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Optimization the Effect of Fly Ash and Grading Agent on the Physicochemical and Mechanical Properties of Portland Cement.

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ABSTRACT

The optimum mix design of clinker, grinding agent and fly ash in cement is one of the best ways in identifying which mixture will yield high compressive strength without compromising good behavior and significance of each variable in every compressive strength test when a certain percentage of clinker, grinding agent and fly ash is being mixed in cement. To determine the mix design that will yield the optimum compressive cement strength, response surface methodology (RSM) is explorer in this study. RSM is an optimization tool explored in the study because it interprets experimental results even in a linear response surface manner and it provides sufficient experimental interpretation as part of the conclusive result. It has modern optimization features that can be useful in most complicated experimental design. Its most important applications are in the field where variables have potential significance in predicted system behavior called response. The combination of factorial application and modern experimental design has outstanding contribution in optimizing experimental procedures in a reduced number of studies and the response is easy to interpret. RSM was used on the data obtained from laboratory experiments conducted by the researchers. The experiments conducted include the influencing factors: clinker percentage (between 60 and 70%), Fly Ash percentage (between 0 and 12%), and debit grinding agent (between 0 and 450 Kg/t) and the interaction effects of these factors in compressive strength test are analyzed in this paper through response surface methodology. The responses of each specimen have showed significant increase in attained strength with respect to the control specimens.

Keywords: fly ash, grading agent, Portland cement, optimization, compressive strength

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INTRODUCTION

The construction industry is growing rapidly [1,2]. The use of cement as a construction material is in great demand, thus requiring the industry to create a wide range for its building components. In order to meet the increasing demand of these components [3,4], it is necessary to use waste material and grinding agent with a very low dose to compensate for the lack of natural resources and to obtain alternative ways of conserving the environment [5,6].

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.

Many research and study in engineering have been developed to use locally available materials for construction due to its economic problems. Fly ash is a by product waste material from boilers of thermal power plants. Since there are a lot of studies about fly ash's applications as substitute to various construction materials, manufacturers these days rarely dispose this waste; instead it is sold at a low cost. 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 milling agent which presents a revolution in the cement industry.

In this context, this study aimed to compare and to assess significance of curing period for the development of compressive strength of the cement pastes containing fly ash and grinding agent which contains aminoalcohol [7-9]. To this end, a global optimization [10-13] 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 and 28 days.

MATERIALS AND METHODS

Cement, FA and grinding agent

The two cement types 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 was used and FA was obtained from a local coal-fueled power plant (Mahammedia-Marrocco). The chemical composition of the two cement types, showing major components as oxides determined by X-ray fluorescence (XRF) are shown in Table 1.It should be noted that the FA complies with the Class C FA for all of its mineralogical compositions.

Table 1: Variation of chemical composition for the cement types and Fly Ash used to manufacture the mortars, expressed as major oxides determined by X-ray fluorescence microscopy (XRF).

%	CEM II/A-L 32.5N	CEM II/A-V 32.5N	Fly Ash
SiO_2	17.7±0.6	17,5±0.2	52.06
Al_2O_3	3.8±0.1	3,8±0.1	22.23
Fe_2O_3	3.0±0.1	2,9±0.0	5.45
CaO	57.1±1.1	56,4±1.0	5.69
MgO	2.5±0.2	2,5±0.1	2.36
SO_3	2.3±0.2	2,3±0.3	0.41
LOI[a]	12.3±1.9	13,2±1.5	7.8
CaO_l[b]	0.7±0.1	0,6±0.1	n.a[d]
Blaine (m2/kg)	3565±2.0	3565±2.0	n.a[d]
Fineness (% wt.)[c]	3.5±0.4	3,5±0.3	14.2

[a]*loss on ignition*
[b]*Free lime*
[c]*Dry sieve percentage passing the No. 325 (80 µm)*
[d]*not applicable*

The milling agent solution was purchased from the company MAPEI. It consisted of (in % vol.): 1,1′,1′′-ntrilotripropan-2-olo (10-12.5), 1-(N,N-bis(2-hydroxylethyl)amino))propan-2-olo (7-9.99%), and water (40-45%). It had a density of 1.04 g/mL and pH=8.2.

Preparation of mortars

All mortars were prepared using distilled water and a binder/sand weight ratio of 1:3, following the procedures described in EN 196-1 [14]. 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 FA was dosed as an addition to the total cement mass (w/w%). Admixtures were added through a syringe to the clinker during the cement grinding as a the apparent volume of cement (v/v%) .

Compressive strength

Three 4 × 4 × 16 cm specimens for each mix given in the experimental work plan were produced to test compressive strength. The samples were cured under water for 7 and 28 days at 20 ± 2 °C temperature and then tested using a hydraulic press following the standard EN 196-1: 2005 [14]. Each compressive strength value was obtained from the average value of three tests.

