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Effect of fly ash and grading agent on the properties of mortar using response surface methodology

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ABSTRACT

In this study, the concept of Response Surface Methodology (RSM) was presented to optimize and compare the effect of fly ash and grinding agent on the cement compressive strength at 7, 28 and 90 days.

This method showed that the optimum paste mix design with the fly ash (FA) to binder at 0%, clinker (KK) to binder at 66.4% and debit grinding agent (GA) to binder at 306 kg/t produced a spread compressive strengths of the hardened paste at 29.0, 38.0 and 50.4 MPa after 7, 28 and 90 days of curing, respectively. The fly ash began to play a significant role for the compressive strength after 28 days of curing, whereas grinding agent was reactive after 7 days of curing, indicative of time dependent contribution of fly ash and grinding agent to the development of compressive strength. These were further supported by the SEM microstructure analysis. Such a delayed involvement of fly ash and grinding agent in the cement chemistry should be taken into consideration with care when translating laboratory research results typically based on 28 days strength to field practice where a shorter curing is typically provided for cost reasons.

1. Introduction

The manufacturing of cement accounts for ~5% of the total anthropogenic release of CO_2 to the atmosphere [31,9]. In an effort to reduce anthropogenic CO_2 emission and for the economic reasons as reducing the clinker grinding energy, the grinding agent has been commonly used to partly replace the Portland cement in concrete and to improve its properties [5]. Equally important to mention is that addition of the grinding agent in Portland cement has generally shown improvement of the workability of the fresh concrete [2], the mechanical strength and durability of the hardened concrete [15,37]. However, as commonly reported, the grinding agent increases the rate of strength development and it also reduces the appearance of cracks and minor deterioration [26,8].

Cement is a widely used construction material worldwide. The raw materials are easily available and it does not require complex or expensive equipment to create. But due to its popularity and demand as a construction material, some of its component should have an alternative source. In this context, fly ash (FA) and grinding agent have been commonly used to partly replace the Portland cement in concrete.

Fly ash is a by product waste material of thermal power plants. The manufacturers of these days use this waste as substitute to various construction materials and it is sold at a low cost. Pozzolanic reaction by fly ash (FA) with the formed $Ca(OH)_2$ produces additional C–S–H gel. Therefore, the curing period should be prolonged for FA-cement concretes due to a slower pozzolanic reaction, especially when a high volume of FA is used. For example, evidence of FA reaction, determined by $Ca(OH)_2$ consumption, was noticed after 7 days of curing and a significant increase of compressive strength in FA-cement pastes was observed after 28 days [10]. Similarly, porefilling effect and pozzolanic reaction in FA cement concrete occurred after 28 days of curing and a significant contribution of fly ash addition to the strength was noticed after 90 days of curing [7].

Due to its increasing demand, it is not easy to find the fly ash available in market, which presents a serious problem. In addition, another solution is applied, the use of grinding agent (GA) which presents a revolution in the cement industry. It is known that this grinding agent is used to facilitate the grinding of the Portland clinker and to improve the cement physicochemical properties. This GA adsorbed on the surface of the material to be comminuted, i.e. either on the exterior surfaces or on the microcrack walls where it manages to enter. An immediate effect is a decrease in hardness accompanied by phenomena of adhesion and clogging [39]. Also, this cement addition gets adsorbed on the surface of the material undergoing grinding and reduces the surface energy, which only leads to further breakage and it facilitates the grinding of the clinker (The Rebinder effect) [30–36]. In

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addition, the effect of this agent on the improvement of cement quality is presented by the formation of a dense film between the cement grains, during the cement hydration, which develops the physicochemical properties of the cement.

Due to the high importance and efficiency of these products, the aim of this research wasto assess significance of curing period for the development of compressive strength of the cement pastes containing fly ash and grinding agent as admixtures. To this end, a global optimization of the mixture was made to find the mix design possessing the minimum of cement grinding time and the maximum achievable compressive strength of the hardened pastes cured for 7, 28 and 90 days.

2. Materials and methods

2.1. Cement, fly ash and grinding agent

- Two types of cement were used: Portland limestone cement type CEM II/A-L 32.5N and Portland siliceous fly ash cement type CEM II/A-V 32.5N in compliance with ASTM C595 were used.The different types of cement were prepared according to the following experiment matrix:

1. Fly ash was obtained from a local coal-fueled power plant (Mahammedia-Marrocco). This fly ash complies with the Class C for all of its mineralogical compositions (Table 1).

