



Research article

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Multiphasic modeling of hydration degree for blended cement pastes by using calorimetry method

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Abstract. The evolution of the mechanical properties of concrete depends greatly on the hydration of the component binders such as cement and mineral additives. However, the prediction of the hydration degree of these binders is extremely difficult due to the complex physic-chemical mechanisms at the molecular level. In this article, the author proposes to use a multiphasic model that considers hydration development and chemical interaction between reactions while taking into account temperature and water content effects on reaction kinetics. The main goal of this study is a semi-adiabatic calorimetry test was applied to determine the input parameters by measuring the heat release during hydration. Based on the test results shown the application of three cases of blended cement paste samples is considered to show the efficiency of the model. Overall, thermogravimetric analyses and its derivative are applied to verify the delay effect of pozzolanic reactions on the hydration degree induced by portlandite content in the paste.

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1. Introduction

Cement hydration is a process that consumes water and releases heat, accompanied by complex thermo-hydric mechanism of multiphases [1–3]. This process has important influence on the formation and evolution of the mechanical properties of cement paste at early age. However, the prediction of hydration degree and its evolution is extremely difficult due to the complex reaction mechanisms at the molecular level, especially when the paste contains some mineral additives (silica fume (SF), fly ash (FA), slag, etc.). In addition, the experiments that measure these quantities requires the modern and expensive equipment [4–6]. In order to resolve these difficulties, the numerical models are proposed to predict the evolution of hydration degree as well as the mechanical properties of cement mortar [7–18]. Several models that using separate global laws for the reactions of clinker and additions [11, 12, 14] reproduce the combined hydrations of these species but do not take account of the effect of water content on hydration. However, this effect explicitly cannot be neglected for a realistic prevision of mortar hydration in a structure. Some other authors report this essential effect and propose models coupling hydration development and water content variation in structures [17–19] as well as integration of hydration laws for mineral additions [15, 16], but this asymptotic approach cannot take account of the desiccation or rehydration effects during hydration. Wang and Lee [6, 7] proposed a shrinking-core model which considers the influences of the water/cement (W/C) ratio, cement compound compositions, and capillary water contents to simulate blended cement hydration. In this hydration model, the reaction of FA and slag is separated from that of cement hydration by considering the production/consumption of portlandite in cement paste. A similar approach to treat the reaction of slag separately from that of cement hydration is also proposed in [8]. The hydration of FA in blended cement is studied by Kinomura and Ishida [9, 10] who considering morphology and intrinsic properties of precipitated C-S-H gels due to pozzolanic reactions. An extensive modeling for continuous

hydration in pozzolanic micro-pore structures to consider the slow-down reaction is also proposed in that model.

In this article, the authors propose using an effort multiphasic model proposed by Lacarrière [1, 5] to predict the hydration degree of the binder composed. This modeling considers hydration development and chemical interaction between reactions. It also takes into account temperature and water content effects on reaction kinetics through thermal and hydric activation. The input parameters of the model are determined based on the chemical analysis of cement and mineral additives as well as the measuring of heat hydration by using Langavant calorimeter [18, 19]. The model is tested for the pure cement and blended cement paste (with FA and SF additives). Thermogravimetric analyses (TGA) and its derivative (DTG) are also applied to verify the delay effect of pozzolanic reactions on hydration degree induced by portlandite content in the paste.

2. Methods

2.1. Hydration Degree and Multiphasic Modeling of Hydration

In the frame of hydration kinetics, the hydration of cement as well as mineral additives is a process that consumes water and releases heat, accompanied by heat and water balance mechanisms between phases. The variation of water content and temperature inside the cement mortar during hydration affect the dynamic hydration process of the component phases (hydro activity and thermal activity). Thus, the multiphasic model of hydration should be established based on the equations, which describing the development of hydration degree, water content, and temperature. A delaying parameter is also added in the case of secondary reactions such as pozzolanic ones when we consider the hydration of a binder composed. It depends on the amounts of some primary reaction products (such as portlandite for pozzolanic reactions).