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, 0–450 Kg/t, X_3), on the dependent response variables, compressive strength (Y) of the hardened pastes (Table 2). Related literature and preliminary studies were used to choose these variables and the respective regions of interest [15,16].

Table 2: Matrix of 2^3 face centered central composite design and the measured dependent variables.

Run	Point[a]	Mix design of independent variables[b]						Measured dependent variables[c]	
		Coded			Uncoded			Y_{7d}(MPa)	Y_{28d}(MPa)
		X_1	X_2	X_3	X_1	X_2	X_3		
1	F	-1	-1	-1	60	0	0	24.27	32.3
2	F	1	-1	-1	70	0	0	32.02	41.5
3	F	-1	1	-1	60	12	0	22.44	30.12
4	F	1	1	-1	70	12	0	30.25	39.4
5	F	-1	-1	1	60	0	500	27	35.5
6	F	1	-1	1	70	0	500	35.5	45.7
7	F	-1	1	1	60	12	500	24.86	33
8	F	1	1	1	70	12	500	30.76	40
9	A	-1	0	0	60	6	250	26.21	34.6
10	A	1	0	0	70	6	250	31.4	40.74
11	A	0	-1	0	65	0	250	28	36.7
12	A	0	1	0	65	12	250	25.7	34
13	A	0	0	-1	65	6	0	27.4	36
14	A	0	0	1	65	6	500	29.6	38.6
15	C	0	0	0	65	6	250	27.7	36.35

[a] *F: factorial point, A: axial point, C: center point.*
[b] *X_1: %kk, X_2: %FA, X_3: debit of milling agent*
[c] *Y_1: 7-, 28-, and 90-days compressive strengths (mean ± standard deviations, n= 3).*

In this study, the binder is defined as the total amount of Portland cement and FA. 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 and 28 days. A mechanical mixer was used to prepare the cement paste specimens in accordance to the ASTM C192.

Response Surface Methodology

One-factor-at-a-time (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 [17,18]. 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 [19,20].

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 [21].

In this study, cement paste specimens were made in triplicate in a 2^3 face centered CCD (Table 2). 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:

$$Y=\beta_0+\sum_{i=1}^{n}\beta_i x_i + \sum_{i=1}^{n}\beta_{ii}x_i^2 + \sum_{i=1}^{n}\sum_{j=1}^{N}\beta_{ij}x_i x_j + \varepsilon \qquad Eq.1$$

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-order (quadratic) coefficient, β_{ij} the coefficient of interaction effect, x_i and x_j the coded levels of the independent variables x_i and x_j, respectively, and ε the associated random error [22].

Mathematical and statistical interpretations of data were done with NemrodW [23]. For the spread percentage and the compressive strength after 7 and 28 days of curing, a total of 15 combinations of independent variable settings were run with one replicate at the center points.

The significance of each of the independent variables to the dependent variable and their interactions were determined by an analysis of variance (ANOVA). Factors with a p-value of 0.05 or lower were determined to be statistically significant, and therefore considered for the predictive regression model. The relationship between the independent variables and the response variables was evaluated by contour plots.

Scanning Electron Microscopy

To evaluate the microstructural differences that could be attributed to time-dependent contribution of fly ash (FA) and grinding agent (GA) to the chemistry of the cement pastes. To avoid the effect of the cement composition, four different specimens of Portland cement were prepared with combinations of FA/GA at the levels of 0%/0Kg/t or 0%/306 Kg/t, 6%/306 Kg/t and 6%/0 Kg/t, respectively.

The percentage clinker was fixed at 97% and 91% in this case. The specimens were cured for 7 and 28 days, and tested for the compressive strength. Then, the microstructure of the fractured surface from the compressive strength test was analyzed by Scanning Electron Microscope operated at 30 keV.

RESULTS AND DISCUSSION

The Y's were measured in the range of 22.44-35.5 and 30.12-45.7 MPa for the hardened pastes cured for 7 and 28 days, respectively. These results clearly showed that the Y was further developed even after 7 days of curing to gain additional Y_1 during late age of curing (i.e., 28 days) (Table 2).

Statistical models of the RSM

The ANOVA for each dependent variable is shown in Table 3. The suitability of the model was validated by checking residual plots and the lack of-fit at a significance level of 0.05. Residual plots confirmed that the residuals were independent, were normally distributed, and had equal variances.

