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Kinetics of Heat Release of Mechanically Activated Cement-Sand Composition

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Abstract

Introduction. Mechanical activation of the dry components of cement composites is utilized in order to increase the initial strength, improve the rheological characteristics of the mixture, as well as to reduce the setting time by increasing the reactivity of the binder. Heat release kinetics of components of cement composites modified by means of mechanical activation methods has been insufficiently investigated and studying its changes is an urgent task. In order to describe heat release kinetics, well-known equations are used relating a degree of hydration and a relative heat release. The aim of this study is to examine heat emission of a mechanically activated cement-sand composition as well as to identify parameters of equations describing heat release kinetics.

Materials and Methods. The investigated mechanically activated composition consists of cement and sand with a weight ratio of 1:2.14, with a specific surface area of 3690.8 cm^2/g. Using an isothermal calorimeter, a calorimetric analysis of a mechanically activated cement-sand composition, as well as non-activated cement, was conducted, and heat flux data were obtained.

Research Results. The cement in the composition of the mechanically activated composition was found to be moderately thermal in terms of heat release, with heat release values of 247 J/g and 281 J/g at the age of 3 and 7 days, respectively. During preliminary mechanical activation of the cement-sand composition, the time of the induction period and that to reach 50% of the maximum heat release of cement are reduced by 1.34 and 1.76–1.79 times, respectively.

Discussion and Conclusion. In the course of the study, the heat release kinetics of a mechanically activated cement-sand composition is described. A decrease in the induction period of the hydration process during mechanical activation of cement has been identified confirming the efficiency of mechanical activation of initial dry concrete components. The results can be practically applied in plants for producing dry building mixtures and concretes while introducing the technology of mechanical activation of concrete composite components.

Keywords: cement, hydration reaction, reaction rate, mechanical activation, cement-sand composition, heat release kinetics

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Оригинальное эмпирическое исследование

Кинетика тепловыделения механоактивированной цементно-песчаной композиции

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Аннотация

Введение. Механическая активация сухих компонентов цементных композитов используется для повышения начальной прочности, улучшения реологических характеристик смеси, сокращения сроков схватывания за счет повышения реакционной способности вяжущего. Кинетика тепловыделения компонентов цементных композитов, модифицированных методами механической активации, на данный момент мало изучена, и исследование ее изменения является актуальной задачей. Для описания кинетики тепловыделения используются известные уравнения, связывающие степень гидратации и относительное тепловыделение. Целью данной работы является исследование процесса тепловыделения механоактивированной цементно-песчаной композиции с определением параметров уравнений, описывающих кинетику тепловыделения.

Материалы и методы. Исследуемая механоактивированная композиция состоит из цемента и песка с отношением по массе 1:2,14, удельная поверхность — 3690,8 см^2/г. С помощью изотермического калориметра проведен калориметрический анализ механоактивированной цементно-песчаной композиции, а также неактивированного цемента, получены данные тепловых потоков.

Результаты исследования. Определено, что цемент в составе механоактивированной композиции по показателям тепловыделения относится к умереннотермичным со значениями тепловыделения в возрасте 3 и 7 суток 247 Дж/г и 281 Дж/г соответственно. При предварительной механической активации цементно-песчаной композиции сокращаются время индукционного периода и время достижения 50 % от максимального значения тепловыделения цемента в 1,34 и 1,76–1,79 раз соответственно.

Обсуждение и заключение. В ходе проведенных исследований описана кинетика тепловыделения механоактивированной цементно-песчаной композиции. Установлено уменьшение индукционного периода процесса гидратации при механической активации цемента, что подтверждает эффективность механоактивации исходных сухих компонентов бетонов. Полученные результаты могут иметь практическое применение на заводах по производству сухих строительных смесей и бетонов при внедрении технологии механической активации компонентов бетонных композитов.

Ключевые слова: цемент, реакция гидратации, скорость реакции, механическая активация, цементно-песчаная композиция, кинетика тепловыделения

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Introduction. Cement is the major binder characterizing structure formation of concrete as a whole. The initial Portland cement powder commonly consists of four main phases: alite (C_3S), belite (C_2S), aluminate (C_3A) and ferrite (C_4AF) [1]. When the cement binder is mixed with water, a hydration process occurs accompanied by heat release. Varying intensity of the reaction and its high dynamism over time was the rationale for dividing the entire hydration process into separate periods that are easy to record while analyzing changes in the heat release of the system [2].