According to [1, 5], the equation describing the development law of the degree of hydration is proposed as a function of the influence coefficients as follows:

$$\alpha_i = A_i \times c_i(\alpha_i, W) \times \prod_i(\overline{r_{mi}}) \times h_i(T) \times g_i, \quad (1)$$

where:

- α_i is the degree of hydration of anhydrous phase “ i ” (clinker or additives), which refers to the development of chemical reactions between clinker, pozzolanic or mineral additions, and water. This variable is defined as the mass of anhydrous substance, which has reacted per unit volume divided by the initial mass, or indirectly by the ratio of heat release during the hydration and the maximal heat released when mortar completely hydrated.
- A_i is a fitting parameter linked to the acceleration of the reaction kinetics due to supersaturation, which determined by experiment of Langavant calorimeter.
- $c_i(\alpha_i, W)$ is chemical activation:

$$c_i(\alpha_i, W) = \frac{V_{Andiss_i}}{V_W} = \frac{\alpha_i \times \frac{m_{An_i}}{\rho_{An_i}}}{V_W}, \quad (2)$$

where: V_W is initial volume of water; m_{An_i} is initial mass of anhydrous phase “ i ”; ρ_{An_i} is anhydrous density of phase “ i ”; V_{Andiss_i} is volumetric concentration in paste of dissolved anhydrous phase “ i ”.

- $h_i(T)$ is thermal activation:

$$h_i(T) = \exp\left(\frac{E_{a_i}}{R \times T}\right), \quad (3)$$

where: E_{a_i} is the activation energy of phase “ i ”; R is the gas constant (8,314 J/mol.K).

- $\prod_i(\overline{r_{mi}})$ is hydration function, which models the water accessibility to anhydrous phases:

$$\prod_i(\overline{r_{mi}}) = \exp\left(B_i \times r_{mi}^{n_i}\right), \quad (4)$$

where: B_i and n_i are fitting parameters, which determined by Langavant experiment; $\overline{r_{mi}}$ is a function (i.e., dissolution radius) of water content, porosity and solid phases in the paste:

$$\overline{r_{mi}} = \frac{C_{Phydr_i} \times \frac{1}{\Phi_P}}{C_{Panh_i} \times W_P}. \quad (5)$$

In which, W_P is the volumetric concentration of water in the paste; Φ_P is porosity of paste; C_{Phydr_i} is the volumetric concentration of hydrate produced from grains of phase "i"; C_{Panh_i} is anhydrous volumetric concentration in paste:

$$\Phi_P = 1 - \sum_i (C_{Phydr_i} + C_{Panh_i}); \quad (6)$$

$$C_{Phydr_i} = R_i \times \alpha_i \times \frac{m_{An_i}}{\rho_{An_i}} \times \frac{1}{V_{paste_{ini}}}; \quad (7)$$

and:

$$C_{Panh_i} = (1 - \alpha_i) \times \frac{m_{An_i}}{V_{paste_{ini}} \times \rho_{An_i}}, \quad (8)$$

where: R_i is volume ratio between hydrates and anhydrous phase for the species "i" (cement or FA); $V_{paste_{ini}}$ is the initial paste volumetric concentration in paste.

– g_i is a factor delaying the hydration kinetics for pozzolanic constituent reactions depending on primary reactions;

$g_i = C_{pCH}$ if $i \neq$ clinker (C_{pCH} is the volumetric concentration of portlandite in the paste);

$g_i = 1$ if $i =$ clinker.

The schema of calculation formulas is summarized in Fig. 1. Note that input parameters in the model (except water content) are interpreted for each individual anhydrous phase "i".

The principles to determine the input parameters are introduced in Fig. 2. In this numerical model, the water content and anhydrous content are fixed for each studied sample. The parameters of clinker cement are determined through analysis of chemical composition (Bogue's composition) [22] and chemical reactions accompanying the hydration process [1]. On the other hand, the mineral additives parameters are calculated or referenced from literature (the detailed calculations are presented in [23]). Particularly, the fitting parameters of the model A_i , B_i , n were determined by the Langavant test.

