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Characterization of alkali-activated binders using the maturity method

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HIGHLIGHTS

- We study the applicability of the maturity method to alkali-activated binders.
- We establish the strength-maturity relationship through laboratorial tests.
- Different alkali-activated mortars were produced varying the type and concentration of the alkaline activator.
- The maturity method was applied to the production of precast alkali activated concrete façade panel.

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ABSTRACT

Presently, the development of innovative eco-efficient cementitious materials is a widespread concern to assure the sustainability of the built environment. In this scope, alkali-activated binders, in which ordinary Portland cement (OPC) is totally or at least partially replaced by industrial by-products, is a promising solution. The alkaline activation of aluminosilicate materials, such as fly ash, consists in adding an alkaline solution to the latter and then submitting it to curing at prescribed temperature, thus promoting the transition from fresh to hardened state. Given the fact that the needed curing temperature is considerably high, presently these new bindings are mainly interesting for the precast industry. Moreover, due to the significant influence of this parameter, the maturity method, if applicable, can represent a most valuable tool, since it allows monitoring and forecast the compressive strength evolution as a function of time and temperature, according to Arrhenius equation.

The study herein described aimed at applying the maturity method to alkali-activated binders and at establishing the strength-maturity relationship through laboratorial tests. First, different mixtures of alkali-activated mortars and a selected alkali activated concrete mixture were produced, varying the type and concentration of the alkaline activator. The influence of OPC as addition was also studied. Afterwards, the maturity method was applied to the production of precast alkali activated concrete façade panels. Results are presented and discussed and most relevant conclusions are drawn. It was proved that the maturity method can be used for monitoring both alkali activated mortars (AAM) and alkali activated concrete (AAC).

1. Introduction

The production of cement is an energy-consuming process and, for this reason, it has a significant environmental impact. Each year, the cement industry is responsible for about 5–8% of the worldwide CO_2 emissions, released into the atmosphere [1]. In order to minimize this negative impact, it is important to find out alternatives to the conventional cement. In this perspective, the study of alkaline activated binders is quite relevant. The alkali activation of fly ash consists in mixing an alkaline solution with the latter, which this way, under a certain temperature, will harden originating a solid material [2,3]. This subject started being studied at the end of the 1930s by Feret [4] and Purdon [5]. More recently,

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the interest in alkali-activated binders registered a significant increase [6–9]. Previous studies [3,10–18] on alkali-activated mortars and concretes, fly-ash based, have shown that the compressive strength is influenced by several factors, such as: curing temperature, curing time, sodium silicate content and sodium hydroxide molarity.

Regarding the effect of curing conditions, it is widely accepted that both higher curing temperature and higher curing time lead to an increase of the compressive strength. Most of the research on alkali activated mortars (AAM) and on alkali activated concretes (AAC) [3,10–13,17,19] was conducted adopting curing conditions corresponding to a relative humidity of circa 95%, a temperature ranging from 30° up to 100 °C, and a curing time from 1 up to 24 h. Palomo et al. [3] state that the temperature is especially important for shorter periods of time (between 2 and 5 h). Another important aspect referred to in the literature [15,16] is that unsuitable curing conditions may favor carbonation at a very early stage, lowering pH levels and, as a result, retarding substantially the ash activation rate and the development of mechanical strength. To prevent this, the curing environment must be kept at high relative humidity. In regard to concentration and nature of the alkaline activators, it can be stated that, under similar conditions, the mechanical strength of alkali activated fly ashes rise at higher rate for higher concentrations of alkaline activator [13,20], thus playing an important role in the initial dissolution of silica and alumina present in the fly ash. In fact, raising the activator concentration leads to an increase of the system's pH, as well as of its sodium oxide (Na_2O) content, and these two factors favor the dissolution of the original ash particles and promote the precipitation of the alkaline silicoaluminate gel [3]. Based on previous studies [3,14,18], it was also concluded that the presence of soluble silicates in the alkaline activator provides a considerable improvement in the development of mechanical strength, when compared to solutions constituted exclusively of hydroxides (sodium hydroxide or potassium hydroxide).

The curing conditions, mainly temperature and time, are important parameters in the development of the mechanical strength of these binders. Therefore, it is of great interest to monitor the evolution of the compressive strength, and to express it as a function of time and temperature. The Maturity Method is a procedure that allows obtaining an estimate of the concrete strength, based on the temperature versus time record. There are two functions to calculate an indicator of maturity, the maturity index, recommended by ASTM C 1074 [21]: (i) the Nurse–Saul maturity function (Eq. (1)), which gives the time–temperature factor; and (ii) the Arrhenius equation (Eq. (2)), which gives the equivalent curing age using a reference curing temperature.

$$M(t) = \sum_{0}^{t}(T_a - T_0)\cdot\Delta t \qquad (1)$$

where M is the maturity of the concrete (°C h), Δt is time interval (h), T_a is average concrete temperature during Δt (°C) and T_0 is datum temperature, (°C).

$$t_e = \sum_{0}^{t} e^{\frac{E_a}{R}\left(\frac{1}{T_S}-\frac{1}{T_S}\right)}\Delta t \qquad (2)$$

where t_e is the equivalent age at a specified temperature (h), E_a is activation energy (J/mol), R is universal gas constant (8.3144 J/mol/K) and T_S is specified temperature (K).

