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Study of Hydration Properties for Blast Furnace Slag-Blended Cement: Prediction of Slag Hydration Kinetics

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Abstract. Blast furnace slag (BFS) is an industrial by-product that has been used to replace clinker in Ordinary Portland Cement (OPC) to reduce the CO₂ footprint impact in the construction field. Moreover, the use of BFS leads to an enhancement in terms of mechanical and durability properties. A solid comprehension of hydration properties is essential to achieve a better understanding of durability properties. In this context, this study deals with the hydration processes (BFS)-blended cement in terms of kinetics. For that purpose, a simplified analytical model is proposed to get the evolution of the hydration degree in time. The input data required are the formulation parameters, *i.e.*, water-to-cement ratio (W/C) and the fraction of slag used respectively. The model is validated with experimental measurements of the evolution of the hydration kinetics, based on the results of a ThermoGravimetric Analysis (TGA), which are performed on different cement pastes formulated with BFS-blended cement. Finally, the present study is supplemented by a comparison between the results obtained by the TGA method and those provided by open-source software (VCCTL).

Keywords: Hydration degree · Blast Furnace Slag · Thermogravimetric analysis · Analytical modeling

1 Introduction

The cement industry emits about 6–8% of all CO₂ emissions. In this sense, reducing this excessive emission is one of the main objectives of studies on alternative binders. Therefore, researchers are focusing on combining Portland cement with mineral additions, or even replacing it, while retaining a beneficial effect on the overall behavior of the structure. One of the most widely used mineral addition is blast furnace slag, especially for concrete structures in marine environments [1, 2]. In [3, 4] several advantages related to low heat of hydration, long-term strength, and high chloride resistance have been

demonstrated. Then, studies show that slag cement increases compressive strength and durability properties such as chloride migration [3, 5, 6] and then corrosion initiation of the mixed material. Slag, combined with ordinary Portland cement, is now recognized as an essential ingredient in the manufacture of durable concrete structures, as it can extend the useful life of structures by fifty to one hundred years [7]. BFS has hydraulic properties as clinker given that both have almost the same chemical components. Activation of slag is however required due to its latent hydraulic properties. Different activation methods have been studied in the literature, such as alkaline, sulfate, or even alkaline-sulfate. The present study is mainly focused on the activation of slag by the Portlandite $\text{Ca}(\text{OH})_2$ formed during the clinker hydration [8–10].

Portland cement in blended cement begins to hydrate quickly after being combined with water. Meanwhile, due to the presence of gypsum in cement, a minor quantity of BFS reacts [11]. The hydration of BFS is then stimulated by the hydration of Portland cement. In this context, several researchers modeled hydration to understand the evolution of concrete properties.

Numerical models have been developed to describe the hydration mechanisms of slag-blended cement. Kolani et al. [12] modeled the hydration degree based on chemical stoichiometric calculations, and mass balance equations for heat and for moisture transfer which can be solved using a finite element method. In addition, the NIST Virtual Cement and Concrete Testing Laboratory (VCCTL) is a widely used numerical model [13]. However, those computations are time-consuming.

In parallel, to simplify the description of a binary formulation, many researchers studied the hydration of each component separately. Until the present, the reaction mechanism of BFS is not well understood and the chemistry of its hydrates remains unclear until now. This lack of knowledge on BFS hydration obstructs subsequently the comprehension of hydration in general and therefore the hydration modeling. Thus, there is no scientific agreement on a specific model. Park et al. [14] presented two hydration models for sodium silicate-activated slag: the first one, based on theoretical kinetics, considers phase equilibrium and mass conservation while the second one is based on empirical hydration kinetics involving calibration parameters. The results of these two models were coherent with the experimental ones. However, the models are valid for alkali-activated slag only. Königsberger et al. [15] suggested a hybrid modeling technique based on calorimetry data mixed with cutting edge hydration models. The investigation of intrinsic CASH nanoparticles associated to clinker and slag hydration, with precipitation space-dependent densities of the corresponding gel phases, distinguishes this model. This model requires experimental isothermal calorimetry observations as input data. Stephant et al. [16] applied Powers' model, valid only for OPC, to describe the hydration of slag-blended cement by extrapolating experimental data to estimate the ultimate bound water content at complete hydration. However, the experimental results were not well produced by the model. Chen and Brouwers [17] also developed a stoichiometric model which provides a relevant description of chemical reactions but without considering hydration kinetics which has been predicted by other researchers Wang [18], Darquennes et al. [19] based on an estimation of the ultimate hydration degree based on heat measurements. Merzouki et al. [20] proposed a kinetic

model of BFS blended cement, based on the Knudsen model [21] which involves calibration parameters estimated from experimental data. The input data of this model are the chemical compositions of cement and slag, the water/binder ratio (w/b), and the environmental conditions respectively. The results obtained showed that the hydration rate of slag-mixed cement pastes with low slag replacements (lower than 30%) is well reproduced. However, it is underestimated for higher percentages of slag.