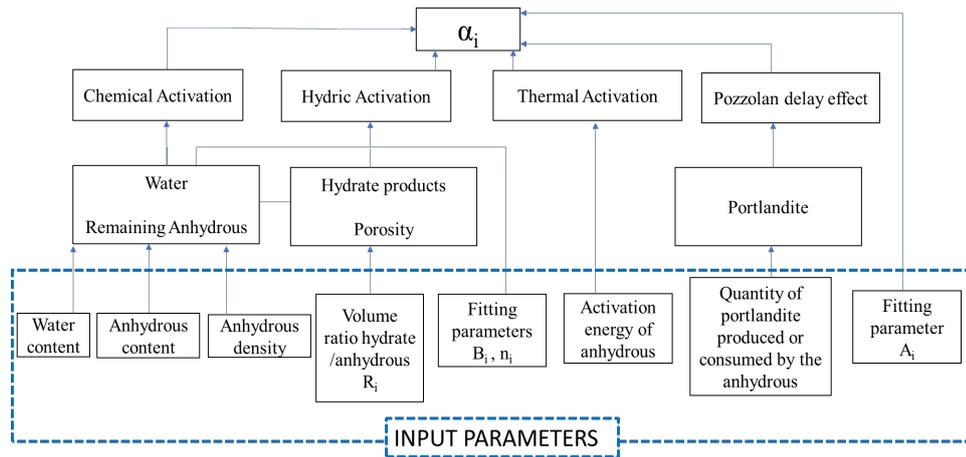


Figure 1. Schema for using input parameters of model.

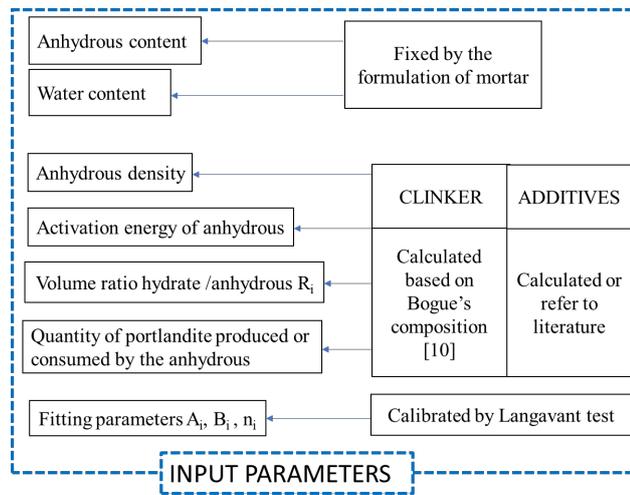


Figure 2. Determination of input parameters.

2.2. Calorimetry Method and TGA

2.2.1. Langavant Test

Langavant test for the heat of hydration of the cement method is semi-adiabatic calorimetry according to EN196-8, which consists of introducing a fresh cement specimen into an isolated Dewar flask (Fig. 3) and monitoring the temperature changes within the specimen during the first early days. After a certain time, the heat of hydration of the cement content in the sample is equal to the sum of the heat accumulated in the flask and the heat emitted to the environment during the test period. The temperature of the mortar is compared with the temperature of an inert sample placed in a reference calorimeter flask. Depending on the type of cement, in general, the heat emitted is from about 200–500 J / 1g of cement.

