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Effect of grinding agent and fly ash: modeling and optimization

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Abstract

This paper aims to optimize the effect of fly ash and grinding agent on the cement performance by using the box Behnken plan which consists to minimize the experiments number compared with classical design. This modeling optimization has shown that we can have a better mechanical property of cement, by staring the fly ash percentage in 5%, clinker percentage in 67% and grinding agent debit in 491Kg/t. In this case, the cement compressive strength reaches to 26.5, 40.0 and 50.8MPa after 7, 28 and 90 days of curing, respectively. these results showed that the milling agent begins to develop the cement compressive strength at a young age (at 7 days) but the fly ash begin to react after 28 days of curing. The reactivity of the fly ash and grinding agents is due to their composition and their structure which was examined by the SEM microstructure analysis. So, the effect of the grinding agent and fly ash should be considered during the cement formulation. The factors effects and their interactions on the cement properties was modeled and analyzed. The final results demonstrate how the dose and the percentage of each factor and their interactions can be manipulated to manufacture a mortar with a better quality and which satisfy the relevant European (EU) and Moroccan (NM) standards.

1. Introduction

Concrete is the most widely used building material nowadays [1-3]. Its properties greatly depend on the proportions and properties of its constituents. As cement is the major component of concrete and usually has relatively low unit cost, the selection of its proper type and use has vital importance in obtaining the balance of its desired properties in most economical way for any particular concrete mix [4-7].

The importance of the using additions such as fly ash and grinding agents is on the one hand to improve the physical, chemical and mechanical properties of the cement [8, 9]; on the other hand, it allows to reduce the cement price [10-12]. Fly ash is used as a supplementary cementitious material (SCM) in the production of Portland cement concrete. A supplementary cementitious material, when used in conjunction with Portland cement, contributes to the properties of the hardened concrete through hydraulic or Pozzolanic activity, or both. As such, SCM's include both Pozzolans and hydraulic materials [13]. Whereas, grinding agents have been traditionally used to improve the efficiency of the cement molturation process [14, 15], one of the most energy consuming processes in cement production (60 - 70% of the total electricity consumed in a cement plant) [16-20]. Grinding agents are normally liquid products, traditionally formulated as water based solutions of organic compounds with high charge density, such as glycols, esters of glycols, alkanolamines and/or carboxylates of alkanolamines [21]. The main function of grinding agents is to partially neutralize the charges present on the surface of cement particles, which develop during milling, reducing the surface free energy of the material being ground. For this purpose, the additive molecules are adsorpted over the surface of the cement particles by weak electrostatic forces, favoring the repulsion and/or a steric hindrance between particles, avoiding their agglomeration and thus improving grinding efficiency [22, 23]. To model the effect of these two additives (fly ash and grinding agent), the Benhken Box Plan has been used. This plan is a design of Experiments (DOE) which is a set of techniques that revolve around the influence study of different variables on the outcome of a controlled experiment with an experience minimum.

The percentage of the fly ash, clinker and grinding agent debit were the chosen factors to be studied. Those factors and their interactions determine the final properties of those mortars. The compressive strength was

defined as the desired response. Benhken Box Plan allows both the simultaneous effect of individual factors and synergic effects, resulting from interactions between factors, to be evaluated. These two effects can be positive, increasing the studied property, or negative, decreasing it. 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 containing was the materials, such as: Response Surface Methodology [24] full factorial designs [25], standard orthogonal arrays [26] or mixture experimental designs [27], as far as the authors are aware no studies have used Benhken Box Plan to assess and to optimize the impact of three factors and the synergic effect of the interactions on the final properties of lightweight cement mortars.
As such, this study aimed to compare and to assess significance of curing period for the compressive strength development of the cement pastes containing FA and grinding agent which contains monoethylene glycol. To this end, a global optimization of the mixture was made to find the mix design possessing the maximum achievable compressive strength of the hardened pastes cured for 7, 28 and 90 days.

2. Experimental details

2.1. *Samples*
Portland limestone cement CEM II/A-L 32.5N and Portland siliceous fly ash cement CEM II/A-V 32.5N are the two cement types which are used in this study and they are in compliance with ASTM C595.
- 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.

