extra hydration peak occurs after about 24 h for mixes 2 and 3, and after about 15 h for mix 4. In the last case, the second, extra peak has a heat production rate that is slightly higher than the first peak. At 35 °C this effect is even more pronounced. This can lead to the conclusion that the more limestone filler is added (decreasing c/p) and the higher the testing temperature, the higher the heat production rate of the extra peak.

The alteration of the hydration reaction can also be noticed for the tests with the CEM I 52.5, though it is much less pronounced. For the CEM I 52.5 HSR LA an alteration of the hydration reaction with the occurrence of a second

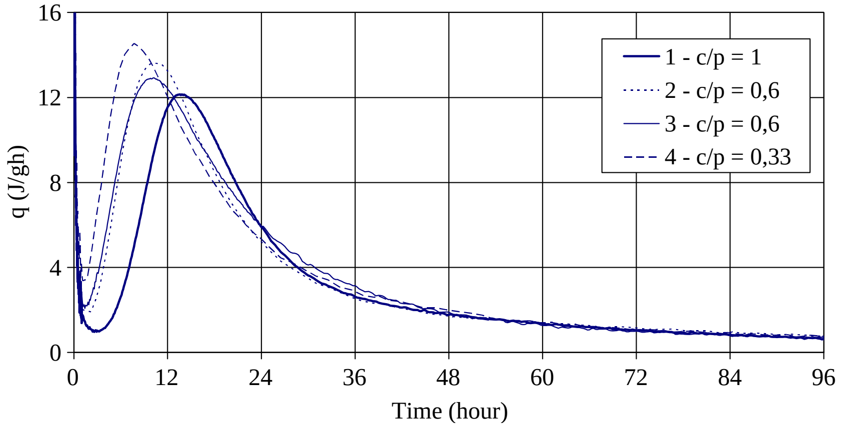

Fig. 4. Heat production rate for mixtures with CEM I 52.5 HSR LA and limestone filler at 20 °C.

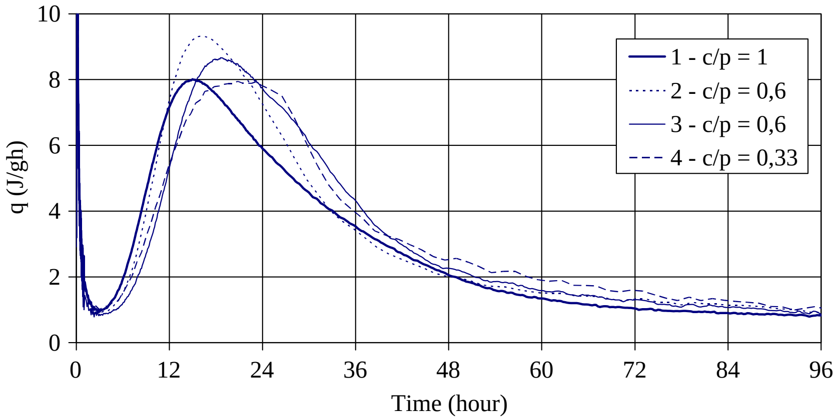

Fig. 5. Heat production rate for mixtures with CEM I 42.5 R and quartzite filler at 20 °C.

hydration peak is not found. Unlike the limestone filler, the quartzite filler does not seem to influence the induction period, and, apart from a slight undulation of the curve of the hydration rate in some cases, no extra peak is observed.

The curves in the Figs. 7 and 8, resulting from the adiabatic hydration tests, confirm the alteration in the hydration mechanism found during the isothermal hydration tests when limestone powder is added to the mix. For both cement types a sudden peak in the heat evolution is observed indicating the presence of a second reaction which is also found during the isothermal tests.

The cause of the change in hydration mechanism and the associated hydration peak is not clear, but a number of hypotheses can be drawn up.

In the literature, the influence of the filler material, and in particular limestone filler, on the hydration of the cement is in most cases considered to be limited to the rate of the reactions. Several authors mention that the setting kinetics are improved, the dormant period is reduced and the hydration process within the first hours is accelerated [13–16]. Kadri and Duval [17] proposed that the filler particles act as sites of heterogeneous nucleation to precipitate more or less crystallized hydrates, and in this way accelerate the hydration.