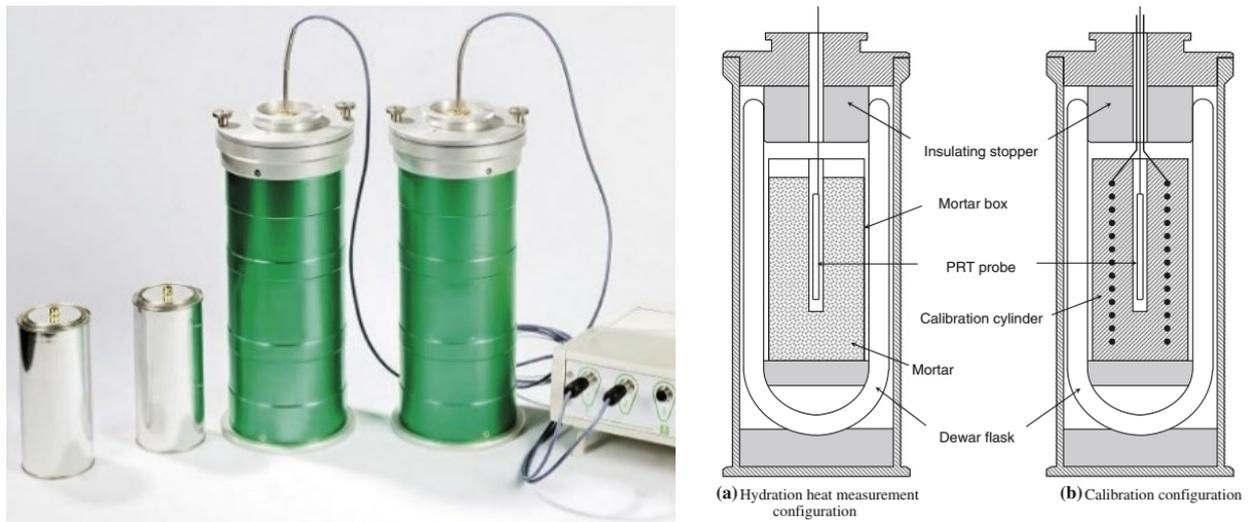


Figure 3. Schematic diagram of a Langavant calorimeter [20].

By the measuring of heat release during the hydration $Q(t)$, the degree of hydration at each time $\alpha(t)$ could be determined such as:

$$\alpha(t) = \frac{Q(t)}{Q_{\max}^t}, \tag{9}$$

where Q_{\max}^t is heat release of complete hydration. The parameters A_i , B_i , n were determined by fitting the numerical curve and experimental curve of heat release and using the least squares method to calibrate.

3.1. TGA

TGA is a technique for measuring very small variations in mass under high gradient temperature which permit to quantify the portlandite content of the cement paste. During a TGA test, the decomposition of cement hydration products is manifested by the weight loss curve and endothermic peaks on the heat flux curve. The representation of the weight loss in the form of its time DTG shows the correspondence between the DTG peaks and those of heat flux.

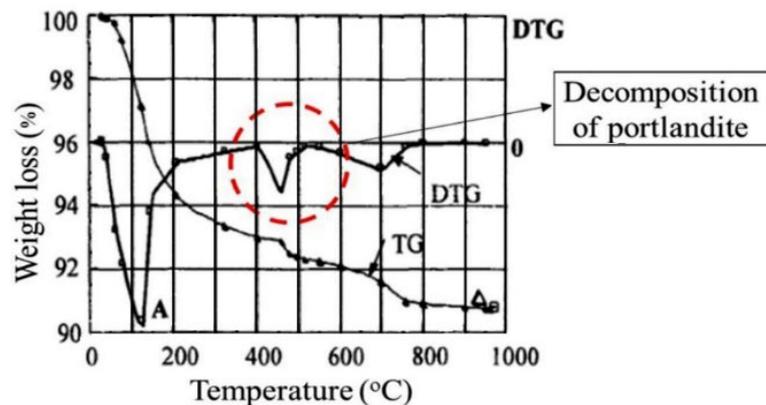


Figure 4. Weight loss of a cement paste sample during a TGA test [24].

Fig. 4 shows the typical result of a TGA test on a cement paste [24]. There are three major phases during the test and the second pic of weight loss correspond to the decomposition of portlandite, which is produced between 400 and 600 °C. This decomposition leads to an evaporation of chemically bound water and the mass content of portlandite in the mortar can be calculated from the following expression (10):

$$m_{\text{Ca(OH)}_2}(t) = \frac{\left[\Delta m_{400^\circ\text{C} \rightarrow 600^\circ\text{C}}(t) + m_{d,400^\circ\text{C} \rightarrow 600^\circ\text{C}}(t) \right]}{m_{\text{total}}} \times \frac{M_{\text{Ca(OH)}_2}}{M_{\text{H}_2\text{O}}}, \quad (10)$$

where: $\Delta m_{400^\circ\text{C} \rightarrow 600^\circ\text{C}}(t)$ is the weight loss of the sample between 400 and 600 °C [kg];

$m_{d,400^\circ\text{C} \rightarrow 600^\circ\text{C}}(t)$ is the mass drift correction of the device between 400 and 600 °C [kg];

m_{total} : the mass total of sample [kg];

$M_{\text{Ca(OH)}_2}$ and $M_{\text{H}_2\text{O}}$ are consequently the molar mass of portlandite and water [kg mol⁻¹].