The chemical composition of the two cement types and fly ash is determined by X-ray fluorescence (XRF) and it is shown in Table 1.

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

%	CEM II/A-L32.5N	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 (m^2/kg)	3565±2.0	3565±2.0	n.a
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 grinding agent solution was purchased from the Chryso Company. The chemical composition and the property of the Chryso adjuvant are presented in the table 2.

Table 2: The chemical composition and **properties** of the Chryso adjuvant

Chemical composition of the Chryso adjuvant	
Components	**vol.%**
1,1',1"-Nitrilotripropane-2-Ol	25-50
Monoethylene glycol	10-25
Water	40-45
Properties of the Chryso adjuvant	
Density	1.06g/mL
pH	9.12
Conductivity	22mS/cm

2.2 *Compressive strength*
Mortar mechanical resistance was assessed after 7, 28 and 90 days of curing by measuring their compressive strengths. Tests were done in triplicates for compressive strength. To assess compressive strength, 4 ×4 × 16cm^3 mortar were submitted to a bending test according to EN 196-1standard: 2005 [28].

2.3 *Response Surface Methodology*
Benhken Box Plan (BBP) [29, 30] has been the most commonly used design method with response surface methodology (RSM) [31-33] in statistically assessing the mathematical relationship between the independent variables and the responses [34]. This study was designed in a three factors, two levels (2^3). Benhken Box Plan aiming to assess the main, quadratic and interaction effects of the independent variables, the clinker percentage (KK, 64–70, X_1), debit grinding agent (G, 0–700, X_2) and percentage fly ash (F, 0–700, X_3), on the dependent response variables, compressive strength (Y) of the hardened pastes (Table 4). The binder is defined as the total amount of Portland cement and fly ash. Benhken Box Plan was utilized to optimize the mix design in order to obtain a 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. The significance of each independent variable to the dependent variable and their interactions were determined by an analysis of variance (ANOVA) [35-37]. Factors with a p-value of 0.05 (5%) 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.

2.4 *Scanning Electron Microscopy*
Scanning Electron Microscopy (SEM) is used to evaluate the cement microstructural differences that could be attributed to time dependent contribution of fly ash and grinding agent to the chemistry of the cement pastes. To avoid the effect of the cement composition, four different cement specimens were prepared with combinations cited in the following table.

Table 3: Various combination of cement treated with SEM

cement composition	**% clinker**	***% gypsum***	***%Fly ash***	***%limestone***	***Grinding agent (Kg/t)***
symbol cement					
G0F0	67.0	3.0	0	30	0
G491F5	67.0	3.0	5	25	491

The cements G491F5 were prepared from the optimum values of the clinker, fly ash and grinding agent. Reference cements (G0F0) were prepared without grinding agent and fly ash. After storage for 7, 28 and 90 days in a moist environment at 20 ± 1°C, the cement samples were dehydrated in an ascending acetone alcohol and they dried at a temperature of 60°C for one hour. Images were obtained at magnifications between 1000 and 4000× and in low-vacuum (30keV).

3. Results and Discussion

The results indicate in the figure 1 and in the table 4 clearly showed that the Y was further developed even after 28 days of curing to gain additional Y during late age of curing (i.e., 90 days).

3.1. Statistical models of the box Behnken plan
The ANOVA of each dependent variable is shown in Table 5. 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. The high regression coefficients (R^2) of 96.8, 97.3 and 98.7 % for the 7, 28 and 90 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 5% (Table 5). The estimated regression models after removing insignificant terms for the Y's are given in Eqs. (1) to (3):

$$Y_{7d} = 24.5 + 2.0X_1 + 0.2X_2 \quad (1)$$

$$Y_{28d} = 33.7 + 2.9X_1 - 3.2X_2 - 1.6X_3 - 4.3X_2^2 + 1.1X_{12} - 1.5X_{13} - 0.9X_{23} \quad (2)$$

$$Y_{90d} = 43.4 + 3.5X_1 - 3.8X_2 - 1.8X_3 - 4.8X_2^2 + 1.3X_{12} - 2.0X_{13} - 1.0X_{23} \quad (3)$$

It should be noted that the coefficient values were for the terms of uncoded independent variables.

