



# The degree of hydration assessment of blended cement pastes by differential thermal and thermogravimetric analysis. Morphological evolution of the solid phases



S.M. Monteagudo<sup>a</sup>, A. Moragues<sup>a</sup>, J.C. Gálvez<sup>a,\*</sup>, M.J. Casati<sup>b</sup>, E. Reyes<sup>a</sup>

<sup>a</sup>Departamento de Ingeniería Civil: Construcción, Escuela Técnica Superior de Ingenieros de Caminos, Canales y Puertos, Universidad Politécnica de Madrid, Madrid 28040, Spain

<sup>b</sup>Departamento de Vehículos Aeroespaciales, Escuela de Ingeniería Aeronáutica, Universidad Politécnica de Madrid, Spain

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## ABSTRACT

The degree of hydration assessment of cement paste from differential thermal and thermogravimetric analysis data has been performed by several authors that have offered a number of proposals for technical application to blended cements. In this paper, two calculation methods are studied in detail. Then, a proposal of the degree of hydration calculation for blended cements, based on the analysis of experimental results of DTA–TG, is presented. The proposed method combines the contributions of the authors and allows straightforward calculation of the degree of hydration from the experimental results. Validation of the methodology was performed by macroscopic and microstructural tests through paste and mortar specimens with blast furnace slag, flying ash and silica fume mineral admixtures being used. Tests of scanning electron microscopy with an energy dispersive analyser on paste specimens, and of mechanical strength on mortar specimens with the same percentages of substitution, were performed. They showed good agreement with the information derived from the differential thermal and thermogravimetric analysis data.

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## 1. Introduction

At present, some of the most commonly used mineral admixtures are blast furnace slag (BFS), flying ash (FA) [1–3] and silica fume (SF) [1]. Among the advantages derived from the use of mineral admixtures are a reduction in the amount of CO<sub>2</sub> generated during the manufacturing of cement [4], recycling of by-products from other industries and the resulting economic benefits. As for the final product, improvement of the engineering properties of the cement-based materials, such as mechanical resistance and durability, reinforces its recommendation.

Pozzolan materials or slag can raise resistance to sulphate attack, hinder chloride access and improve mechanical strength [10].

Complex processes of dissolution, transport and chemical reaction of the dissolved ions form part of the hydration mechanisms of ordinary Portland cement (OPC) [5–7]. Regarding processes that take place in the presence of mineral admixtures, as complexity increases use of additional techniques is required in understanding their development in a better way. Short-term heat exchange in the early samples plays an important role in the hardening and performance of the material as shown by calorimetry studies, which are commonly used to assess the first 45 h of hydration [8–12]. The evolution of the hydration heat curves depends on both the cement and the addition type. In blended cements, the mineralogical composition of the Portland cements has a significant impact in the process [10]. Those cements with a high C<sub>3</sub>A and low C<sub>3</sub>S content produce earlier and more intense hydration reactions, together with shorter setting times. Conversely, those Portland cements with a low C<sub>3</sub>A and a high C<sub>3</sub>S content have delayed and less intense reactions, as well as longer setting times [10]. Regarding the additions, there are discrepancies in the literature about whether they stimulate or reduce the heat of hydration during the first hours [10]. Several

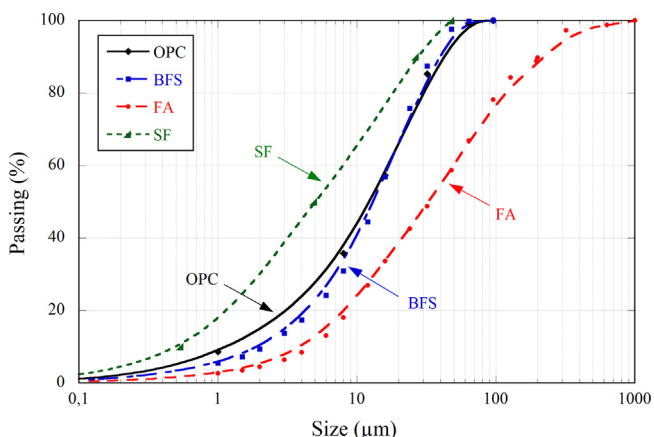
*Abbreviations:* OPC, ordinary Portland cement; BFS, blast furnace slag; FA, flying ash; SF, silica fume;  $\alpha$ , hydration degree; DTA–TG, differential thermal and thermogravimetric analysis; SEM–EDX, scanning electron microscopy with an energy dispersive X-ray analyser; CH, calcium hydroxide; w/b, water to binder ratio; Ldh, dehydration; Ldx, dehydroxylation; Ldc, decarbonation; Ldc<sub>a</sub>, anhydrous materials carbonation; XRD, X-ray diffraction analysis.

\* Corresponding author.

*E-mail addresses:* [sm.monteagudo@alumnos.upm.es](mailto:sm.monteagudo@alumnos.upm.es) (S.M. Monteagudo), [amoragues@caminos.upm.es](mailto:amoragues@caminos.upm.es) (A. Moragues), [jaimc.galvez@upm.es](mailto:jaimc.galvez@upm.es), [jaimc.galvez@caminos.upm.es](mailto:jaimc.galvez@caminos.upm.es) (J.C. Gálvez), [mariajesus.casati@upm.es](mailto:mariajesus.casati@upm.es) (M.J. Casati), [encarnacion.reyes@upm.es](mailto:encarnacion.reyes@upm.es) (E. Reyes).