3. Results and Discussions

In this report, three cement paste samples were made using CEM I 52.5N cement (France Standard) with the water/binder (with or no mineral additives) ratio chosen as W/B = 0.5. The first one is pure cement paste (100 % cement), the next is mixed cement paste with FA (70 % cement and 30 % FA), and the last one is mixture of 70 % cement and 30 % SF. The chemical compositions (%) of these materials are given in Table 1.

Table 1. Chemical compositions of cement CEM I 52.5N, FA, and SF (%).

Types of materials	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃
Cement	64.66	20.20	5.23	2.26	3.69
FA	1.00	60.76	29.72	3.49	0.09
SF	3.68	85.49	0.13	0.45	0.05

The input parameters of model are calculated or referenced from literature and showed in Table 2.

Note that the quantity of heat released by the complete hydration Q_{max}^T of clinker cement could be determined directly from the Bogue's composition and the theoretical heat releases associated with each anhydrous [25] while the activation energy E_{ai} of clinker cement is identified by the formulas of Schindler [26]. On the other hand, these parameters of FA and SF are all extracted from document [15]. The others parameters of model such as Q_{th}^W (the quantity of water necessary for complete hydration of each anhydrous species), R (the volume ration hydrate/anhydrous), and Q_{CH} (the quantity of portlandite produced/consumed by clinker hydration or pozzolanic reactions) are determined by considering the chemical reactions and also the type of hydrate product when hydration occurs [1, 2]. The detailed formulas of calculation procedure can be found in [23].

Table 2. Input parameter of binder compose.

Parameters	Cement	FA	SF
Q_{max}^T (J/g)	453.9	560	845
E_{ai}/R (K)	5669	12000	11600
Q_{th}^W (g/g)	0.33	0.64	0.8
Q_{CH} (g/g)	0.27	-1.51	-1.37
R (m ³ /m ³)	1.85	0.6	0.64
ρ (g/cm ³)	3.153	2.2	2.2

Table 3. Fitting parameters by calibration of model for each phase.

Parameters	Cement	FA	SF
B	3.147	30	6.08
n	0.230	0.098	0.483
A	3.80E+08	5.0E+30	1.24E+18

The three fitting parameters (A, B, n) were then calibrated using results from the Langavant tests performed on a binder composed (shown in Table 3). It should be noted that the calibration of the parameters of each anhydrous compound is done by successive calibration: first, the cement parameters are identified by a test on the pure cement and then, the parameters of the additives are based on a test on a mixture (the clinker parameters being known).

Fig. 5 showed the of evolution of heat release during the hydration at early age for three studied samples. It seems that in the first hours of the hydration, the obtain results of calibrated model are quite consistent with the results of the Langavant test, especially the case of pure cement sample. However, in the next stage, small differences between numerical model and experiment were observed and the differences were less obvious in the case of blended cement samples.

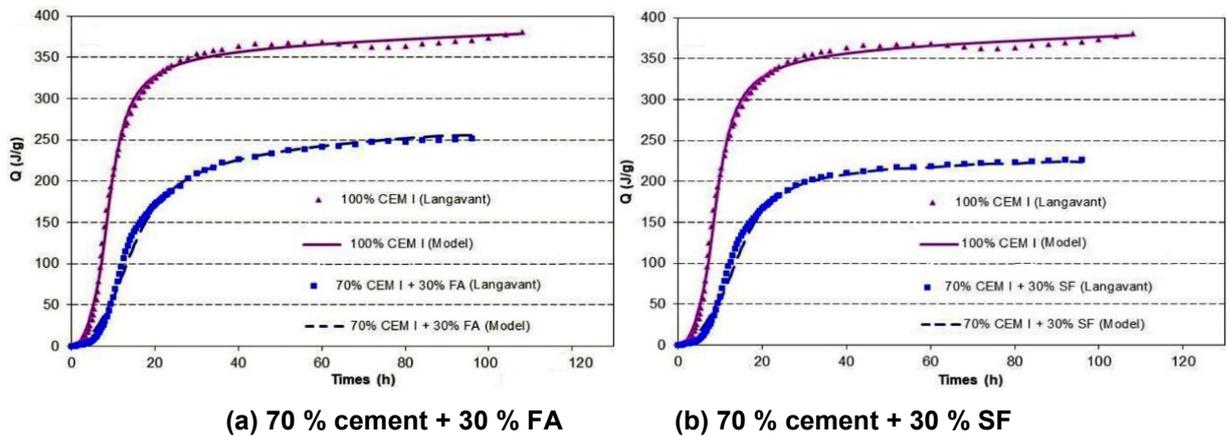


Figure 5. Heat release calculation of calibrated model and Langavant at early age.