Generally, the hydration degree of the composition is the average degree of all its components as presented in Eq. (1).

$$\alpha = f_{clinker} \times \alpha_{clinker} + f_{slag} \times \alpha_{slag} \quad (1)$$

where $f_{clinker}$, and f_{slag} are the cement, and the slag content in the mix, respectively. As the α_{slag} cannot be determined experimentally, the hydration degree is calculated by referring to the Thermogravimetric Analysis (TGA) results.

Based on (TGA), a method developed by Monteagudo et al. [22] and recently improved by Deboucha et al. [23] was developed to determine the hydration degree. This method is based on a measurement of the chemical bound water of mineral additives blended cement paste at time t “ $W_B(t)$ ” with respect to the ultimate chemically bound water ($W_{B\infty}$) and mass of equivalent binder (L_{eq}) in the sample as described in Eq. (2).

$$\alpha = (W_B(t))/(W_{B\infty} \times L_{eq}) \quad (2)$$

($W_{B\infty}$) is considered equal to 0.24, similar to that of 1 g of pure cement as assumed in [23]. The equivalent binder mass L_{eq} (g) is calculated as Eq. (3) by introducing the activity coefficient $K = 0.9$ for the studied slag according to EN NF 206-1 and NF P18-305 standards.

$$L_{eq} = m_C + K \times m_S \quad (3)$$

The calculation methods of m_C and m_S are given in Eqs. (4) and (5).

$$m_C = [m_{sample} - m_B \times (f_S + W/B)] / [(1 + LOI_C)] \quad (4)$$

$$m_C = [m_{sample} - m_B \times ((1 - f_S) + W/B)] / [(1 + LOI_S)] \quad (5)$$

where m_B , f_S , W/B , and LOI represents: the binder mass (g), the replacement rate of OPC by slag, the water/binder ratio, and the loss on ignition respectively. W_B can be calculated as follows:

$$W_B(t) = Ldh + Ldx + 0.41(Ldc - Ldc_S) - (m_C \times LOI_{CC} + m_S \times LOI_{SC}) + m_d \quad (6)$$

where Ld_h , Ld_x , Ld_c are defined in the next Sect. 2.2. Ldc_S , LOI_{ic} , and m_d are the relative mass loss during the decarbonation phase (between 600 and 1000 °C) of the anhydrous material, the loss on ignition of anhydrous powder mass, and the device’s drift which is the mass variation of an empty crucible (g) subjected to a high temperature respectively. The constant “0.41” represents the conversion factor, which allows assuming the bound water is derived from carbonated Portlandite [23].

The present study deals with the development of a simplified analytical approach to BFS-based cement kinetics by taking into account the physicochemical mechanisms involved during the hydration process, which can be applied to different BFS content formulations. The model is distinguished by the ability to estimate the evolution of the hydration degree with few input data related only to the formulation parameters (slag content and water to binder ratio).

2 Experimental Campaign

2.1 Materials

The experimental campaign was conducted on cement paste formulations by considering three substitution rates (by mass) of clinker by BFS, *i.e.*, 0, 50, and 80%, and are named P0, P50, and P80 respectively. A constant water/binder ratio of 0.5 was adopted in this study. An OPC CEM I 52.5 N produced by Lafarge is used in this study. The used BFS is produced by Ecocem in Dunkerque, France. The chemical compositions of OPC and BFS are shown in Table 1.

Table 1. Chemical composition of used OPC and BFS

Components (%)	OPC	BFS
SiO ₂	23.2	34.9
Al ₂ O ₃	3.1	10.9
Fe ₂ O ₃	2.24	0.5
CaO	66.4	42.3
MgO	0.76	6.9
SO ₃	2.22	0.1
K ₂ O	0.18	0.45
Na ₂ O	0.11	0.35
TiO ₂	0.15	0.7

The mixtures of the dry mix were prepared according to NF EN 196-3 standard and were cast into 40 * 40 * 160 mm³ prisms molds. Immediately after casting, molds were covered with plastic film for 24 h to avoid water evaporation. After demolding, samples were cured in water.