For the occurrence of the second hydration peak a few different hypotheses can be followed: The effect might be related to the hydration of the C_3A in the cement. Bensted [11] indicates that a C_3A content of more than 12% results in a visible extra hydration peak during a 20 °C isothermal hydration test. In earlier research [6,8] it was observed that this hydration peak, related to the transformation of ettringite into monosulphate can also be seen in Portland cements with lower C_3A content (e.g. 7.5%) when tested at temperatures of 40 °C to 50 °C. This transformation might be activated by the presence of the limestone filler, explaining the observed alteration even during the isothermal tests at the lower temperatures of 10 °C and 20 °C. This would also explain the increasing hydration rate of the second reaction with decreasing c/p. For the mixtures based

Table 4
Maximum heat production rate q_{max} (J/g/h)

Mixture	q_{max} at 10 °C	q_{max} at 20 °C	q_{max} at 35 °C
1/42.5 R	4.03	8.01	17.15
2/42.5 R—LF	4.81	9.54	19.40
3/42.5 R—LF	5.07	9.14	21.90
4/42.5 R—LF	5.40	10.48	27.87
1/52.5	7.20	14.32	30.06
2/52.5—LF	8.57	16.42	33.54
3/52.5—LF	8.26	15.52	34.58
4/52.5—LF	8.53	17.49	35.02
1/52.5 HSR LA	6.42	12.14	23.44
2/52.5 HSR LA—LF	7.37	13.62	27.44
3/52.5 HSR LA—LF	7.05	12.93	26.04
4/52.5 HSR LA—LF	7.07	14.54	23.58
1/42.5 R	4.03	8.01	17.15
2/42.5 R—QF	4.76	9.35	14.88
3/42.5 R—QF	4.99	8.66	15.71
4/42.5 R—F	4.40	7.95	16.85

Table 5
Total heat Q_{max} (J/g)

Mixture	Q_{max} at 10 °C	Q_{max} at 20 °C	Q_{max} at 35 °C	Theoretical heat at complete hydration
1/42.5 R	256.8	280.8	289.0	421.5
2/42.5 R—LF	291.4	319.1	313.6	421.5
3/42.5 R—LF	317.5	341.3	353.2	421.5
4/42.5 R—LF	349.6	365.0	362.9	421.5
1/52.5	342.7	357.1	367.4	442.8
2/52.5—LF	396.0	402.6	383.4	442.8
3/52.5—LF	413.9	430.3	440.2	442.8
4/52.5—LF	456.8	468.2	461.3	442.8
1/52.5 HSR LA	297.4	311.9	331.0	431.2
2/52.5 HSR LA—LF	344.0	349.6	354.6	431.2
3/52.5 HSR LA—LF	354.6	362.1	378.2	431.2
4/52.5 HSR LA—LF	372.5	377.9	352.9	431.2
1/42.5 R	256.8	280.8	389.2	421.5
2/42.5 R—QF	283.2	319.6	278.0	421.5
3/42.5 R—QF	330.1	324.7	336.2	421.5
4/42.5 R—QF	323.0	331.6	401.5	421.5

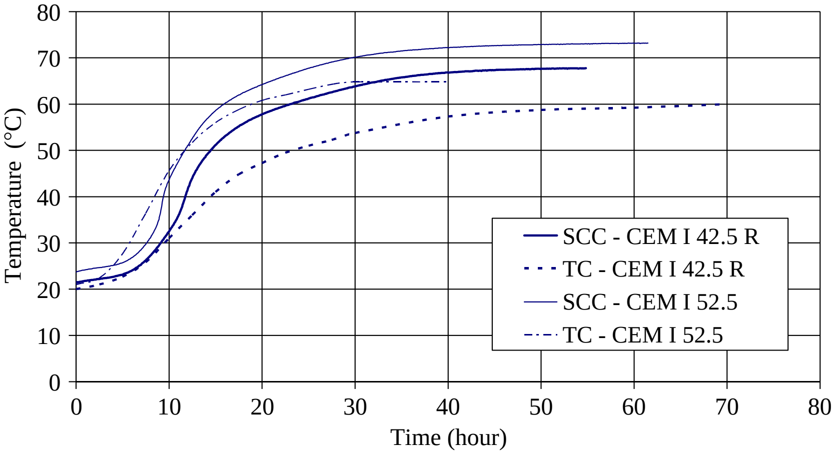

Fig. 6. Results of adiabatic hydration tests.

