



# Monitoring and modelling the early age and hardening behaviour of eco-concrete through continuous non-destructive measurements: Part I. Hydration and apparent activation energy



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## ABSTRACT

A recent scientific and industrial interest has been brought to combine multiple supplementary cementitious materials (SCM) in large quantities in concrete binders. The growing use of binary, ternary or quaternary cement binders calls for the development of simple, efficient and accurate experimental means to characterise and predict the behaviour of such concretes. Continuous non-destructive testing constitutes a major opportunity since it generally consists in simple test setup requiring low human interaction. In this study, isothermal calorimetry and ultrasonic pulse velocity (compression and shear waves) are performed on various concrete compositions with massive incorporation of limestone filler and blast-furnace slag. Indeed, these two additions present interesting complementary effects and positive synergies in terms of fresh properties, mechanical behaviour and durability potential. The hydration process of these concretes is investigated, and a new model is presented for representing the hydration degree of SCM-based binders. Then, the apparent activation energy is studied through tests at various temperature. Ultrasonic measurements are shown to provide a good alternative to isothermal calorimetry for the monitoring of the apparent activation energy of concrete during the first days of hydration.

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## 1. Introduction

The recent awareness of the cement industry with current environmental imperatives has motivated much research on the possibilities of substitution of clinker, responsible for substantial emissions of CO<sub>2</sub> by mineral additives, by-products of various industries. Thus the use of blast furnace slag (BFS) or limestone micro-filler (LMF) in large quantity has gradually become common, especially as these additions provide technical advantages in terms of strength, workability, or durability as well as economic benefits [1,2]. Recently, a growing scientific and industrial interest has been brought to combine multiple supplementary cementitious materials (SCM) in concrete binders. The term eco-concrete has thus emerged, and currently has no standardized definition. From an overall perspective, eco-concrete refers to a concrete mix which was designed taking into consideration sustainable development issues such as CO<sub>2</sub> emissions due to clinker production, possibility

to use recycled materials or aggregates, reduced use of water etc. In this work, this term refers to concrete which clinker has been substituted by massive amount of SCM. However, the clinker substitution rate by itself is not sufficient to define eco-concrete, as its behaviour has to be at least equivalent to traditional concrete from a performance point of view.

In this framework, amongst the various possibilities as regards the combination of two or more SCM in concrete, LMF and BFS present interesting complementary effects and positive synergies in terms of fresh properties, mechanical behaviour and durability potential [3–9]. This combination therefore fully complies with the criteria for developing eco-concrete, since it generates concrete with low amount of cement, which is replaced by by-products of the industry, what results in concrete equivalent or better than most traditionally used concrete.

Continuous non-destructive testing (CNDT) constitutes a major opportunity since it generally consists in simple test setup requiring low human interaction during the test. Among these methods, isothermal calorimetry (IC) and ultrasonic pulse velocity (UPV) are efficient tools that have been widely used for the determination of concrete properties. This paper is the first part of a

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study aiming at applying these two methods to the measurement of hydration, apparent activation energy and mechanical properties of various types of eco-concrete. This first part focuses on the measurement of the hydration degree and apparent activation energy of concrete.

From heat release measurements, a degree of hydration can be computed [10,11]. The determination of the degree of hydration is of primary importance for concrete behaviour modelling purposes, as well as for the understanding of the chemical behaviour of cement containing mineral additions. Therefore, in this study, its determination is made from heat flow measurements, and the effect of massive clinker substitution by individual and combined limestone filler and blast furnace slag is investigated. Then, a new model is proposed for the prediction of the hydration degree, which can be applied for any of the tested cement-based materials.

In addition, one of the foremost phenomena altering the hydration kinetics is temperature, through the apparent activation energy ( $E_a$ ), which is known to be highly affected by the nature and amount of SCM. CNDT testing at various temperatures can yield the apparent activation energy of the tested binder, as well as its evolution through hydration [11–13]. The few studies existing on this topic do not allow detailed observations of this evolution. In addition, these studies are only based on Portland cement concrete, and do not assess the issue of mineral additions, which are nowadays already commonly used. The apparent activation energy of four concrete compositions are investigated through classical measurements and various CNDT methods. A new method for the determination of the apparent activation energy at the concrete scale is developed, based on the ultrasonic method.