Lack of fit for each response resulted in p-values much greater than 0.05, indicating that the models accurately fit the data. The high regression coefficients (R^2) of 98.1 and 98.0% for the 7 and 28 days compressive strengths, respectively, also described the adequacy of the model. The main, quadratic and interactive effects of independent variables (X's) on the dependent variable (Y) were also assessed at a significance level of 0.05 (Table 3). The estimated regression models after removing insignificant terms for the Y_1's are given in Eqs. 2 and 3:

$$Y_{7d} = 27 + 3.52X_1 - 1.28X_2 + 1.13X_3 \qquad Eq.2$$

$$Y_{28d} = 36.5 + 4.18X_1 - 1.52X_2 + 1.35X_3 \qquad Eq.3$$

It should be noted that the coefficient values were for the terms of uncoded independent variables. As shown in Eq. 2 and 3, the three linear terms of X_1, X_2 and X_3 factors significantly affected the Y at different ages (7 and 28 days) with the X_1 influencing the most. Thereby, the X_2 and X_3 had the same effects on Y, but the variable X_2 has a negative effect, which means that the value of Y decreases as the percentage of fly ash increase. A significant increase of the Y could be attributed to an enhanced fluidity caused by the surfactants coated on the chemical composition and the percentage of clinker and the fly ash used, also the surfactants coated on the grinding agent. An enhanced compressive strengths of cement due to the addition of fly ash was also reported previously [24,25].

In other words, the 7 and 28 days compressive strength of the paste was predicted to decrease by the replacement of Portland cement with FA, but only slightly. This could be due to the slow development of compressive strength in FA cement pastes [26,27].

The Y_{7d}, and the Y_{28d} were predicted with first order polynomial models. Thereby, the 7 and 28 days compressive strength of the paste was predicted as a function of the linear terms of clinker percentage and FA and also of the flow rate of the milling agent. Although, no interactions between the independent variables were found to be statistically relevant to any of the dependent variables (Table 3). Thereby, they were not factored into the prediction models.

Table 3: ANOVA and full regression models statistics

Term	Y: Compressive strength			
	7-days		28-days	
	p-value	coefficient	p-value	coefficient
Constant	<0.01	27.83	<0.01	36.50
X_1	<0.01	3.52	<0.01	4.18
X_2	0.004	-1.28	0.005	-1.52
X_3	0.007	1.13	0.007	1.35
X_{12}	0.11	NS	0.11	NS
X_{22}	0.09	NS	0.10	NS
X_{32}	0.25	NS	0.26	NS
X_1X_2	0.30	NS	0.30	NS
X_1X_3	0.62	NS	0.66	NS
X_2X_3	0.19	NS	0.20	NS

[a]X_1: %kk, X_2: %FA, X_3: debit of milling agent

[b]NS: The contribution of the terms was not statistically significant.

Response optimization of the cement pastes

Table 4 summarizes the optimization goals of the RSM to find the best combination of independent variable settings that could produce the greatest Y's at the different curing periods.

Table 4: Optimization criteria for each dependent variable.

Dependent variable	Measured Y's		Optimization	
	Lower	Upper	Goal	Target
Y: compressive strength (MPa)				
7 days	22.4	35.5	Maximum	35.5
28 days	30.1	45.7	Maximum	45.7

The desirability function was utilized to simultaneously optimize the responses. As shown in table 5, the optimum independent variable setting at 66.4% X_1, 0% X_2 and 306Kg/t X_3 resulted in the Y_{7d} and Y_{28d} at 29.0 and 37.9 MPa, respectively. This was attained with the global desirability value at 99.9% and with the response-specific desirability values at 100, 99.75 and 99.95% for the Y_{7d} and Y_{28d}, respectively. It should be noted that the optimization goals could be assigned at different weights and importance. In the current study, however, the target responses had an equal weight and importance.

The maximum compressive strength of the cement for the three ages can be reach at the same time, with just using a milling agent as the additive cement and which will improve that the quality of cement a remarkable way