on Portland cement CEM I 52.5 (C_3A content of 8.3%) however, the activation by the presence of the limestone filler is apparently much less pronounced. This might be explained by the finer grading of the cement, which initiates the second reaction earlier during the hydration process and as a consequence coincides with, and is masked by, the first peak. For the case of Portland cement CEM I 52.5 HSR LA, no extra peaks can be observed, neither for the pure cement, nor for the mixes with the limestone filler. For this latter cement, the C_3A content is indeed very low (2.5%), which seems to confirm the hypothesis.

Another approach is starting from the principle that limestone filler is not inert and does not only act as an activator for some reactions, but actually takes part in the hydration reactions. Research carried out by Bonavetti et al. [18] revealed that in Portland cements, the limestone filler modifies the reactions. Firstly, ettringite formation is accelerated by the presence of the filler, and secondly, the ettringite conversion to monosulphate is delayed or even stopped when a large amount of carbonate is present in the paste. After 3 days monocarboaluminate is detected in the hydrating paste. This compound was also found by other researchers at different times related to the C_3A content of the cement. This hydration product is probably supplied by the transformation of monosulphoaluminate to monocarboaluminate the latter compound being more stable. This reaction might cause the second hydration peak detected in the isothermal and adiabatic hydration tests on CEM I 42.5 R and CEM I 52.5. If this is indeed the case, this transformation provides a new source of sulphate ions in the concrete, with possible delayed ettringite formation as a consequence. Further research has begun to look more closely into this, and to determine if ettringite is actually formed.

4.2. Heat production rate

A more detailed investigation of the maximum heat production rates during the isothermal tests (Table 4) leads to the finding that this maximum rate also seems to be influenced by the addition of limestone filler. Addition of

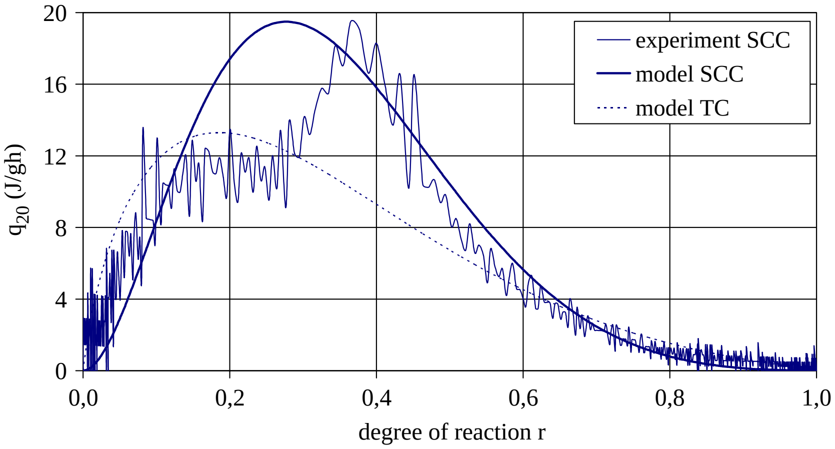

Fig. 7. Adiabatic test CEM I 42.5 R.