**Table 1**  
Chemical composition of OPC and mineral admixtures (%).

	OPC	BFS	FA	SF
SiO <sub>2</sub>	19.20	37.00	55.1	95.02
Al <sub>2</sub> O <sub>3</sub>	6.07	11.84	30.00	0.76
Fe <sub>2</sub> O <sub>3</sub>	1.70	0.60	5.80	0.19
CaO	63.41	43.5	3.00	0.13
MgO	2.56	6.35	1.60	0.06
SO <sub>3</sub>	3.38		0.30	0.12
K <sub>2</sub> O	0.82	0.45		0.65
Na <sub>2</sub> O	0.33	0.18		0.13
Cl <sup>-</sup>		0.01	0.002	
TiO <sub>2</sub>		0.43		
Lost ign. (%)	2.09		1.42	2.04
Specific surface (m <sup>2</sup> /g)	1.0199	1.4358	0.7991	16.8489



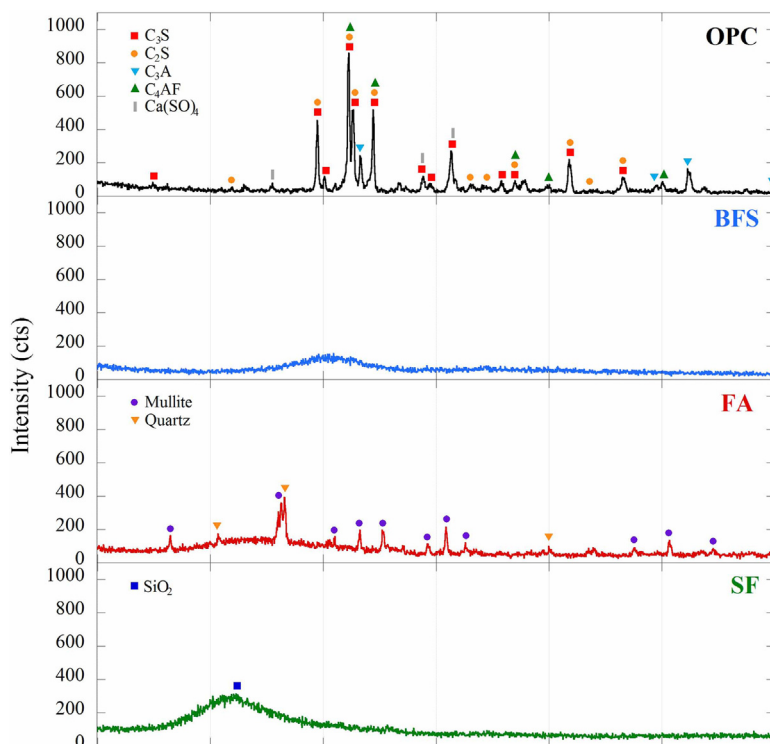
**Fig. 1.** Particle size distribution of the anhydrous materials.

authors have reported that the use of FA delays the hydration reactions and increases the ultimate  $\alpha$  [11,12]. Similar results may be found by the use of SF [9].

The two main effects that modify the hydration kinetics of cement by the use of additions are the dilution effect and heterogeneous nucleation [8]. The dilution effect comprises an increase in the w/b ratio, as a consequence of the replacement rate and subsequent decrease in the amount of cement. The presence of less cement implies fewer hydrated products. Heterogeneous nucleation is related to the new particles that enlarge the specific surface and favour the chemical reactions. Some other aspects, like the particle size distribution and mineralogical composition of the additions also have a significant influence in the general hydration.

In explaining pozzolanic activity, some studies have claimed that the rapid dissolution of silica leads to an immediate consumption of calcium [8,12]. As a consequence, the lack of availability of CH reduces the speed of the addition hydration since the reaction depends on the CH concentration [9]. Other studies have shown that readily available SO<sub>3</sub> seems to be another factor responsible for delaying the aluminate reaction [12]. Furthermore, other theories support the view that the transformation of the primary SCH in a more stable SCH proceeds at a slower rate [12].

Although at first sight it would seem inappropriate to account for the whole process by means of a single degree of hydration ( $\alpha$ ), practice has shown that the general parameter provides a reliable description of the kinetics. During the induction period, heat exchange with the medium decreases which means that evaluation of  $\alpha$  from the calorimetric test becomes more complex [13,14]. The reactions that occur during this period are responsible for much of the reduction of the porosity and increase of the strength. Despite this, there has been little study of this period [9]. For advanced curing times, the use of differential thermal and thermogravimetric analysis (DTA–TG) is proposed as one of the most effective tests for the evaluation of hydrated compounds and hydration degree [4,5,15]. The above test, initiated by Le Chatelier and later improved by Robert-Austen [16] has been used by several



**Fig. 2.** XRD analysis of the anhydrous materials.

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