Table 5: The desirability functions of Y's

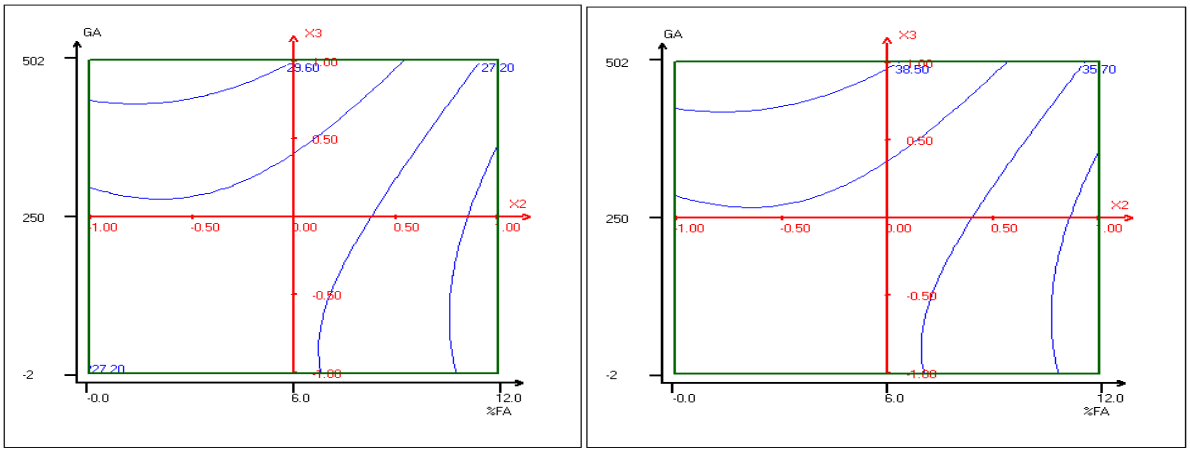
Dependent variable	Name of dependent variable	Value	desirability values %
Y_{7d}	CS at 28days	37.89	99.75
Y_{28d}	CS at 7days	28.98	100.00
	Global Desirability		99.90

**CS : Compressive strength*

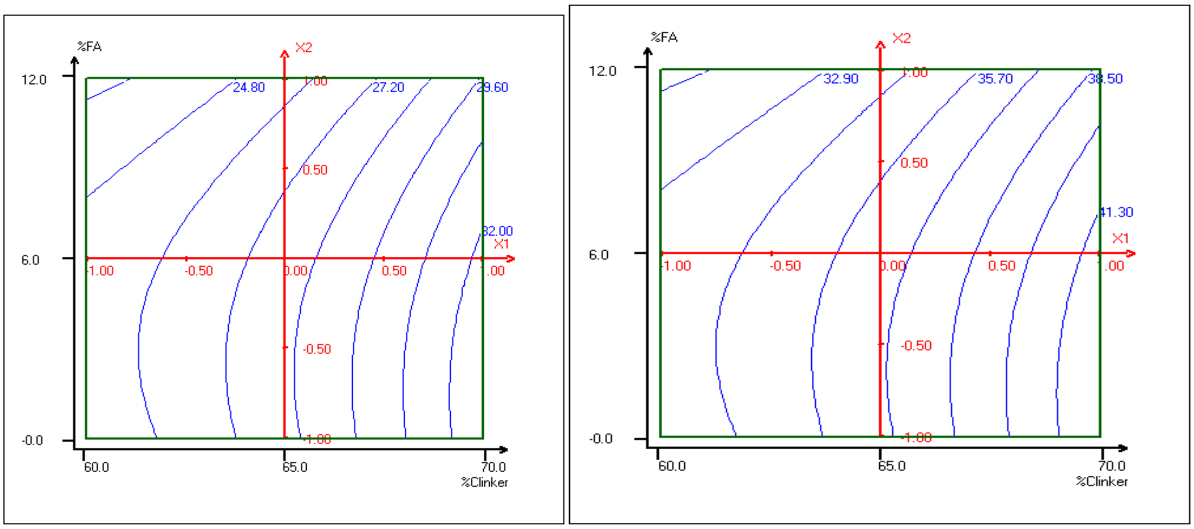
Contour plots for RSM at the optimum settings

Contour plots of the dependent responses were drawn in a function of two independent variables while the third independent variable was held at its optimal value. As shown in Fig. 1, the Y_1 increased with the decrease of X_2 and increase of X_3 at the same time, while the X_1 was held at the optimum level of 65%. So, the values of Y decrease as the percentage of FA exceed the 6% despite the increased throughput of the grinding agent. This shows that the FA has an effect on the pozzolanic cement, which leads to improving the compressive strength of the cement, but when the percentage of AF exceeds 6%, this product has a negative effect on the cement curing.