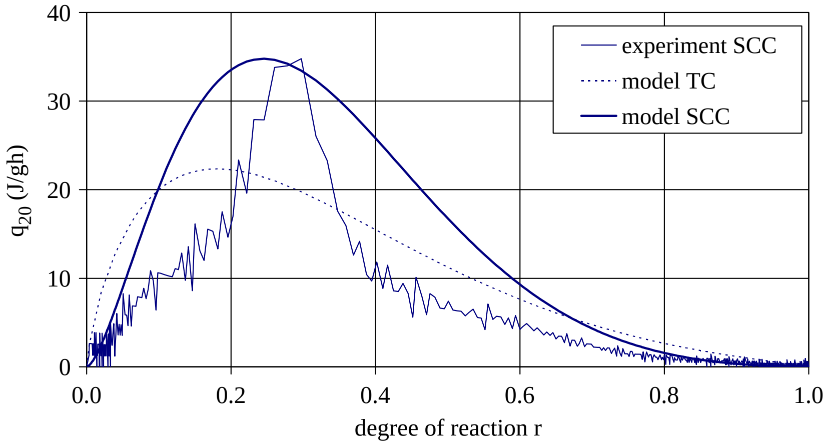

Fig. 8. Adiabatic test CEM I 52.5.

the limestone filler causes an increase in the maximum hydration production rate, unlike the addition of quartzite filler, where the maximum heat production rate hardly changes with varying *c*/*p* ratio. For the cement–limestone combinations, there seems to be a linear relationship between $q_{\max}$ at 20 °C and *c*/*p* ratio (Fig. 9).

4.3. Cumulative heat

The values of the cumulative heat at the end of the tests are given in Table 5 for the different mixtures. The theoretical cumulative heat at complete cement hydration can be estimated as the sum of the heats released during the hydration of the individual cement constituents [5]. It can be noticed that more heat is released for the mixtures with limestone addition than for the mixtures without, and this can be explained by the varying *w*/*c* ratio of the different cement–filler mixtures. For the case of CEM I 52.5 it should be noted that for mixture 4 the experimentally obtained cumulative heat is higher than the theoretical heat at complete hydration of the cement: the reason for this finding is not clear at this moment.

5. Hydration model

For the further evaluation of the hydration process in SCC in comparison with TC, the hydration model developed in [8] is used. In this model, the heat production rate of a Portland cement is calculated as follows:

$$q = q_{\max,20} \cdot f(r) \cdot g(\theta) \tag{3}$$

$$f(r) = c \cdot [\sin(r\pi)]^a \cdot \exp(-br) \tag{4}$$

$$g(\theta) = \exp\left[\frac{E}{R}\left(\frac{1}{293} - \frac{1}{273+\theta}\right)\right] \tag{5}$$

with $q_{\max,20}$ the maximum heat production rate at 20 °C, a, b and c the parameters, E the apparent activation energy and R the universal gas constant.

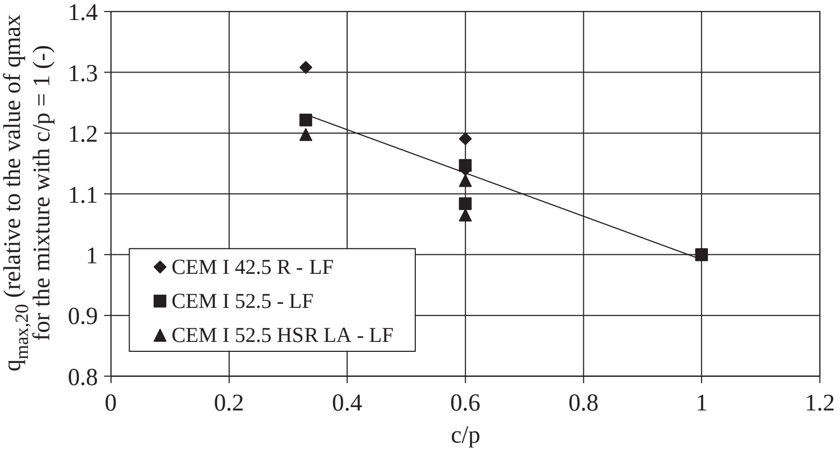

Fig. 9. Maximum heat production rate $q_{\max}$ at 20 °C.

When looking into the results of the isothermal tests on the mixtures and the results of the adiabatic tests on mix 1 and mix 3, it can be clearly seen that this hydration mechanism cannot be described as one function as done for a traditional concrete with Portland cement. The presence of the limestone makes a second reaction appear, as already discussed before. This reaction has to be modelled separately in the same way as done for a traditional concrete with blast furnace slag cement. The superposition principle is then applied in order to obtain the total cement reaction.