When the binder is mixed with water, intense but short-term heat generation occurs, lasting only a few minutes (initial stage). This stage is associated with the initial activity of C_3S due to the wetting effect, as well as the reaction of the aluminate phases. The heat release is then stabilized to a relatively constant rate during the induction period, and the solution is stored in a relatively ductile liquid state for about 2 hours. The reasons for the induction period and its completion are extensively discussed, and the most popular hypothesis is currently the formation of protective membranes around cement particles, which requires the expenditure of thermal energy [2; 3]. Notwithstanding a specific mechanism, the end of the induction period is characterized by a sharp increase in the rate of heat release largely caused by the formation of reaction products: calcium hydrosilicates (HSC) and calcium hydroxide (GC). This acceleration period ends with the maximum heat release (after about 10-12 hours). After that, heat generation slows down (the deceleration stage) to a steady level (the period of hardening or slow interaction) when hydration continues for a period of several months to several years. As a result of the long reaction period, the structure and, more importantly, the porous structure of the hardened cement paste will constantly change over time.

Concrete heat generation and strength gain are known to be closely linked [4, 5]: the faster the hydration process occurs, the more intense the growth of crystallohydrates becomes forming the solid structure of cement stone and the entire composite. The degree of hydration α_τ associated with the reaction rate can be determined based on the mechanical and physical properties represented by the kinetics of strength gain and heat release kinetics:

$$\alpha_\tau = \frac{R_\tau}{R_{28}} = \frac{Q_\tau}{Q_{max}},$$

where τ — solidification time, days; R_τ — strength at the age of τ, MPa; R_{28} — strength at the age of 28 days, MPa; Q_τ — the value of the integral heat dissipation at a time τ, J/g; Q_{max} — maximum value of cement heat release at the age of 28 days, J/g.

In order to describe the heat release kinetics related to that of strength gain, an equation similar in structure to EN 1992-1-1[6; 7] is used[1]:

$$\frac{Q_\tau}{Q_{max}} = \exp\left(k \cdot (1 - \left(\frac{28}{\tau}\right)^d)\right), \quad (1)$$

where *k, d* — are the equation parameters.

There are a few more equations used in order to describe the heat release rate of the binder during hardening. In the Russian literature, the formula by I.D. Zaporozhets is used in order to describe the heat release kinetics [8, 9]:

$$\frac{Q_\tau}{Q_{max}} = 1-(1 + A \cdot \tau)^{-n}, \quad (2)$$

where A — coefficient of growth rate of heat release at the hardening temperature t +20 ºC, h^{-1}; n — indicator depending on the properties of the cement.

In overseas practice a formula by T. Knudsen [10–12] is used:

$$\frac{1}{Q_\tau} = \frac{1}{Q_{max}} + \frac{\tau_{50}}{Q_{max}\cdot(\tau - \tau_s)}, \quad (3)$$

where τ_{50} — release time of 50 % from Q_{max}; τ_s — duration of the induction period.

The heat release of cement depends on a host of factors, such as the phase composition, the amount of mixing water, storage and hardening conditions, etc. Another major factor is the surface condition of the cement grains and their size. Cement is known to have the highest chemical activity immediately following its manufacture and to decrease over time. In order to increase the reactivity of the binder, there are some methods of its activation, mechanical activation being the most common one [13]. During mechanical activation, a fresh, developed and chemically active particle surface is formed helping to increase thehydration reaction rate and change the nature of heat release.

The authors have developed a mechanically activated cement-sand composition (CSC) that has been proven to be efficient in the production of cement composites [14]. It is shown that the preliminary joint mechanical activation of the dry components (cement and sand) that constitute the concrete mixture increases the initial strength of the composite by more than 2 times, improves the rheological characteristics of the mixture and reduces the setting time. The heat release kinetics of components of cement composites modified by mechanical activation methods is insufficiently investigated and studying its changes is an urgent task.

The aim of the study is to investigate heat release of a mechanically activated cement-sand composition as well as to identify the parameters of the equations describing the heat release kinetics.

Materials and Methods. The investigated mechanically activated composition consists of cement and sand with a weight ratio of 1:2.14. In the experiments, Portland cement Eurozem 500 super, CEM I 42.5H manufactured by Petersburg Cement LLC, Russia was utilized. Construction sand, fractional composition: 70% is a fraction of 2.5–1.25 mm, 30% is a fraction of 0.63–0.315 mm. The mechanical activation was performed in a Retsch Emax laboratory ball mill designed for ultrafast and ultrafine grinding. The activation mode is 5 minutes long at a speed of 1000 rotations/min. The specific surface area of the mechanically activated composition is 3690.8 cm^2/g [14].