In a function of X_3 and X_2, while $X_1$was at 65%



In a function of X_2 and X_1, while X_3 was at 250Kg/t



In a function of X_3 and X_1, while X_2 was at 6%

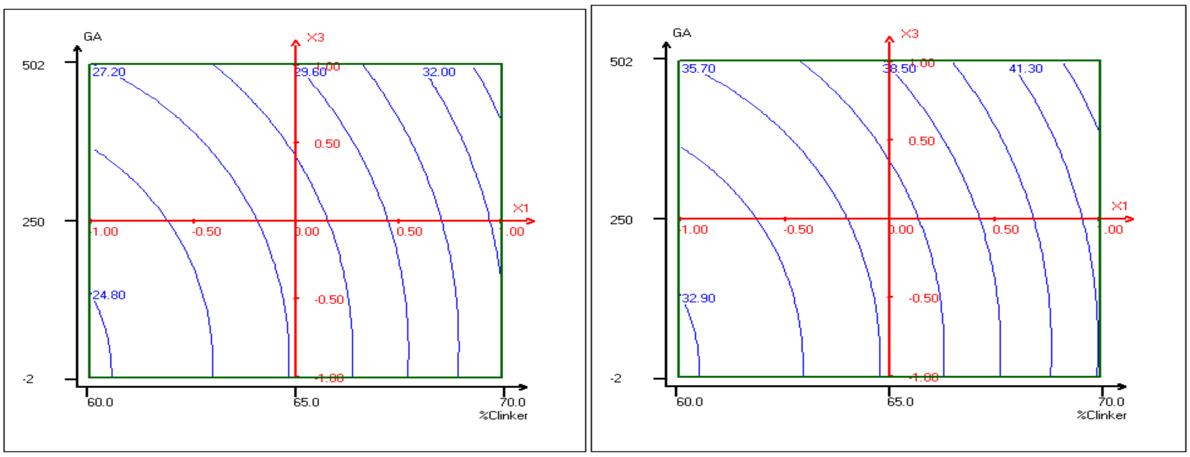


Figure 1: Contour plots of the Y_{7d} and Y_{28d} in an interactive effect of two independent variables while the third one was held at its optimum level.

When the X_2 was held at the optimum level of 6%, the simultaneous increase of X_1 and X_3 enhanced the Y. With the X_3 held at the optimum level of 250Kg/t, an enhanced Y_1 was found with the increase of X_2 and the increase of X_1 at the same time.

Overall, a greater Y was found with a greater X_1 or X_3 but with a lesser X_2. The increase of X_3 would facilitate fluidity of the cement pastes attributed to the polymer (amino alcohol) coated on the grinding agent. This was in agreement with Ouyang et al. [24] who documented an increased fluidity of cement with the addition of grinding agents. As shown in Table 1, the FA had higher Fineness than the Portland cement used in the study. Therefore, more volume of coarse FA was required for making the same weight to the Portland cement, resulting in a reduced Y. This effect was counteracted with an increase of the X_1 and X_3 in the mixture. The extent of contribution that independent variables had on the development of compressive strength was time-dependent.

Increasing Y with the increase of X_3 implies that the addition of grinding agent was effective in supporting the chemistry in cement paste at later curing stages and probably the hydration reactions and hydrate formation were favored and improved. This resulted in higher strength development due to densification of the cement pastes [28-32].

Model validation

The accuracy of the prediction model was validated by performing another set of experiment where the specimens were made in triplicate at the global optimum mix ratio obtained in Section 3.2 and by comparing the predicted and measured responses. As shown in Table 6, the lowest absolute relative percent error (PE) of the Y's, was found 5.1% for the Y_{28d} and 8.6% for the Y_{7d}. Therefore, the models generally predicted the dependent variables of Y_{28d} with good accuracy.

Table 6: Validation results of the optimum independent variable settings for the dependent variables.

Optimum mix (wt.%)			Y: compressive strength (MPa)[a]					
X_1: KK	X_2: FA	X_3: GA	7 days			28 days		
			Pred.	Meas.	PE[b]	Pred.	Meas.	PE
66.4%	0%	306Kg/t	31.2	28.8 (±0.7)[c]	8.6	40.3	38.4 (±1.1)	5.1

[a] Data are the average of triplicate samples.

[b] $Absolute\ relative\ percent\ error = \left|1 - \frac{Value_{predicted}}{Value_{measured}}\right| \times 100\%$

[c]Standard deviations (n= 3).

Fly ash and milling agent effect on cement grinding time

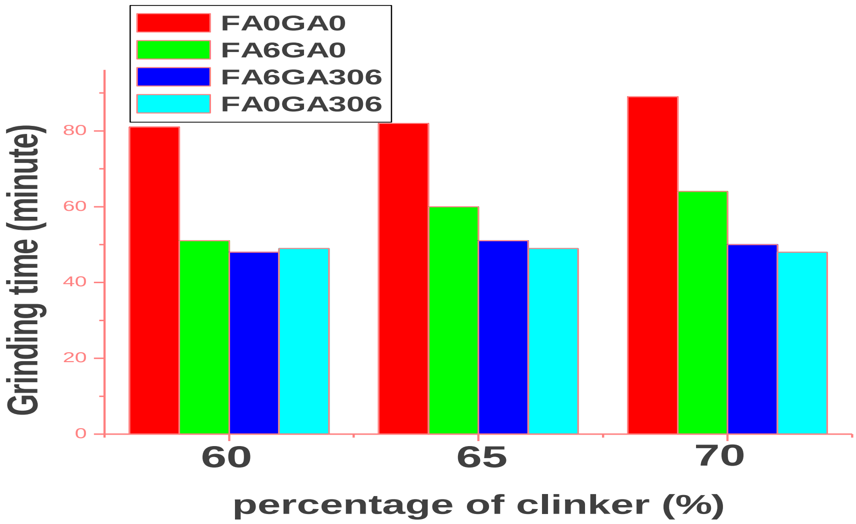


Figure 2: Grinding time of the cement pastes prepared.