The evolution of hydration degree of FA–cement sample at early age is presented in Fig. 6. We can observe that the hydration degree of pure cement sample is greater than this one of the “cement phase” in the mixture. This can be explained cause reducing the cement concentration in the anhydrous mixture may reduce the degree of hydration. In addition, the delaying effect of mineral additive is also observed at the first hours after mixing when the hydration degree of “FA phase” equal zero. After 5–7 hours, the pozzolanic reactions will active the FA and induce the evolution of its hydration degree [19].

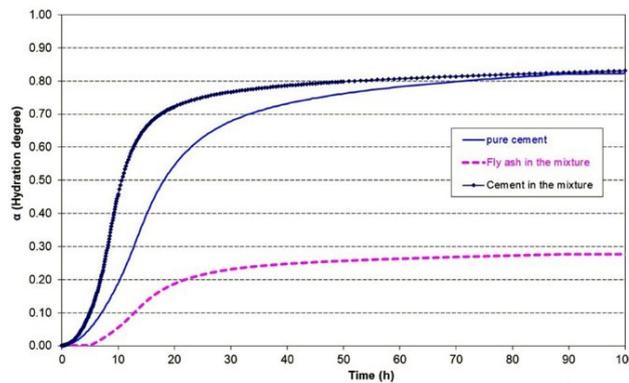


Figure 6. Evolution of Hydration degree of pure cement paste and binder composes of FA–cement paste at early age.

The evolution of hydration degree of clinker cement for long term are introduced in Fig. 7. It can be seen that the hydration degree evolved strongly in the first hours after mixing and occurs more slowly when the samples reach the age of 14 days. The hydration then progresses leisurely and become almost constant at 28 days. The maximum level of hydration reaches nearly 95 % for the case of pure cement sample.

The average hydration degree of the blended cement (α_{mix}) for long term is determined by the following equation (11):

$$\alpha_{mix} = \alpha_{cement} \times P_{cement} + \alpha_{additives} \times P_{additives}, \tag{11}$$

where: α_{cement} and $\alpha_{additives}$ are hydration degree of cement and mineral additive (i.e., FA and SF) in the mixture; p_{cement} and $p_{additives}$ are mass proportion of cement and mineral additive in the mixture. In this case, $p_{cement} = 0.7$ and $p_{additives} = 0.3$.

The evolution of hydration degree for the blended cement paste are introduced in Fig. 8. The maximum level of hydration reaches 80 % for the case of FA–cement sample and only 76 % for the case of SF–cement sample. It can be observed also that the replacement of clinker (30 %) by mineral additives leads to a significant decrease of hydration degree. On the other hand, the notable difference in hydration degree evolution between the sample of FA and SF is also observed. This can be explained by the presence of a large amount of SF while maintaining the same water/binder ratio leads to internal shrinkage and decreasing of relative humidity in the paste. This phenomenon may thereby cause lack of water for hydration reactions. It should be remembered that SF is a highly absorbent mineral [27] and in the production of concrete and cement containing additives, the replacement content is usually recommended not exceed 8 %.