To use the Maturity Method, a datum temperature (T_0) must be first determined. The ASTM C 1074 standard [21] provides a procedure for obtaining the latter and the activation energy (E_a), based on the development of mortar compressive strength at three different curing temperatures (see Section A1.1.8). Both methods require measuring and recording of concrete temperature along time, and then they differ only on the approach when analyzing the results (a linear equation (Eq. (1)) or a nonlinear one (Eq. (2)). Arrhenius equation has been found more reliable on previous works when applied to conventional concrete, however in the present paper both methods were used, to check their accuracy when alkali-activated binders are used. The results provided from both methods are presented and compared in the following sections.

Given that this method has been successfully applied to OPC-based mortar and concrete mixtures, the study herein presented was conducted aiming at checking if the same procedure can also be applicable to alkaline activated binders. With this goal, the maturity method was applied to alkali-activated binders and the corresponding strength-maturity relation was established. Within this experimental program, the most relevant parameters were assessed, namely datum temperature and activation energy. The variation of the alkaline activator type and the influence of ordinary Portland cement, as addition, were also taken as variables in this study. The influence of these parameters on the setting was quantified.

2. Experimental work

2.1. Materials and methods

A fly ash from a Portuguese steam power plant was used in this work. According to ASTM 618-C3 [22], the latter is class F, and the corresponding chemical composition, assessed using X-ray fluorescence spectrometer, is presented in Table 1.

It presents a density of 2.38 kg/dm^3, a specific surface of 2.33 m^2/g, and it consists mostly of silica (Si) and alumina (Al) oxides. The adopted fly ash has, according to Fernández-Jiménez and Palomo [10], all the characteristics needed to be used as binder by alkali activation.

The alkaline solutions (AS) were produced with sodium hydroxide pellets (98% pure) and water glass, a sodium silicate solution (Na_2O = 8.4%, SiO_2 = 27.8%, and H_2O = 63.8%) with a density of 1.37 kg/dm^3. Siliceous sand, in dry condition, with a maximum particle size of 4 mm and density of 2.63 kg/dm^3 was adopted to produce the AAM mixtures and two different aggregates, in dry condition, were used to produce the AAC mixtures: a siliceous sand 0/8 mm with 2.62 kg/m^3 bulk density and a granitic coarse aggregate 5/15 mm with 2.66 kg/m^3 bulk density. It was also used CEM I 52.5R as addition to the binder (Bi) in the AAM mixtures. For the alkali activation of fly ash, four different AS were adopted: (A) containing only sodium hydroxide solution, with 8 M and 12 M; (B) mixture of 60% of sodium hydroxide solution (12 M) and 40% of sodium silicate solution; and (C) mixture of 40% of sodium hydroxide solution (12 M) and 60% of sodium silicate solution.

First, different mixtures of AAM were prepared, considering both the type of AS and the binder as variables. The influence of replacing 10% of the binder by Portland cement was also studied. The binder dosage was kept constant (700 kg/m^3). The adopted AAM mixtures are presented in Table 2.

Then, an AAC mixture was studied with the purpose of obtaining the maturity – compressive strength relation. The importance of the latter is justified with the final objective mentioned before of applying this mixture to the production of façade panels in the precast industry. In Table 3, the constituents of the AAC produced mixture are presented. A binder dosage of 500 kg/m^3 was adopted.

When a combination of sodium silicate solution and sodium hydroxide solution was used as AS, these were first pre-mixed with at least 24 h advance, as recommended by Hardjito and Rangan [23]. Then the mixtures were produced according to Sumajouw and Rangan [24] procedure: first the solid constituents (aggregates and fly ash) are blended during 3 min and, afterwards, the AS is added to the mixture and blended for another 4 min. Although ASTM C 1074 [21] standard recommends the use of 50 mm cubic specimens, in the present work prismatic (40 × 40 × 160 mm^3) mortar specimens and cubic (100 mm) concrete specimens were produced, according to European standards, EN 196-1 [25] and EN 12390-2 [26], respectively. The specimens were cast immediately after the preparation of the mortar and the concrete. The mortar mould, according to the standard EN 196-1 [25], was firmly clamped to a jolting table. A first layer of mortar, halfway high, was introduced into each mould compartment, spread and compacted using 60 jolts. Then the second layer of mortar was added, leveled and compacted with

Table 1
Chemical composition of FA (%).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2
54.0	22.0	8.5	6.0	1.6	1.6	1.0	1.2