According to this study, we note that:

- The milling time increases with the increase of the clinker percentage (case of Grading Agent = 0kg / t and Fly Ash = 0%), which indicate that the grinding time depends strongly to the clinker percentage.
- The presence of fly ash can reduce the grinding time
- The effect of the milling agent was better than FA and this GA keeps the grinding time of the cement fixed whatever the clinker percentage. The efficiency of GA was due to the improvement of grinding through the milling agents which is a result of mechanical-chemical effects that affect the fragmentation of grains (Rehbinder effect) and to change in the rheological properties and agglomeration of the powder cement. [7].

Microstructural analysis

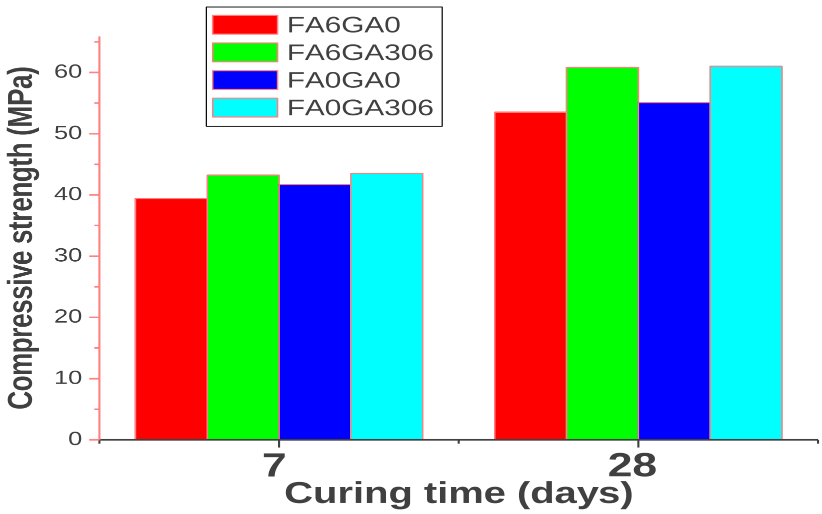
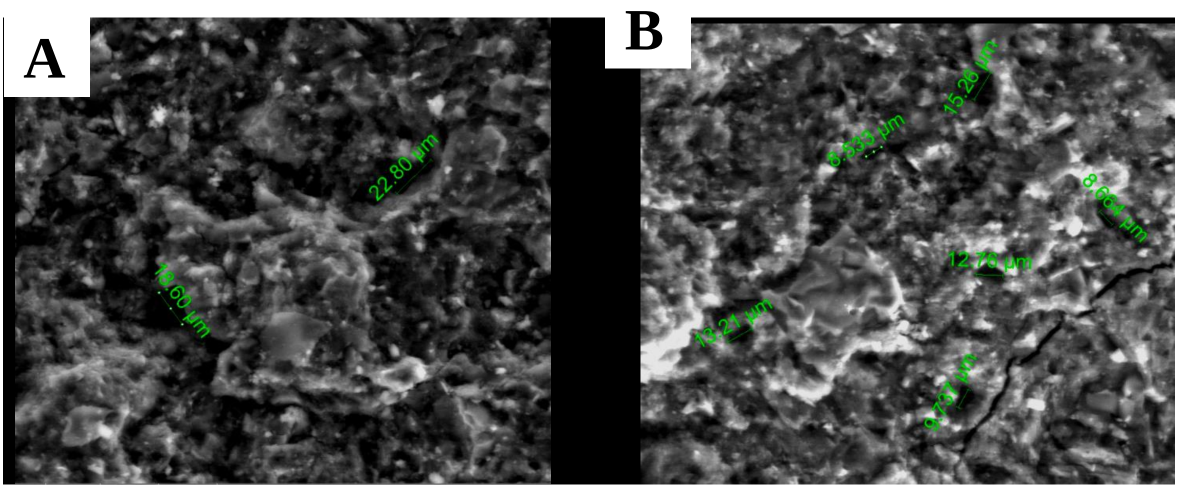


Figure 3: Compressive strength of the cement pastes prepared for the SEM analysis. Data are means with the standard deviations (n = 3).