Table 4: Matrix experimental of box Behnken plan and the measured dependent variables

Run	Mix design of independent variables[a]						Measured dependent variables[b]		
	Coded			Uncoded			Y_{7d}	Y_{28d}	Y_{90d}
	X_1	X_2	X_3	X_1	X_2	X_3		(MPa)	
1	-1	-1	0	64	0	6	22.1	31.5	40.9
2	1	-1	0	70	0	6	26.3	34.7	44.6
3	-1	1	0	64	700	6	22.7	23.0	30.7
4	1	1	0	70	700	6	26.5	30.4	39.6
5	-1	0	-1	64	350	0	22.4	30.9	39.3
6	1	0	-1	70	350	0	26.4	40.0	50.8
7	-1	0	1	64	350	12	22.4	30.8	39.8
8	1	0	1	70	350	12	26.4	34.0	43.5
9	0	-1	-1	67	0	0	24.1	33.2	42.8
10	0	1	-1	67	700	0	24.4	28.5	37.3
11	0	-1	1	67	0	12	23.8	31.7	41.1
12	0	1	1	67	700	12	24.1	23.5	31.5
13	0	0	0	67	350	6	24.5	33.7	43.4

[a]*X_1: %Clinker, X_2: debit of milling agent and X_3: %Fly ash*
[b]*Y: Compressive strengths at 7, 28, and 90 days (mean ± standard deviations, n= 3).*

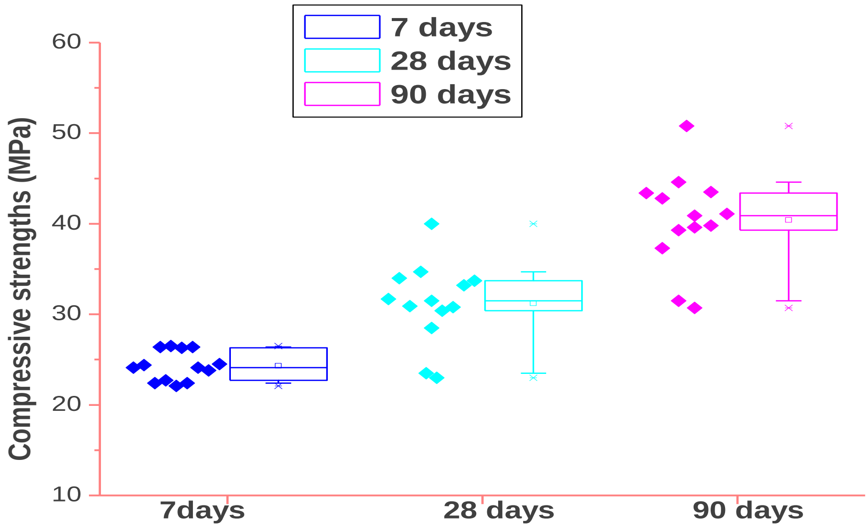


Figure 1: Box plots of the compressive strength

As shown in Eq. 1, 2 and 3, the two linear terms of X_1 and X_2 factors significantly affected the Y at different ages (7, 28 and 90 days) but the X_3 has only a significant effect on the model of 28 and 90 days. Thereby, the Y_{7d} was predicted with first order polynomial model, so the compressive strength of the paste at 7 days was predicted as a function of the linear terms of clinker percentage and grinding agent debit. But the Y_{28d} and Y_{90d} were predicted with 2nd order models. Although, the interactions X_{12}, X_{23} and X_{23} were found to be statistically relevant to the dependent variables Y_{28d} and Y_{90d} which had a negative effects on Y (Table 5). So, the value of Y decreases as the fly ash percentage and grinding agent debit increases. So, they were factored into those prediction models. A significant increase of the Y could be attributed to an enhanced fluidity caused by the surfactants coated on the chemical composition, the percentage of the clinker and the fly ash [38, 39].

3.2. Statistical model validation

Statistical model validation is provided by the Fisher test and statistical analysis of residues. This study will allow to prove that the three models Y_{7d}, Y_{28d} and Y_{90d} are statistically significant and validated, so those models can be used to predicate the cement compressive strength at 7, 28 and 90 days.