The chemical composition of the two cement types and fly ash, showing major components as oxides determined by X-ray fluorescence (XRF) are shown in Table 2.

1. The grinding agent solution was purchased from the MAPEI Company. The chemical composition and the property of the MAPEI adjuvant are presented in the Table 3.

2.2. Preparation of mortars

All mortars were prepared by using distilled water and a binder/sand weight ratio of 1:3, following the procedures described in EN 196-1 [12]. Due to the different fines content of the aggregates, and in order to get suitable workability, the water/binder ratio was 0.5 for mortars made with standard silica sand. The fly ashand grinding agent were dosed as an addition to the total cement mass (w/w%). These two additives were added to the clinker before cement grinding.

Table 1
Experience matrix of the prepared cements.

N° Experience	Clinker	Fly Ash	Gypsum	Limestone	Grinding agent debit
	%	%	%	%	Kg/t
1	60	0	3	37	0
2	70	0	3	27	0
3	60	12	3	25	0
4	70	12	3	15	0
5	60	0	3	37	500
6	70	0	3	27	500
7	60	12	3	25	500
8	70	12	3	15	500
9	60	6	3	31	250
10	70	6	3	21	250
11	65	0	3	32	250
12	65	12	3	20	250
13	65	6	3	26	0
14	65	6	3	26	500
15	65	6	3	26	250

2.3. Compressive strength

Three cubical specimens (4×4×16 cm) for each mix given in the experimental work plan were produced to test compressive strength. The samples were cured under water for 7, 28and 90 days at 20 ± 2 °C temperature and then tested using a hydraulic press following the standard [12]. Each compressive strength value was obtained from the average value of three tests.

2.4. Specimen design and preparation

This study was designed in a three factors, two level (2^3) face centered, central composite design aiming to assess the main, quadratic and interaction effects of the independent variables, the percentages of clinker (KK, 60–70, X_1), Fly Ash (FA, 0–12, X_2) and debit grinding agent (GA, 0–700, X_3), on the dependent response variables, compressive strength (Y) of the hardened pastes (Table 4). Related literature and preliminary studies were used to choose these variables and the respective regions of interest [4–41].

In this study, the binder is defined as the total amount of Portland cement and fly ash. Response Surface Methodology (RSM) was utilized to optimize the mix design in order to obtain a time-dependent maximum compressive strength of cement pastes cured for 7, 28 and 90 days. A mechanical mixer was used to prepare the cement paste specimens in accordance to the ASTM C192.

2.5. Response surface methodology

Onefactoratatime (OFAT) methodology is a conventional approach for optimizing multifactor experiments. OFAT is a changeable single factor method for a specific experiment design while other factors are kept constant and this method is unable to generate appropriate output because the effects of interaction among all factors in the design are not examined truly, and so it is not capable of reaching the true optimum value [13,20]. Hence, response surface methodology (RSM) has been introduced for parameter optimization in a way that number of experiments and interaction among the parameters are reduced to minimal value [3–17].

Central composite design (CCD) has been the most commonly used design method with RSM in statistically assessing the mathematical relationship between the independent variables and the responses. For example, CCDs with RSM were employed to optimize the amount of the Portland cement and silica fume to yield an acceptable mechanical strength of ultra-high performance fiber reinforced concrete Aldahdooh et al., [1]. Understanding these effects allows manipulation of the levels of the studied factors to manufacture sustainable light weight mortars with durable properties. While there is limited research using statistical designs to produce mortars or concrete, such as: Response Surface Methodology [33] full factorial designs [34], standard orthogonal arrays [16] or mixture experimental designs [42], as far as the authors are aware no studies have used Response Surface Methodology to assess and to optimize the impact of the fly ash, grinding agent and the synergic effect of the interactions on the final properties of lightweight cement mortars.

In this study, cement paste specimens were made in triplicate in a 2^3 face centered CCD (Table 4). Face centered central composite design (CCF) is a special case of CCD where α is equal to one. This forces the axial points of CCF to locate on the surface of the cubic, instead on the sphere space as in CCD, and therefore makes CCF a three-level CCD. The second order polynomial equation (Eq. (1)) was used to fit the data of the CCF:

$$Y=\beta_0+\sum_{i=1}^{n}\beta_i x_i+\sum_{i=1}^{n}\beta_{ii}x_i^2+\sum_{i=1}^{n}\sum_{j=1}^{N}\beta_{ij}x_ix_j+\varepsilon \quad (1)$$

Where Y represents the predicted response (i.e., compressive strength), β_0 the intercept, β_i the first-order (linear) coefficient, β_{ii} the second-