This work aims at

- developing a new model for the hydration of concrete with massive amount of SCM, – developing a new ultrasonic method for the determination of  $E_a$  and its evolution through hydration
- evaluating the individual and combined effect of limestone filler and blast-furnace slag on hydration and  $E_a$ .

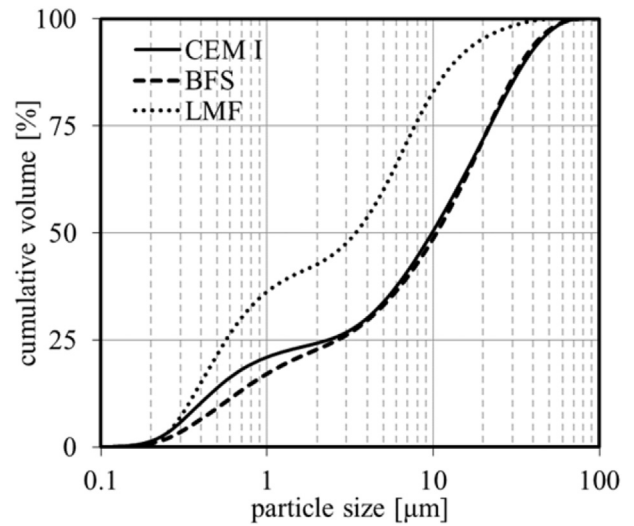
## 2. Materials and method

### 2.1. Materials

The materials used for this study are a CEM I 52.5 N, limestone micro-filler, blast-furnace slag and gypsum. Their chemical compositions are detailed in Table 1, and their particle size distribution is shown in Fig. 1.

**Table 1**  
Chemical composition and physical properties of materials.

		CEM I	BFS	LMF	Gypsum
C <sub>2</sub> S	[%]	12.6	–	–	–
C <sub>3</sub> S	[%]	63.49	–	–	–
C <sub>3</sub> A	[%]	8.09	–	–	–
C <sub>4</sub> AF	[%]	9.8	–	–	–
SiO <sub>2</sub>	[%]	20.12	33.3	–	–
Al <sub>2</sub> O <sub>3</sub>	[%]	5.03	12.5	–	–
CaO	[%]	64.53	41.5	–	–
MgO	[%]	0.98	7	–	–
CaCO <sub>3</sub>	[%]	–	–	98	–
CaSO <sub>4</sub>	[%]	4.5	–	–	79
SO <sub>3</sub>	[%]	3.36	0.16	0.00	46.5
Blaine	[cm <sup>2</sup> /g]	365	450	647	354
Density	[kg/m <sup>3</sup> ]	3.15	2.89	2.71	2.31



**Fig. 1.** Particle size distribution of the cement and binder components.

### 2.2. Mix designs

Four concrete compositions are studied (Table 2). Two compositions containing high amounts of limestone filler (C3) and blast furnace slag (C2) are investigated, as well as a reference mix which binder containing only Portland cement (C1). Then, a fourth concrete is tested for the combined effect of both additions (C4). The binders therefore include variable contents of cement, LMF, BFS and gypsum. Amongst all these compositions, it is ensured that the granular skeleton, water/binder ratio (w/b), paste volume and

**Table 2**  
Composition and properties of concrete mixtures (kg/m<sup>3</sup>).

	C1	C2	C3	C4
Aggregate 10/14	873	873	873	873
Aggregate 6/10	210	210	210	210
Sand 0/4	853	853	853	853
Cem I 52.5	432	104	285	103
BFS	0	291	0	164
LMF	0	0	126	124
Gypsum	0	22	10	22
Water	173	167	169	165
Density [kg/m <sup>3</sup> ]	2540	2519	2525	2514
w/b	0.4	0.4	0.4	0.4

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