As part of the study, a calorimetric analysis of the mechanically activated CSC was conducted in compliance with GOST 310.5-881[2]. This method enables us to investigate the kinetics of the cement hydration reaction by analyzing the hardening thermodynamics of the solution. In order to examine the processes of heat release, an 8-channel TAM Air isothermal calorimeter was used allowing analyzing the heat release of reactions in the isothermal mode, as well as the kinetics of exothermic and endothermic reactions. In the isothermal mode of operation of the device, the thermostat maintains a constant system temperature throughout the entire experiment with any thermal effects associated with a chemical or physical process in the sample being recorded. Isothermal measurements provide quantitative information reflecting the rates of the investigated processes.

All of the channels of the calorimeter are double, including a sample and a reference standard in 20 ml ampoules. The principle of operation of the calorimeter is to compare and then record the heat flow velocity of the test sample with a reference standard represented by an inert material. 6 grams of the investigated mechanically activated CSC and 3 grams of water were placed in each ampoule. Quartz sand was used as the reference standard with its mass calculated based on ensuring the equivalence of the heat capacity of the components of the investigated sample and the reference standard, and in this study it is 21 grams. The experiment was 120 hours long. The accuracy of the device temperature setting is ± 0.02 °C, the sensitivity limit is 4 μW, the error is ± 23 μW. The heat dissipation was investigated using 4 samples of a mechanically activated CSC, and the experimental results waswere statistically processed in order to adequately evaluate the resulting data.

[1] EN 1992-1-1 (2004) (English): Eurocode 2: Design of Concrete Structures — Part 1–1: General Rules and Rules for Buildings. European Committee for Standardization Edt; 2004. 227 p.

[2] GOST 310.5-88 "Cements. Method for determining heat dissipation": introduction. 01.01.1989. Moscow: Publishing House of Standards; 1988. 6 p.

Considering that in the investigated mechanically activated composition, it is only cement (as a reactant) that has heat generation when mixed with water, the obtained values of the integral heat generation of the mixture were recalculated by 1 gram of cement. In order to compare the processes taking place in the activated and non-activated system and to confirm the efficiency of mechanical activation relative to the rate of hydration reaction, a calorimetric analysis of untreated cement was also performed.

Research Results. The result of the calorimetric analysis of the investigated samples was a set of heat flow data that was used to obtain integral heat dissipation data, which is subject to further processing. As the most active heat release of the investigated systems takes place in the first 24 hours, the values of integral heat release were selected for further research in 2-hour increments on the first day, then in 12-hour increments at the age of 2–5 days. Table 1 shows the values of the integral heat release of the mechanically activated CSC (Q), the recalculated values of heat release per 1 g of cement ($Q_{отн.CEM}$) and the values of relative heat release obtained by dividing the values of heat release at time τ by the maximum value of heat release of cement at the age of 28 days (Q/Q_{max}). The value of the maximum heat release of cement CEM I 42.5N (M500) in compliance with SP 41.13330.20121 is Q_{max} = 385 J/g (kJ/kg) SP 41.13330.2012[3] is Q_{max} = 385 J/g (kJ/kg).

Table 1

Heat dissipation indicators of a mechanically activated CSC

τ, h	Q, J/g	$Q_{отн.CEM}$, J/g	Q/Q_{max}	τ, h	Q, J/g	$Q_{отн\ CEM.}$, J/g	Q/Q_{max}
2	0.951	2.988	0.008	22	49.511	155.465	0.404
4	2.976	9.346	0.024	24	51.834	162.758	0.423
6	7.246	22.752	0.059	36	62.415	195.984	0.509
8	13.692	42.993	0.112	48	69.605	218.560	0.568
10	20.969	65.843	0.171	60	74.738	234.677	0.610
12	28.257	88.728	0.230	72	78.786	247.387	0.643
14	34.909	109.613	0.285	84	82.000	257.481	0.669
16	39.881	125.226	0.325	96	84.320	264.763	0.688
18	43.712	137.257	0.357	108	85.911	269.761	0.701
20	46.847	147.100	0.382	120	87.000	273.180	0.710

The values of the integral heat dissipation of the activated CSC represent the average values for 4 samples. Integral heat dissipation data was statistically processed for each time point, the average values of statistical indicators for the entire array of processed data are: standard deviation of the arithmetic mean m = 0.29; accuracy index ε = 0.59%; coefficient of variation V_c = 1.18%.