Table 5: ANOVA and full regression models statistics

Term	Y: Compressive strength					
	7 days		28 days		90 days	
	p-value	coefficient	p-value	coefficient	p-value	coefficient
Constant	< 0.01	24.50	< 0.01	33.70	< 0.01	43.40
$\mathbf{X_1}$	< 0.01	2.00	0.01	2.86	0.02	3.48
$\mathbf{X_2}$	2.97	0.17	< 0.01	-3.21	0.02	-3.79
$\mathbf{X_3}$	19.80	NS	0.07	-1.58	0.20	-1.79
$\mathbf{X_{12}}$	32.70	NS	15.20	NS	39.40	NS
$\mathbf{X_{22}}$	10.00	NS	0.02	-4.25	0.06	-4.81
$\mathbf{X_{32}}$	10.00	NS	41.30	NS	34.00	NS
$\mathbf{X_1X_2}$	21.90	NS	0.84	1.05	1.63	1.30
$\mathbf{X_1X_3}$	100.00	NS	0.28	-1.48	0.46	-1.95
$\mathbf{X_2X_3}$	100.00	NS	1.46	-0.87	3.21	-1.03

X_1: %kk, X_2: grinding agent debit, X_3: %FA
NS: The contribution of the terms was not statistically significant.

3.2. 1. Fisher test

The Fisher test allowed us to determine whether we accept or reject the null hypothesis H_0. In our study, we want to prove that the developed models of cement compressive strength are significantly predictive. The null hypothesis is therefore that the established models are not predictive. The F of this test is calculated by the following formula:

$$F = \frac{\text{explained variance}}{\text{unexplained variance}} \quad (4)$$

The analysis of the results of the Fisher test “F” showed that the developed models are very significant. Indeed, the “F” values of the cement compressive strength models at 7 to 90 days, equal to 212.20, 145.07 and 190.72, respectively and they are significant at 1%. The p-value of the three models is less than 1% which means the model of cement compressive strengths at 7, 28 and 90 days are significant at this level significance. Those results indicate that the tree models explain a significant variables variance proportion of the cement compressive strength at 7, 28 and 90 days.

Model of cement compressive strengths at (days)	F	p-value (%)
7	212.20	4.22
28	145.07	1.72
90	190.72	2.20

Table 6: Statistical models validation data

3.2. 2. Statistical analysis of residues

The residues of the tree models were minimized by using the least squares method, which is important to ensure that those residues are not abnormally large in some points. For this, we used the graphic method of Henry which is presented in figures 2:

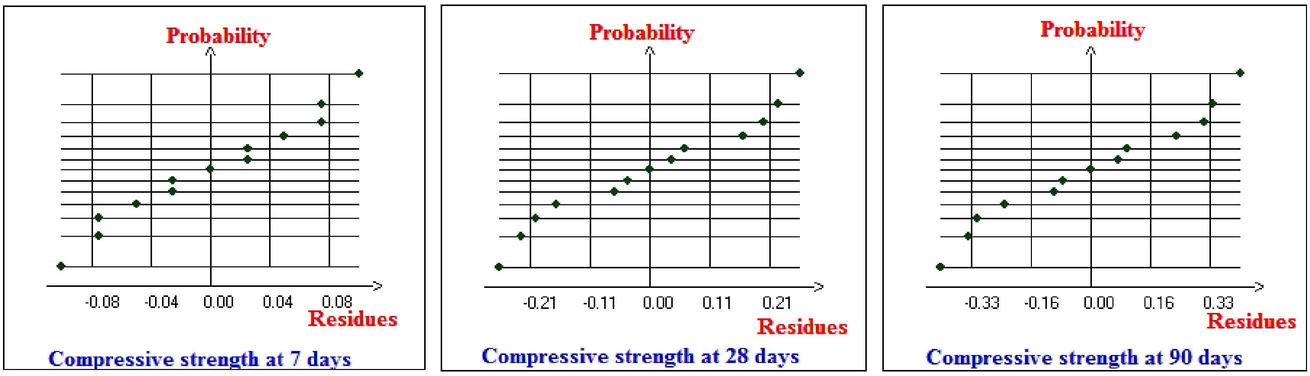


Figure 2: Henry line of model residuals of CHRYSO adjuvant

Construction straight Henry gives point clouds whose alignment is close to a straight. Indeed the p-value calculated by this test shows that the distribution of the population tested follows a normal distribution.
The statistical validation is a major step to achieve before moving to the optimization process because it shows that the models of cement compressive strength have excellent predictive qualities and its results can be used for design the Contour plots of Y_{7d}, Y_{28d} and Y_{90d}.