2.2 Characterization Methods

As mentioned before, the hydration degrees will be estimated from TGA measurements based on the method proposed by Deboucha et al. [23]. The samples dedicated to TGA are prepared with the protocol described by [23–25]. The decomposition of cement hydrates

was demonstrated from the endothermic peaks on the derivative TGA. In general, cement hydrate decomposition may be divided into different stages:

- between 25 and 400 °C:
 - removal of the evaporable water (25 to 105 °C),
 - dehydration reaction of CSH noted as “Ldh” (105 to 400 °C),
- between 400 and 600 °C: dehydroxylation of Portlandite, noted as “Ldx”.
- between 600 and 800 °C: decarbonation of CaCO₃ noted as “Ldc”.

Furthermore, the temperature ranges of each phase vary slightly from author to author with low differences depending on the type of machine used.

3 Experimental Results

3.1 Evolution of Ca(OH)₂ Content from TGA Measurements

In general, the presence of the Portlandite in the slag-blended materials depends on two aspects: the hydration of the clinker which produces Ca(OH)₂ and the hydration of the slag which consumes the Ca(OH)₂ present. Thus, Ca(OH)₂ content can be used to determine the degree of hydration of cement in the OPC pastes. However, this principle cannot be applied in this study as the BFS is used. As shown in Fig. 1, the percentage by weight of the Portlandite for the OPC increases from 17.2% to 20.48% and then to 23.9% at 7, 28, and 360 days respectively. According to [26, 27], a fully hydrated OPC, produces about 20 to 25 wt.% Ca(OH)₂. Moreover, the increase in the BFS content leads to a general decrease in the produced Portlandite, which can be explained by the dilution effect of the slag. Figure 1 shows a decrease of the Ca(OH)₂ from 17.2% to 8.4% and 4.034% at 7 days when increasing the BFS from 0 to 50 and 80% respectively. At 28 days of hydration, those percentages increased from 8.4% to 10.7% for P50 and from 4.034% to 5% for P80, due to the reaction of the C₃S and C₂S of the cement with water in those formulations. After 360 days, the percentage of Ca(OH)₂ decreased from 10.7 to 9.9% for P50 and from 5% to 4.2% for P80. This decrease indicates that the consumption of Ca(OH)₂ by the slag is much higher than its production by the hydration of the clinker. In general, the hydration of slag blended cement takes a long time since it relies on the release of Ca(OH)₂ from the hydration of the OPC to produce new hydrates. These new hydrates cause pore refinement, with big pores becoming finer [28]. Ling and Poon [36] linked this reduction to the extra CSH created by the slag hydration process, which increased pore volume efficiency and decreased pore size. In this approach, the presence of BFS in the matrix reduces its porosity and, as a result, the tortuosity of the material. This reduction should result in a decrease in permeability and ion diffusion. The reduction in permeability, porosity, and water absorption might be regarded as the primary benefits of using BFS, with this reduction resulting in a high service and durable matrix.

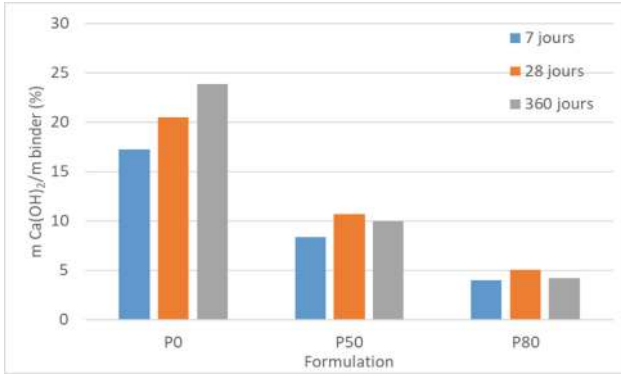


Fig. 1. Evolution of the Portlandite in the sample.

3.2 Analysis of the Hydration Evolution

In this section, the hydration degree results estimated from TGA measurements are discussed. As mentioned before, the hydration degree of a composition is the average hydration degree of all its binder components. In this study, the two components (OPC and BFS) hydrate with distinct kinetics. In general, the hydration kinetic of BFS is much lower than that of clinker. Figure 2 shows the evolution of the hydration degree with time for the three cement paste formulations considered in the present study. The 7 days hydration degree is 0.67, 0.47, and 0.42 for P0, P50, and P80 respectively. Those values can be explained by the dilution effect of the cement by slag at an early age as confirmed in Fig. 1.

At 28 days, hydration degree values of 0.84, 0.53, and 0.38 are now reached for P0, P50, and P80 respectively. Similarly, to the results obtained at 7 days, the higher the replacement of clinker by slag, the lower the hydration degree value at 28 days. This is probably due to the latent-hydraulic properties of slag.