5.1. First reaction

When applying the model as in (3), (4) and (5) to the first reaction of the SCC hydration, the parameters calculated by the least squares method (in the same way as done for the traditional concretes), seem to be influenced by the addition of the filler. When the parameter a_1 is graphically interpreted as a function of c/p for the different mixtures, as has been done for $q_{\max,20}$, it is found that a second degree relationship exists between these variables (Fig. 10). This relationship still exists when b_1 is fixed at 3. This means that the first hydration reaction can be described mathematically as follows:

$$q_1 = q_{1,\max,20} \cdot c_1 \cdot [\sin(r_1\pi)]^{a_1} \exp(-3r_1) \times \exp\left[\frac{E_1}{R}\left(\frac{1}{293} - \frac{1}{273+\theta}\right)\right] \quad (6)$$

$$a_1 = -0.18(c/p)^2 + \gamma_1(c/p) + \gamma_0 \quad (7)$$

$$c_1 = -0.28(c/p)^2 + \beta_1(c/p) + \beta_0 \quad (8)$$

with β_1, β_0, γ_1 and γ_0 being parameters depending on the type of cement and filler used in the mixture, E_1 the apparent activation energy of the first reaction and R the universal gas constant. The values for β_1, β_0, γ_1 and γ_0, determined with the least squares method, are given in Table 6. The values for E_1 for the different cements are determined using the $q_{\max}$ values at different temperatures and the Arrhenius function in combination with the least squares method (Fig. 11). These values are also given in Table 6.

Table 6
Parameters of the hydration model for SCC

	CEM I 42.5 R—LF	CEM I 52.5—LF	CEM I 52.5 HSR LA—LF	CEM I 42.5 R—QF
E_1 (kJ/mol)	43.13	38.59	32.49	43.13
E_2 (kJ/mol)	81.79	–	–	–
β_1	0.69	0.69	0.69	0.56
β_0	2.30	2.43	2.43	2.43
γ_1	0.46	0.46	0.46	0.38
γ_0	0.47	0.56	0.56	0.56

5.2. Second reaction

The second reaction activated by the presence of the limestone filler in mixtures with Portland cement CEM I 42.5 R (cement with a considerable C_3A-content) can be described as (Fig. 12):

$$q_2 = q_{2,\max,20} \cdot [\sin(r_2\pi)]^{a_2} \cdot \exp\left[\frac{E_2}{R}\left(\frac{1}{293} - \frac{1}{273+\theta}\right)\right] \quad (9)$$

with $q_{2,\max,20}$ the maximum heat production rate of the second reaction at 20 °C, a_2 the parameter, E_2 the apparent activation energy of the second reaction (because the two reactions have a different temperature sensitivity, E_1 and E_2 do not have the same value) and R the universal gas constant. The values for E_2 are given in Table 6.

Because the second reaction is much less pronounced during the hydration of the mixtures with CEM I 52.5, it is

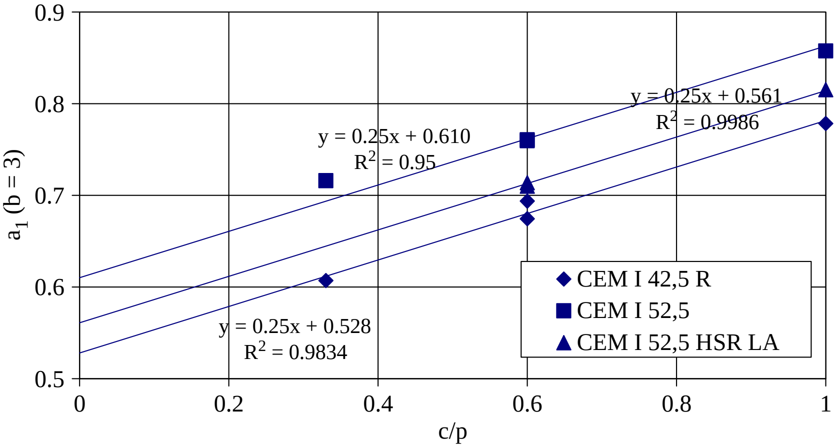

Fig. 10. Parameter a_1 of the hydration model as a function of c/p.