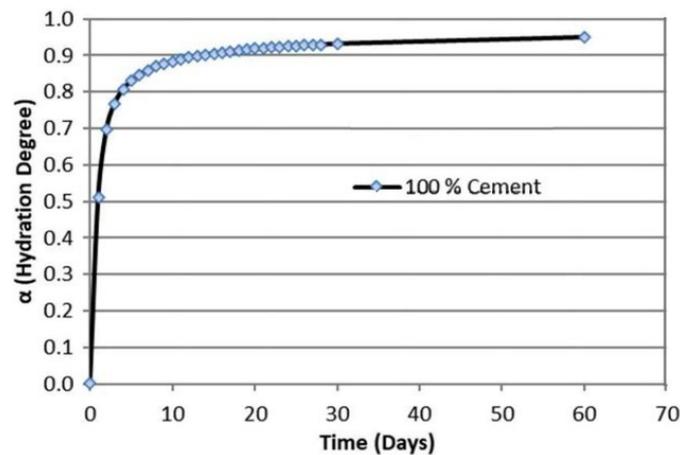
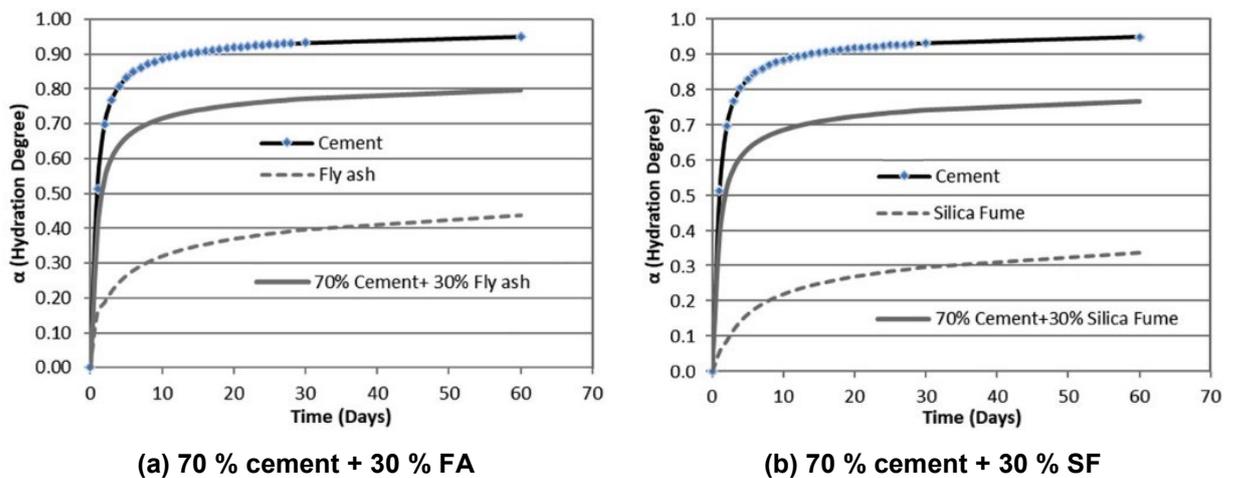


Figure 7. Evolution of hydration degree α of pure cement paste in long term.



(a) 70 % cement + 30 % FA

(b) 70 % cement + 30 % SF

Figure 8. Evolution of hydration degree α for blended cement paste.

The series of TGA/DTG tests on mortar were also carried out at a date of 3, 7, 28, and 60 days to estimate the effect of mineral additives on portlandite content in the paste. The evolutions of the TGA and DTG curves for each type of cement are related to the initial mass of the studied sample and presented in Figs. 9a–c. By comparing the DTG curves, we find that the mortar with 100 % cement has a large peak between 400 and 600 °C, and these peaks for mortars containing pozzolanic additions are less significant. This also prove that the portlandite produced in pure cement mortar is greater than that in mixtures paste as shown in Fig. 9d. The replacement of clinker by FA and SF reduces the clinker content and obviously the portlandite content produced in hydration process.

On the other hand, the portlandite content increases throughout the hydration process for the case of pure cement sample, but this increasing become slower after 28 days, corresponds with the completely hydration of clinker. In contrast, with mixtures containing FA and SF, the portlandite content begins to decrease after about one or two weeks and the speed of reduction becomes faster after 15 days due to the

effect of pozzolanic reactions. In addition, we can find that the portlandite content in the SF–cement sample is lower than that in the mixture contains FA. There are maybe two hypotheses to explain this phenomenon: first, the SF consume a lot of water, causing a lack of water for the hydration and obviously the quantity of portlandite produced is reduced. The secondary, pozzolanic reaction of SF is stronger than FA and it consumes more portlandite. However, when considering the hydration degree of the binder composites observed in Fig. 8, we can notice that the first hypothesis is more reasonable.