As shown in Fig. 3, at young ages (7 days) all the pastes had approximately the same compressive strength value, but the deference between different pasta begins to appear at the 28 days. The compressive strength of the paste containing the milling agent (FA6GA306 and FA0GA306) had 12% higher Y_{28d} than those with FA (i.e, FA6GA0). These were in good agreement with the results discussed in Sections 3.1 and 3.3 that X_1 and X_3 have played an important and significant role in the development of Y at the late age of curing. The above findings were qualitatively supported by the SEM microstructural analysis (Fig. 4).

FA0GA0 at 28 days **FA0GA0 at 90 days**



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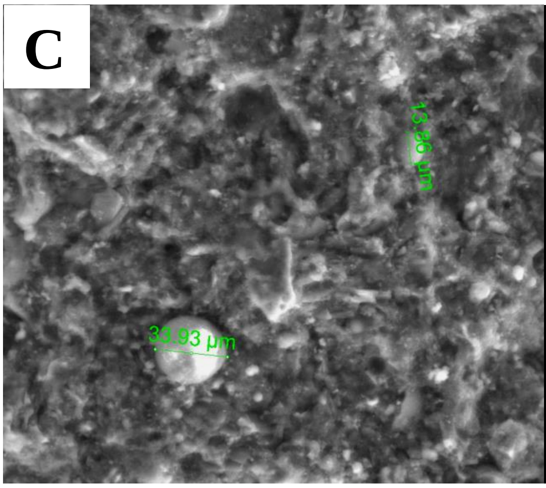
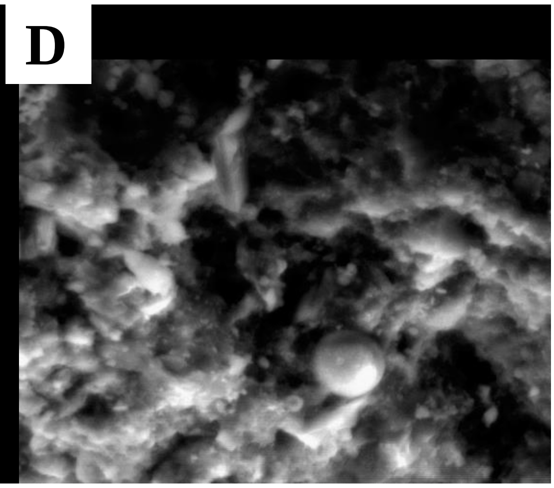
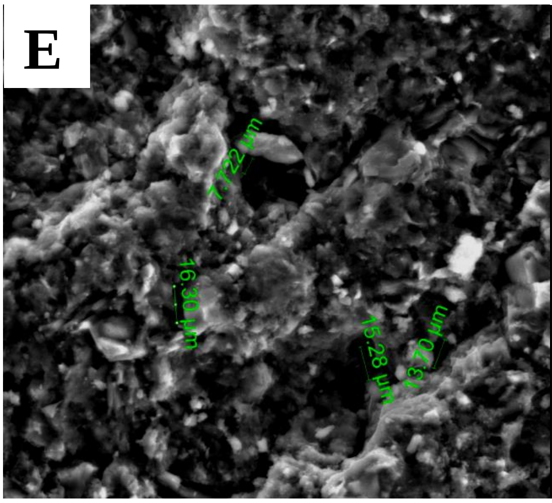
FA6GA306 at 28 days