While an increase of hydration degree values is observed at 56 days (0.86, 0.63, and 0.47 for P0, P50, and P80 respectively), it is worth noting that the kinetic of hydration of P0 becomes slower than those of P50 and P80, highlighting that the hydration of slag is more important. Those results are consistent with Fig. 1.

At 180 days, the hydration degrees of the slag-blended cement pastes significantly increase compared to that of the P0 formulation. The hydration degrees became 0.89, 0.68, and 0.51 for P0, P50, and P80 respectively.

At 360 days, the hydration degree of P0 formulation stabilizes while increases in the hydration of slag-blended cement were observed. After 360 days of wet curing, the hydration degree for P0, P50, and P80 were 0.91, 0.73, and 0.65 respectively. Those results are in agreement with those presented in Fig. 1, which can confirm that the hydration of the cement is almost stabilized and the hydration of the slag is still evolving.

Those experimental results are compared with those estimated by the VCCTL, and as seen, the VCCTL perfectly fits the hydration degree for the OPC paste, but it underestimates the hydration degree when high percentages of slag are used by keeping the same order of decrease while increasing the slag content.

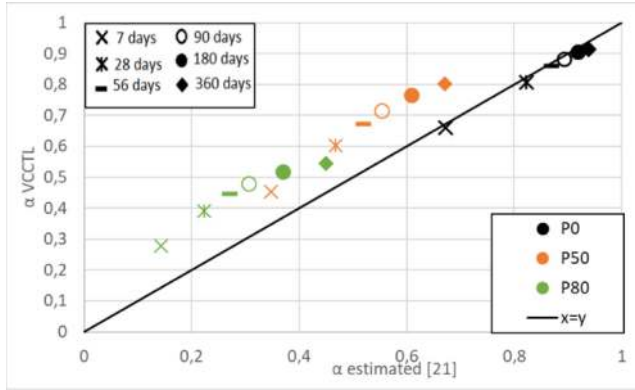


Fig. 2. Evolution of experimental and VCCTL hydration degree.

In this context, the following section will focus on proposing a kinetic hydration model, which can be applied to slag blended materials.

4 Proposition of Analytical Model for the Calculation of the Hydration Degree

Based on previous studies [29–31], an exponential type function (Eq. 7), known as Avrami equation [32–34], is used to model the evolution with time (days) of the hydration degrees of clinker and slag respectively.

$$\alpha_i = \alpha_{i,\max} \times \left[1 - \exp\left(-S_i \times t^{n_i}\right) \right] \quad (7)$$

where i represents the clinker or the slag.

For the hydration of the clinker, α_{\max} represents the ultimate hydration degree that can be reached when t tends toward infinity. The parameter S_{clinker} allows taking into account the ability of the clinker particle to hydrate while the parameter n_c is the accessibility of the water to this clinker. In this study, the values of S_{clinker} , and n_c are considered constants and equal to 0.62, and 0.37 respectively no matter the amount of slag contained in the mixture, because the hydration of the clinker is considered independent of that of the BFS as for [20, 35].

The ultimate hydration degree of cement, Eq. (8) can be estimated from the following relationship proposed by Waller [29].

$$\alpha_{\text{clinker},\max} = 1 - \exp(-D \times (W/Cl)) \quad (8)$$

where D and W/Cl are a parameter related to the curing condition and the water-to-clinker ratio respectively. For air-cured materials, [29] a value of D equal to 3.3 has been proposed by Waller [29]. For water-cured specimens, a higher value of D equal to 5 is more suitable. From a calculation point of view, the use of W/Cl ratio in Eq. (8) leads to an increase of the α_c by adding slag due to the decrease in the amount of clinker. This

is consistent with the dilution effect mentioned previously and which results in more available water for the hydration of clinker particles when increasing the BFS content [31].

For the BFS, and as seen previously, the increase of the fraction of BFS decreases the availability of the $\text{Ca}(\text{OH})_2$. A high rate of BFS leads then to a decrease in the contact between the slag and the $\text{Ca}(\text{OH})_2$. For this reason, Eq. (7) takes into account the two parameters S_{slag} and n_S which are related to the presence of Portlandite in the formulation, and the accessibility of the slag to the Portlandite respectively. By fitting the experimental results calculated with Eq. (9), the coefficients S_{slag} and n_S varied as represented in Table 2. As seen in Table 2, when increasing the percentage of slag, the two terms “ $-S_{\text{slag}}$ ” and “ n_S ” decrease. Knowing that Eq. (1) can be written as follow:

$$\alpha_{\text{slag}} = \frac{\alpha - f_{\text{clinker}} \times \alpha_{\text{clinker}}}{f_{\text{slag}}} \quad (9)$$