3.3. Response optimization of the cement pastes
Table 7 summarizes the optimization goals of the response surface methodology to find the best combination of independent variable settings that could produce the greatest Y's at the different curing periods. The greatest compressive strengths measured (Figure 1) were selected as the optimality criteria of Y's.

Dependent variable	Measured Y's		Optimization	
	Lower	Upper	Goal	Target
Y: compressive strength (MPa) at				
7 days	22.1	26.5	Maximum	26.5
28 days	23.0	40.0	Maximum	40.0
90 days	30.7	50.8	Maximum	50.8

Table 7: Optimization criteria for each dependent variable

The desirability functions were utilized to simultaneously optimize the responses. As shown in table 8, the optimum independent variable setting at 67% X_1, 491kg/t X_2 and 5% X_3 resulted in the Y_{7d}, Y_{28d} and Y_{90d} at 24.3, 31.5 and 40.8 MPa, respectively. This was attained with the global desirability value at 99.97% and with the response specific desirability values at 99.99, 99.93 and 99.99% for the Y_{7d}, Y_{28d} and Y_{90d}, respectively (table 8). It should be noted that the optimization goals could be assigned at different weights and importance.
The maximum compressive strength of the cement for the three ages can be reach at the same time, with just using a grinding agent and fly ash as the additives cement and which will improve the cement quality in remarkable way.

Dependent variable	**Name of dependent variable**	**Value (MPa)**	**Desirability values (%)**
$\mathbf{Y_{7d}}$	CS* 7days	24.3	99.99
$\mathbf{Y_{28d}}$	CS 28days	31.5	99.93
$\mathbf{Y_{90d}}$	CS 90days	40.8	99.99
	Global Desirability		99.97

**CS: Compressive strength*

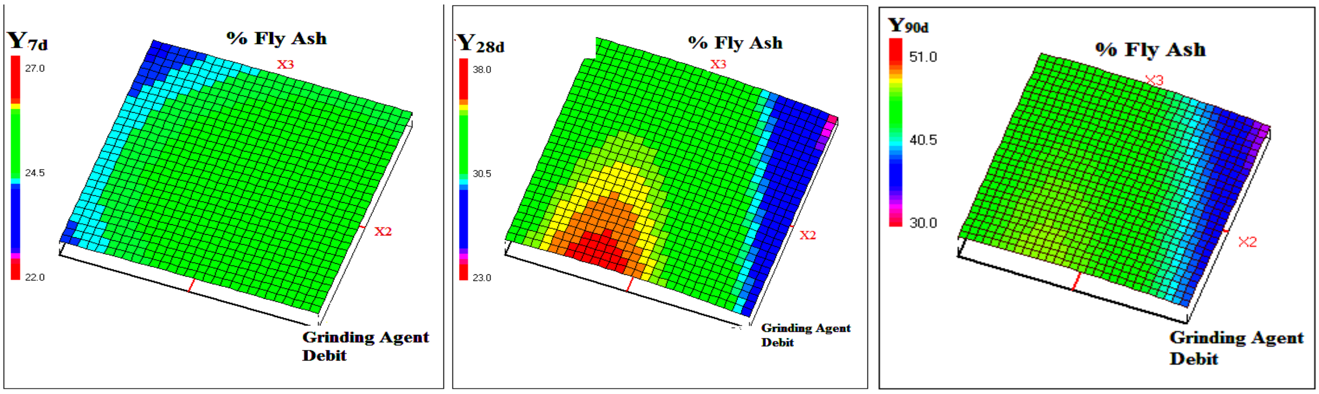
Table 8: The desirability functions of Y’s

3.4. Contour plots for box Behnken plan 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 Figure 3, the Y_{7d} increased with the increase of X_2 but it isn't influenced by fly ash values, on the other hand, Y_{28d} and Y_{90} increase with the decrease of X_2 and X_3 at the same time, while the X_1 was held at the optimum level of 67%. So, at 28 and 90 days, the values of Y decrease as the percentage of fly ash exceed the 5% despite the increased throughput of the grinding agent. This shows that the fly ash has an optimal effect on the pozzolanic cement, which leads to improving the compressive strength of the cement, but when the percentage of AF exceeds 5%, this product has a negative effect on the cement curing. In the long term (28 to 90 days), fly ash and Chryso adjuvant have a remarkable effect on the cement compressive strength.