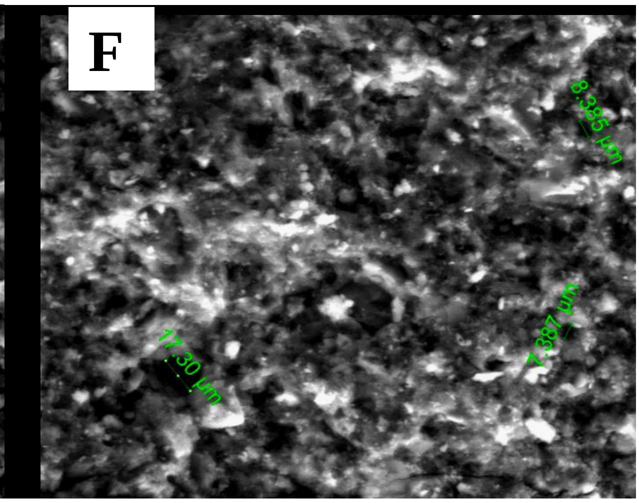
FA0GA0 at 90 days



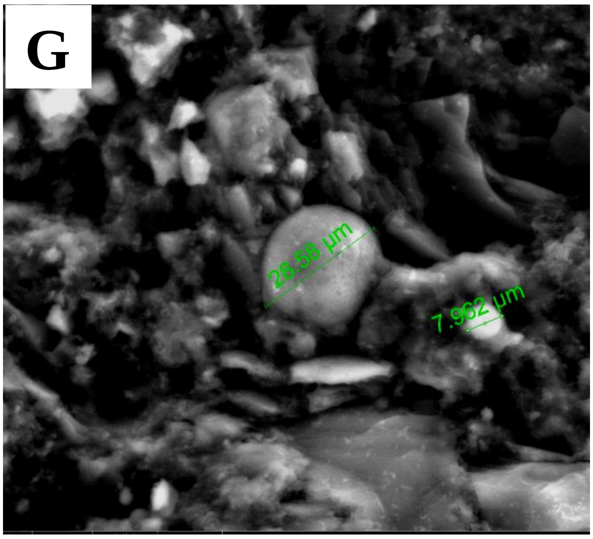
FA0GA306 at 28 days



FA0GA306 at 90 days



FA6GA0 at 28 days



FA6GA0 at 90 days

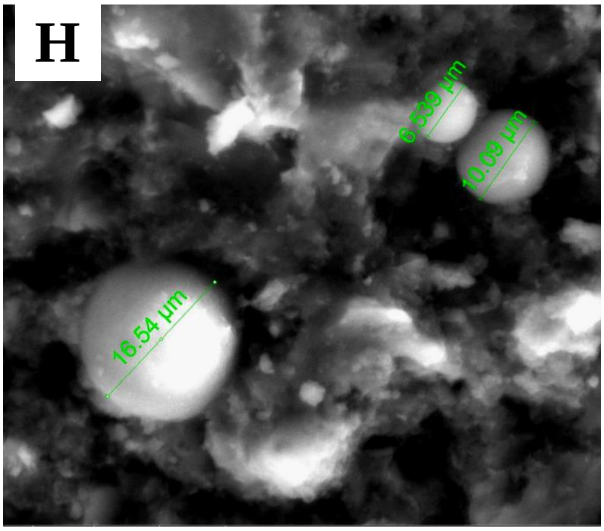


Figure 4: Scanning Electron Microscopic images of the hardened cement pastes cured for 7 and 28 days. FAxGAy stands for x% of FA and y% of GA in the mixture.

After a 28 days curing, FA0GA3 (Fig. 4E) has a microstructure similar to the control paste (Fig. 4A) having the hydration products of C–S–H gel, $Ca(OH)_2$ and ettringite phases. For FA6GA0, unreacted FA particles were visible after a 7 days curing (Fig. 4G), but they were under reaction after 28 days curing (Fig. 4H). In this case, there is the presence of FA grains which fill the pores of the paste. These phenomena are not observed for FA0GA306 and FA0GA0 (Fig. 4A and E).

In these age (28 days), the paste containing the GA (Fig. 4F and D) became denser, but they had more flexible microstructures that the control dough (Fig. 4B). This was in agreement with the corresponding results of the compressive strength (Fig. 3) and the time-dependent involvement of FA and GA in cement chemistry (Fig. 2). The pasta had denser microstructures after 28 days of curing (Fig. 4D and F), which is in line with similar 28 days compressive strengths of the pastes (Fig. 3). This implies that GA creates links result of potential reactions occurring between the GA and the various chemical components of the cement paste at early stage of curing, which leads to the formation of clutches between the grains filling the pores which increase the compressive strength of the cement.

CONCLUSION

In this investigation, mix ratio of the cement paste was optimized for the desired spread percentage of the fresh paste and the maximum possible compressive strengths of the hardened pastes for 7 and 28 days. The following conclusions can be made from the experimental results:

- The desirability functions found that the optimum independent variable setting at 36.0% W/B, 0% Fly Ash and resulted in the 7 and 28 days compressive strengths at 22.1 and 60.4 MPa, respectively.
- The compressive strength of pastes significantly increased with the increase of flow rate of the milling agent but with the decrease of FA.
- Increasing FA beyond 6% generally decreased the compressive strength at different age of curing (7 and 28 days).
- Grinding agent and clinker play a significant role in the development of compressive strength at different age of curing, whereas the addition of GA was found effective in supporting the chemistry in cement paste at early age (i,e., 7 days) and later curing stages, especially after a 28 days curing.
- The statistical model predicted the dependent variables of 7 and 28 days compressive strengths with good accuracy.
- SEM microstructural analysis supported delayed contribution of FA and GA to the development of compressive strength at late age of the hydration.

In summary, the extent of contribution that clinker, grinding agent and flay ash had on the development of the compressive strength was time-dependent for the cement pastes. Such a time-dependency of the cement chemistry with FA and GA 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 period is typically provided for cost reasons, leading to failure to meet the design life requirements of concrete structures.

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