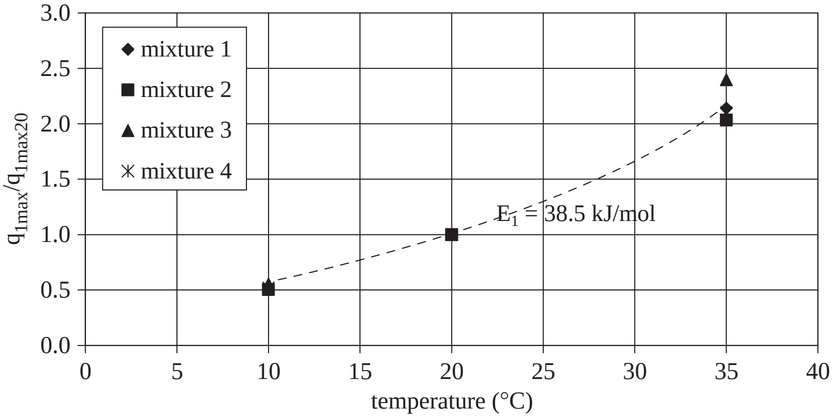

Fig. 11. E_1 determined by means of isothermal hydration tests for CEM I 42.5 R.

nearly impossible to separate the two reactions in a correct way. Therefore the reaction mechanism for these mixtures is modelled as one reaction.

As can be seen in Fig. 12, the second reaction does not start immediately after water addition. It is not yet clear why this is happening. Further tests are being done which will investigate the nature of the second reaction, and how and at which moment during hydration it is activated.

6. Conclusions

Based on isothermal and adiabatic hydration tests on self-compacting and traditional concrete, incorporating different types of Portland cement, the following conclusions are obtained.

- In some cases, the reaction mechanism of the Portland cement is clearly influenced by the addition of the limestone filler. The induction period is shortened and an extra heat production peak sometimes occurs, even at the lowest testing temperatures. These phenomena are not found when a quartzite filler is used in the mixes.
- The heat production rate of the second peak is clearly influenced by the presence of the limestone filler. The higher the amount of filler and the higher the testing temperature, the more pronounced the peak is. A linear dependency of the maximum heat production rate on the c/p ratio can be accepted in a first approximation.
- Because of the modification of the hydration reaction in the case of SCC, the hydration model developed for TC shows some discrepancy with experimental results when applied to the case of SCC. Using the results of the hydration tests, the model can be modified with inclusion of the second peak and adjustment of the parameters. Fixing parameter b_1 at 3, the parameters a_1 and c_1 seem to be dependent from c/p.

In this part of the research the influence of the chemical admixtures is not included yet. This will be the object of further investigations.

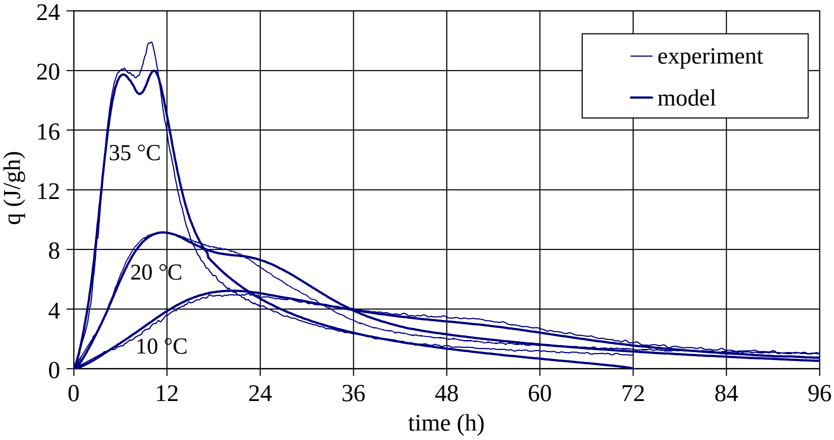

Fig. 12. Hydration of mix 3, CEM I 42.5 R and LF.

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