In order to determine the nature of the thermicity of the activated cement-sand composition, the values of integral heat generation at the age of 3 and 7 days were identified. Heat dissipation at the age of 3 days was 247 J/g, the value at the age of 7 days was obtained by means of extrapolation and amounted to 281 J/g. According to GOST 23464-79[4] , low-thermal cements include cements with heat release up to 230 and 270 kJ/kg in 3 and 7 days, respectively, and moderate-thermal cements — up to 315 kJ/kg in 7 days [7]. Hence the cement in the composition of the mechanically activated cement-sand composition is moderately thermal.

Table 2 shows the values of the integral heat release of inactive cement and those of the relative heat release.

[3] SP 41.13330.2012 "Concrete and Reinforced Concrete Constructions of Hydraulic Structures": introduced on 01.01.2013. Moscow: Ministry of Regional Development of the Russian Federation; 2012. 72 p.
[4] GOST 23464-79 "Cements. Classification": introduced on 01.07.1979. Moscow: Standardization Publishing House; 1985. 11 p.

Table 2

Indicators of heat release of cement CEM I 42,5N

τ, h	Q, J/g	Q/Q_{max}	τ, h	Q, J/g	Q/Q_{max}
2	1.328	0.003	22	107.582	0.279
4	4.829	0.013	24	116.539	0.303
6	10.538	0.027	36	153.226	0.398
8	19.580	0.051	48	175.406	0.456
10	31.527	0.082	60	191.484	0.497
12	43.570	0.113	72	201.146	0.522
14	57.606	0.150	84	206.723	0.537
16	71.197	0.185	96	211.234	0.549
18	83.883	0.218	108	214.707	0.558
20	95.495	0.248	120	217.540	0.565

The values of the integral heat release of the inactive cement represent the average values for 5 samples. Integral heat dissipation data was statistically processed for each time point, the average values of statistical indicators for the entire array of processed data are: the standard error of the arithmetic mean m = 1.55; the accuracy index ε = 2.30%; the coefficient of variation V_c = 4.29%.

Fig. 1 shows graphs of the heat release kinetics of a mechanically activated cement-sand composition (activated CSC) and non-activated cement (CEM I 42.5N). The integral heat release curves obtained experimentally and the theoretical values obtained by means of calculation using the equations of function (1), (2), (3) are shown.