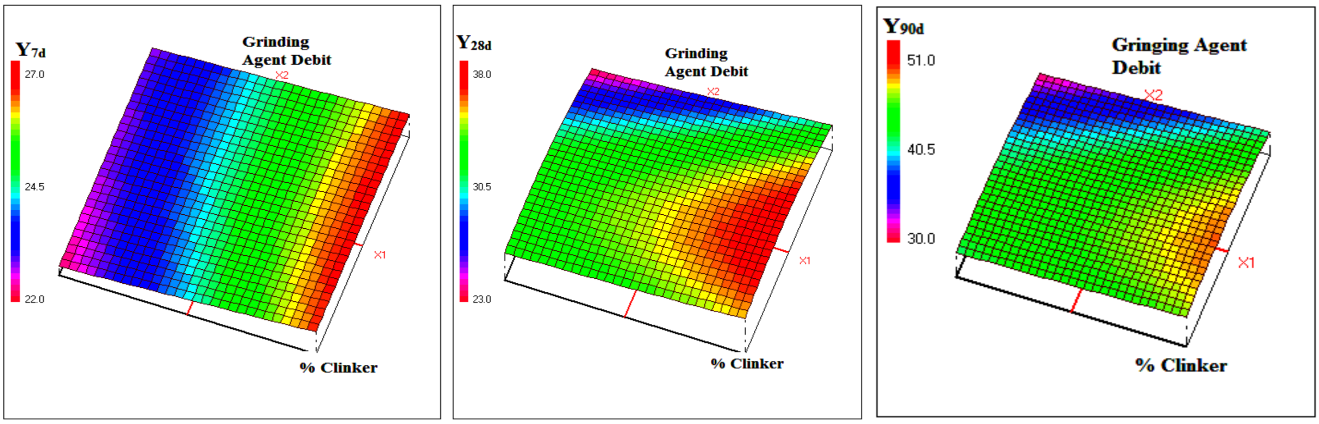
When the X_3 was held at the optimum level of 5%, the simultaneous increase of X_1 and X_2 enhanced the Y_{7d}, but for the Y_{28d} and Y_{90d} values increased with the increase of X_1 and the decrease of X_2. With the X_2 held at the optimum level of 491Kg/t, an enhanced Y was found with the increase of X_1 at all the times and the decrease of X_3 for Y_{28d} and Y_{90d}.

Overall, a greater Y was found with a greater X_1 and X_2 but with a lesser X_3. The increase of X_2 would facilitate fluidity of the cement pastes attributed to the polymer (monoethylene glycol) coated on the grinding agent. This was in agreement with Ouyang et al. [40] who documented an increased fluidity of cement with the addition of grinding agents. 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 [41, 42]. The extent of contribution that independent variables had on the development of compressive strength was time-dependent.