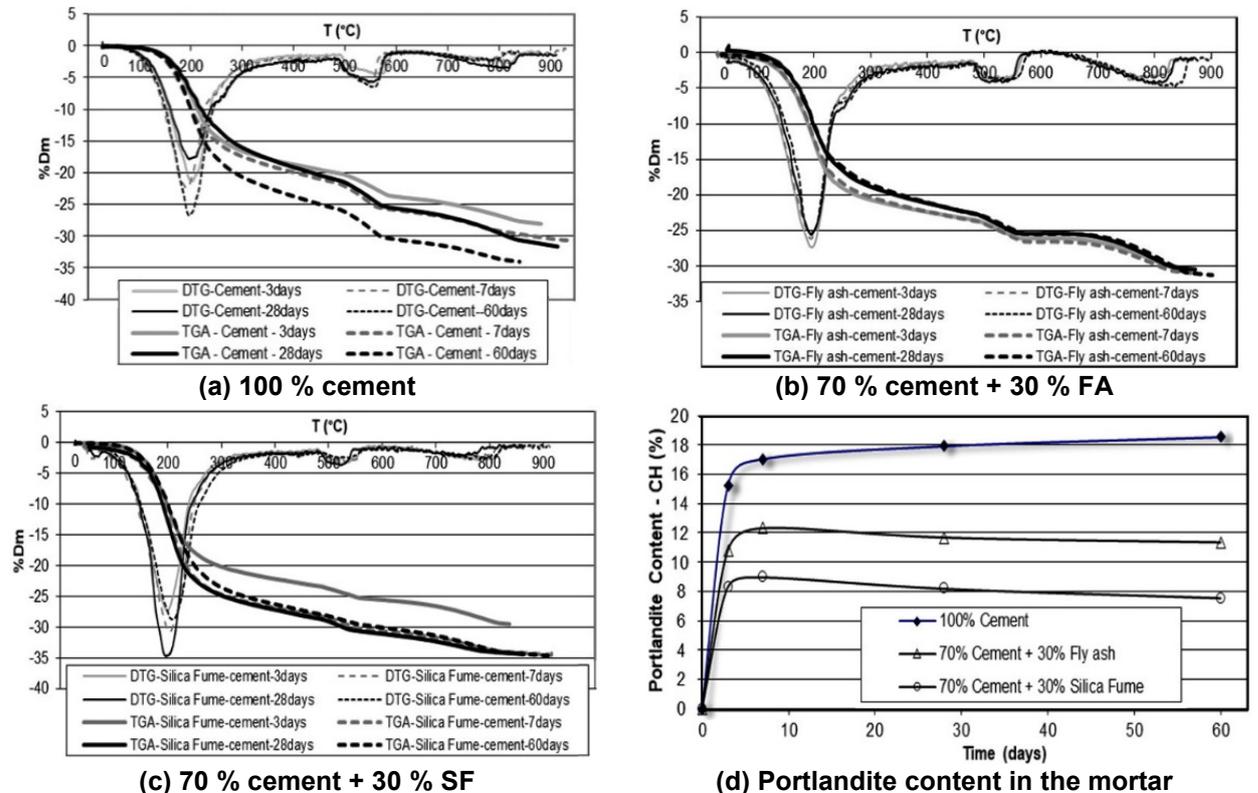


Figure 9. TGA/DTG results and the portlandite content of the studied samples at 3, 7, 28, 60 days.

4. Conclusions

In this article, a numerical model predicting the hydration development of multicomponent cement-based materials at an early age has been applied. The hydric-thermal activation and the delaying effect of mineral additives have been taken into account in the model. The fitting parameters of the model are determined by measuring the heat released during the hydration process using Langavant test. The model is applied to calculate the hydration degree of three cement paste samples containing pure cement and blended cement with 30 % replacement of FA or SF.

With the replacement of clinker by mineral additives leads to a significant decrease of hydration degree (from 95 to 75÷80 %) even if the effect of pozzolanic reactions, which increase the hydration degree of blended cement in the long term. The obtained results of TGA/DTG tests have allowed to calculate the portlandite content in the studied samples at difference term and showed us the correspondence with the results of established model.

In addition, the hydration degree of the mixture containing SF is lower than that of FA due to the lack of water in the paste when mixing a large amount of SF. From this result, it can be shown that the replacement content of SF is usually recommended not to exceed 8 %.

Finally, the obtained results have also shown the efficacy of multiphasic model in prediction of hydration kinetics of blended cement paste, especially at the early age.

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