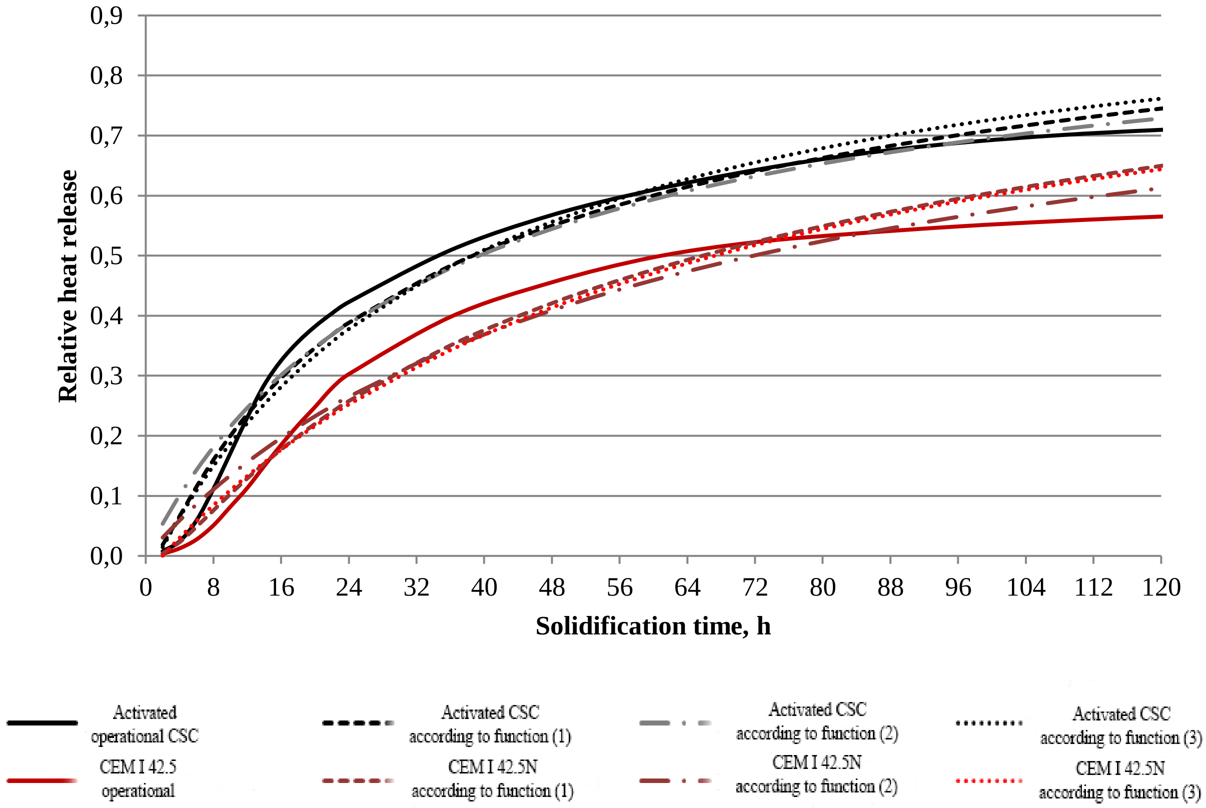

Fig. 1. Experimental and calculated relative heat releases of mechanically activated CSC and inactive cement

Parameters of the equations of functions (1), (2), (3) for the results shown in Fig. 1 are in Table 3 which also provides a comparison of the obtained parameter values with other researchers' data.

Table 3

Parameters of the equations of the heat release kinetics

Equation	$\frac{Q_\tau}{Q_{max}} = \exp(k \cdot (1 - \left(\frac{28}{\tau}\right)^d)$		$\frac{Q_\tau}{Q_{max}} = 1-(1 + A \cdot \tau)^{-n}$		$\frac{1}{Q_\tau} = \frac{1}{Q_{max}} + \frac{\tau_{50}}{Q_{max} \cdot (\tau - \tau_s)}$	
Designation	function (1)		function (2)		function (3)	
Parameter	k	d	A	n	τ_{50}	τ_s
Calculated for activated CSC	0.20	0.52	0.037	0.77	37.13	1.45
Calculated for CEM I 42,5N	0.32	0.5*	0.018	0.83*	65.30	1.97
G.V. Nesvetaev et al., for CEM I 42,5N [6, 15]	0.16–0.21	0.46–0.51	0.022	1.55	44.5	6

*The parameter is taken as [16]

According to the data, the value of the induction period for the activated cement-sand composition is τ_s = 1.45 hours. The estimated time of the induction period for the inactive cement was 1.97 hours. The end of the induction period of the activated composition occurs 1.34 times faster relative to the inactive cement.

The obtained parameter τ_{50} of the mechanically activated composition for equation (3) is 37.13 hours, which correlates with the experimental value of the time to achieve 50% heat release, which, according to experimental data, is 34.45 hours, the discrepancy between the results is 2.68 hours or 7.2%. The cement parameter τ_{50} for equation (3) is 65.30 hours, which also correlates with the experimental value, which is 61.59 hours, the discrepancy between the results is 3.71 hours or 5.7%. The time to reach 50% of the heat dissipation value for the activated composition is reduced by 1.76 and 1.79 times for the calculated and experimental values, respectively. Hence the efficiency of mechanical activation of cement is confirmed in relation to reducing the induction period and earlier start of the structure formation of a cement composite modified by means of mechanical activation of the components.

Discussion and Conclusion. As a result of the study, the values of integral heat generation for a mechanically activated cement-sand composition were obtained. It was found that the cement in the mechanically activated composition is moderately thermal in terms of heat release, with heat release values of 247 J/g and 281 J/g at the age of 3 and 7 days, respectively. The parameters of the equations describing the heat release kinetics are obtained. The values of the duration of the induction period for the mechanically activated composition (1.45 hours) and the inactive cement (1.97 hours) were identified. The time to reach 50% of the maximum heat release value of cement for the activated composition is 34–37 hours, while for the non-activated cement it is 61–65 hours.

The data obtained in the course of the study confirm the efficiency of mechanical activation of the initial dry components of concrete in terms of assessing the heat release kinetics and changes in the nature of thermality. When the cement-sand composition is mechanically activated, the time of the induction period and the time to reach 50% of the maximum heat release of the binder are reduced by 1.34 times and 1.76–1.79 times, respectively, indicating the efficiency of using the mechanically activated cement-sand composition as an additive in concrete. The results can be practically applied in plants for producing dry building mixtures and concretes while introducing the technology of mechanical activation of the components.

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Claimed contributorship:

AV Puzatova: formation of the basic concept, aims and objectives of the research, conducting the experiments and calculations, preparing the manuscript, forming the conclusions.

AD Kogai: conducting the experiments, forming the theoretical part.

MA Dmitrieva: scientific supervision, analysis of the research results, correction of the manuscript, correction of the conclusions.

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