In a function of X_3 and X_2, while $X_1$was at 67%



In a function of X_1 and X_2, while X_3 was at 5%



In a function of X_1 and X_3, while X_2 was at 491Kg/t

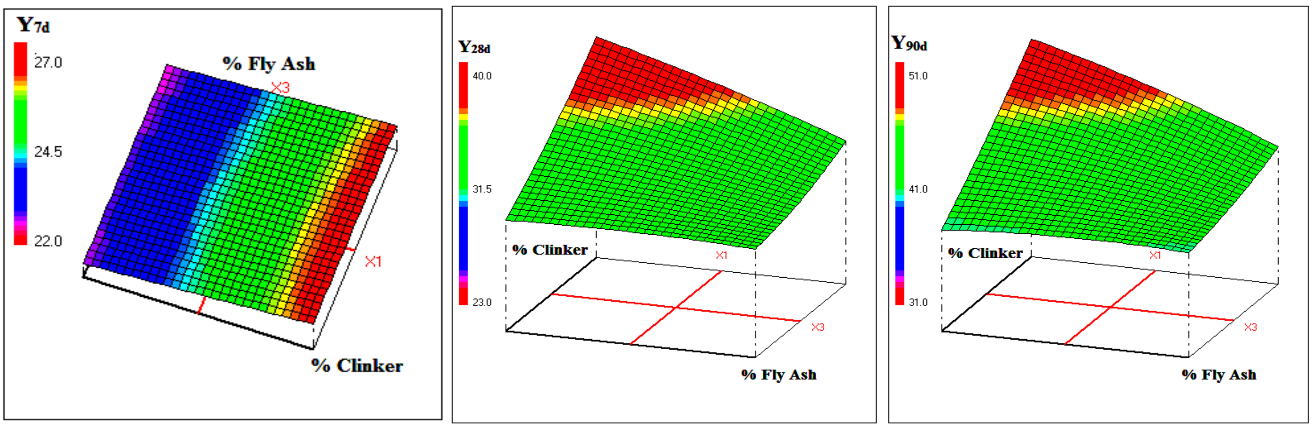


Figure 3: Contour plots of the Y_{7d}, Y_{28d}, and Y_{90d}

3.5 Model validation

3.5.1. Experimental validation

The three prediction models were validated by performing another set of experiment where the specimens were made in triplicate at the global optimum mix ratio obtained in Section 3.3 and by comparing the difference between predicted - measured responses and the absolute relative percent error (PE). As shown in Table 9, the lowest PE of the Y's, was found for the Y_{90d} (1.3), followed by 1.6 for the Y_{28d} and 2.0 for the Y_{7d}. Therefore, the three models generally predicted the dependent variables of Ys with good accuracy.

Table 9: Validation results of the optimum independent variables

Optimum mix (wt.%)			Y: Compressive strength (MPa)[a] at								
			7 days			28 days			90 days		
X_1: KK	X_2: GA	X_3: FA	Pred.	Meas.	PE[b]	Pred.	Meas.	PE	Pred.	Meas.	PE
67 %	491 %	5 %	25.7	25.5	2.0	39.5	39.3	1.6	45.4	45.0	1.3

[a] Data are the average of triplicate samples
[b]Absolute relative percent error=|1- value$_{predicted}$/value$_{measured}$|×100 %
[c]Standard deviations (n= 3).

3.5.2. Validation by SEM analysis

This experiment consists to compare and analyze the compressive strength and microstructure of cement samples prepared from the composition indicated in the table 3 and which have the following compressive strength (table 10).

Table 10: Compressive strength of the cement pastes prepared for the SEM analysis

symbol cement	*compressive strength at (days)*		
	7	**28**	**90**
G0F0	24.1	33.2	42.8
G491F5	25.5	39.3	45.0

The compressive strength of the paste containing the grinding agent and fly ash (G491F5) at 7,28 and 90 days, had 5.8%, 18.4% and 5.1% higher than those without fly ash and grinding agent (G0F0) (table 10). X_2 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 (Figure 4).