Table 2. Values of $-S_{\text{slag}}$ and n_S for the two formulations

Sample Codes	$-S_{\text{slag}}$	n_S
P50	-0,12	0,515
P80	-0,18	0,485

This drop is related to the weak formation of the hydrates which leads to a drop in the presence of Portlandite and then to a decrease in the accessibility of the slag to this Portlandite as shown in Fig. 1. In this study, the decrease of those two parameters is considered linear following the two Eqs. (10), and (11).

$$S_{\text{slag}} = 0.2 \times f_{\text{slag}} + 0.02 \quad (10)$$

$$n_S = -0.1 \times f_{\text{slag}} + 0.565 \quad (11)$$

In parallel, the ultimate hydration degree of slag is fitted for the two studied percentages of BFS and it is assumed to vary inversely proportional and linearly with the presence of slag. The variation can be attributed to the decrease of $\text{Ca}(\text{OH})_2$ responsible for the activation of the slag. The relation can be written as in Eq. (12):

$$\alpha_{\text{slag,max}} = -0.7579 \times f_{\text{slag}} + 1.0542 \quad (12)$$

Then, α_{slag} can be written as Eq. (13):

$$\alpha_{\text{slag}} = [-0.7579 \times f_{\text{slag}} + 1.0542] \times [1 - \exp(-(0.2 \times f_{\text{slag}} + 0.02) \times t^{-0.1 \times f_{\text{slag}} + 0.565})] \quad (13)$$

In this order, the calculation of the total hydration degree of the material is given in Eq. (14):

$$\alpha = f_{clinker} \times [(1 - \exp(-5 \times (W/C))) \times (1 - \exp(-0.62 \times t^{0.37}))] + f_{slag} \times [(-0.7579 \times f_{slag} + 1.0542) \times (1 - \exp(-(0.2 \times f_{slag} + 0.02) \times t^{-0.1 \times f_{slag} + 0.565}))] \quad (14)$$

The total hydration degree is then calculated and presented in Fig. 3; the validation of this model is done by comparing the results to the experimental ones. The coefficient of determination is equal to 0.86 for the proposed equations for the studied formulations.

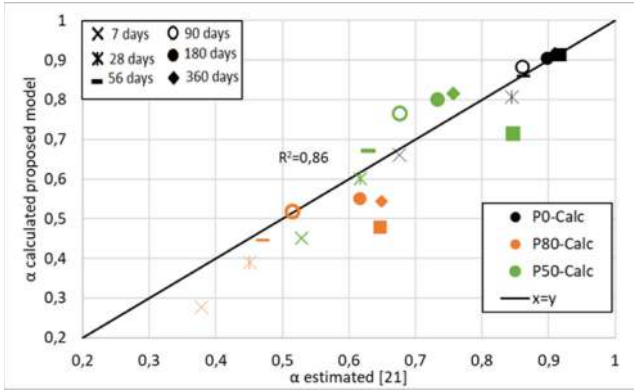


Fig. 3. Model validation with experimental results.

Finally, as shown in Fig. 3, this model is validated with experimental data obtained from TGA results until one year of curing. This duration is already higher than a lot of studies presented in the literature. In addition, this model can be applied to many formulations containing different and high rates of BFS by taking into consideration the formulation parameters only, and without introducing experimental results which are time-consuming. Finally, this model can estimate the hydration kinetics of all the components of a formulation separately “ α_c ”, and “ α_s ” in addition to the average hydration degree “ α ”.

5 Conclusion

This study proposed an analytical model of hydration kinetics of slag-blended cement based on physicochemical mechanisms involved during the hydration process: the accessibility of the water to clinker, the presence of the Portlandite, and finally the accessibility of the Portlandite to the slag. The input data of the model are the water-to-clinker ratio, the percentage of slag used, and the curing method. The TGA results also showed that:

- The percentage of CH present in the sample increase during the hydration for an OPC. However, it increases for a BFS-blended cement due to the hydration of the cement, and then it decreases to the BFS hydration.

- A compatibility is observed between the hydration degree calculated using the VCCTL and the experimentally estimated one for the OPC. The results are less convergent when increasing the BFS content.
- The results of the proposed model have been validated through a comparison with experimental data obtained from TGA results.

Several advantages can be associated with this proposed model:

- It can be applied whatever the fraction of BFS considered.
- The validity is done over one year of curing.
- The calculation of the hydration degree does not require any specific experimental results.

Finally, as already discussed, the comprehension of hydration properties is essential to clearly understand the durability and the mechanical behavior. In this regard, an experimental program is being investigated in order to complete and to confirm the influence of BFS on both mechanical and durability properties.

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