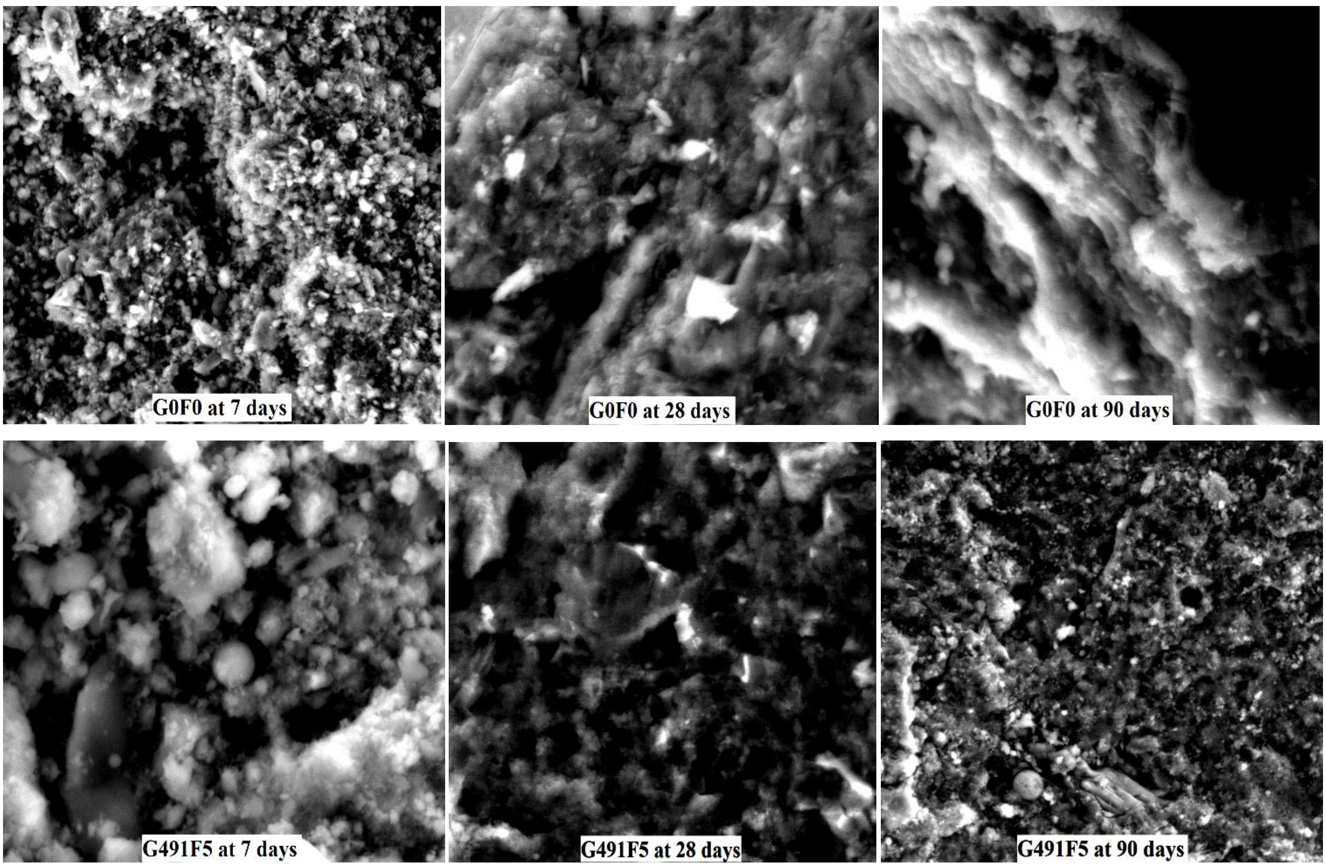


Figure 4: Scanning Electron Microscopic images of the hardened cement pastes cured for 7, 28 and 90 days. G_xF_y stands for x % of grinding agent (G) and y% of fly ash (F) in the mixture

At 7 days of curing, G491F5 paste has a microstructure similar to the control paste (G0F0), by having the hydration products of C–S–H gel, $Ca(OH)_2$ and Ettringite phases. For the samples which contain fly ash and grinding agent, we notice that fly ash particles were reactive after a 7 days curing (G491F5), especially in after 28 and 90 days curing. In this case, the fly ash grains were filling the pores of the paste and these phenomena are not observed for G0F0, for this we were observed the appearance of vacuum between particles in this age (28 days). The paste containing the grinding agent and fly ash became denser that the control dough which is in line with similar 90 days compressive strengths of the pastes. This implies that grinding agent creates links result of potential reactions occurring between the grinding agent 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 that increase the compressive strength of the cement.

Conclusions

The study undertaken in this paper indicates that 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. The experimental results conducted through this study allow drawing the following general conclusions:

- The model of cement compressive strength at 7 days is an equation of the first degree which is influenced by the clinker percentage and the grinding agent debit.
- The compressive strength at 28 and 90 days is presented by 2nd degree equations which are presented by the main variables effect of their interactions.
- Increasing fly ash percentage beyond 5 % and grinding agent debit over 491%, generally decreased the compressive strength at 28 and 90 days of curing (7, 28 and 90 days).
- the effect of milling agent begins to appear at an early age (7 days), while the fly ash have slow effects because they begin to react after 28 days of curing.
- SEM microstructural analysis has proved that fly ash and grinding agent have a significant effect on the improvement and development of the cement microstructure which leads to progress of the cement compressive strength.

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