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Prevention of the cement's quality of CPJ₃₅ and CPJ₄₅: Statistical model.

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ABSTRACT

In this paper, it is aimed to propose prediction approaches from the 2, to 28-days compressive strength (CS) of Portland composite cement (CPJ₃₅ and CPJ₄₅) by using the Multilinear Regression (MLR). The main groups which characterize the CS were the percentages of the different components of the cement, the clinker's chemical composition, as well as the distributions of the cement particle size. The maximum variance of the compressive cement strength (CCS) at 2, 7 and 28 days, explained by the multilinear regression, is 95%, 94% and 97%, respectively. In fact, the utility of the models is in the potential ability to control processing parameters to yield the desired strength levels and in providing information regarding the most favorable experimental conditions to obtain maximum compressive strength at the target.

Keywords: Compressive cement strength, cement, clinker, Portland composite cement, prediction, multilinear regression

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INTRODUCTION

Compressive strength of Portland cement mortar at 28 days is the major property that defines its quality and depends on several factors that need to be controlled during manufacture [1]. But, it's a long time for the industry to wait for 28 days to get the experimental results for the compressive cement strength (CCS) [2].

The rapid determination of the compressive cement strength would be very beneficial for cement plant. It could be performed either by experimental test or by using mathematical models. These last years, the mathematical approach showed its compatibility for modelling the cement composition [3,4]. The CCS depends on many different factors namely C_3S , C_2S , C_3A , C_4AF and the fineness of the milled product and has varying degrees of effect on strength. The effect of these factors on the compressive strength has been extensively studied by using analytical models, including statistics model [1]. Thus, the use of multivariate analysis techniques such as multilinear regression (MLR) seems a simple and promising approach for predicting CCS.

Furthermore, several studies have been conducted to predict the compressive strength of Portland cements without addition by employing nonlinear regression [5], fuzzy logic [6], the gene expression programming (GEP) [7], neural networks [3] and the methods of regression [8,9]. Interestingly, there is no published work in the literature that makes use of statistical modeling approaches on the prediction of the CS of Portland composite cement. This paper makes such an attempt by using MLR [10,11] for the prediction of compressive strength of CPJ_{35} and CPJ_{45} from the clinker chemical and mineralogical composition.

In this fact, the data from the local cement industry «ASMENT TEMARA-Morocco » for standard curing were employed in this research by feeding them to a MLR based to create a model to describe the cement strength (because testing the strength of cement is ordinarily performed by mixing cement with sand and water [3], the cement mortar is also commonly referred to as cement). The data were collected during eight (8) months of plant operation. Because the plant operational parameters occasionally vary, so the data had some variations that must be taken care of before modeling. In other words, the average strength of the shipped product varied as a function of time. Therefore, modeling the system for the data for the first seven (7) months and testing the quality of the model for the remaining one month could produce a biased model. To alleviate this issue, the model used is evaluated by using the Multivariate Analysis Of Variance (MANOVA) [12] and the coefficient of multiple determinations.

MATERIALS AND METHODS

Materials

The compressive strengths at 28 days of the Portland composite cement CPJ_{35} and CPJ_{45} , equals respectively 35 and 45 MPa. therefore, those cements are prepared in Asment Temara industry, and its compressive strengths are determined according to the standard Moroccan NM.10.01.005 [13] and the European standard EN 196-1 [14].

Methods

The estimation of compressive strengths of cement type CPJ_{35} and CPJ_{45} , at the three different ages (2, 7 and 28 days) was performed by stepwise backwards regression method (MLR) [15]. The constructions principles of the linear model are based on the least squares method to estimate the mathematical equation parameters [16]. Indeed, this model requires the definition of two types of variables, endogenous and exogenous. The exogenous variables X_j represent the input of the model and endogenous variables Y , the output of the model.

The contribution rationale of each variable in the model is performed by the Student test, Fisher and Durbin-Watson test, after determining the significance level. For this, it was necessary to provide a training sample for model development and another for the validation test.

The validation test of the predictive model is used to test if the effect of exogenous factors is significant or not, in addition to the study of the coefficients of multiple determinations R^2 which should get close to 1 (or 100%).

The experimental validation of the linear model is performed by evaluating the difference between the observed and predicted values of the dependent variable. For a perfect model, this difference should be less than the standard error of the established model.

Data Collection

The data of the chemical, mineralogical composition of the clinkers and the compressive cements strengths corresponding to CPJ₄₅ and CPJ₃₅, were collected from the company “Asment Temara” during an 8 months of plant operation (from January to August 2014). To establish the predictive model of cement quality, we defined 17 variables that they are considered as the inputs of the MLR and there is also three outputs (CCS at 2, 7 and 28-day CSC) that will be predicted (table 1). The data for the first 7 months (data for 148 days of production) are used as the training data, and those of the 8th month (data for 25 days of production) are used as the test data for the MLR algorithms.

RESULTS AND DISCUSSION

Determining variables

The studied cements CPJ₃₅ and CPJ₄₅ are consist of four materials: clinker, limestone, fly ash and gypsum. Each one of this material has his own physic-chemical characteristics. In this case, the chemical composition of this material is evaluated by measuring the magnesium oxide MgO, the silica SiO₂, the alumina Al₂O₃, the iron Fe₂O₃, the sulfur SO₃ and the free lime CaO. The measured physical parameters are the LOI and the finesse (refusal at 80 microns).

For the mineralogical parameters which characterize the quality of the clinker are the silica modules: The alite C₃S, the belite C₂S, the tricalcium aluminate C₃A and the tetracalcium aluminoferrite C₄AF. In total, there is 17 parameters which are the exogenous variables of the multilinear regression model and they are developed for each cement according to: NM.10.01.004 [17] and the European standard EN 197-1 [18]. In this study, the number of the cements studied is 148 samples.

The quality of the cements CPJ₃₅ and CPJ₄₅ is designed by the compressive cements strengths at 2, 7 and 28 days. They are appointed by y_2 , y_7 , y_{28} , and they present the exogenous variables of the model. Table 1 shows the format and the coding of the individual input and output variables.

Table 1: Coding of the variables needed to build the statistical models

Code	Input variables	Code	Output Variables
d ₁	C ₃ S		
d ₂	C ₂ S		
d ₃	C ₃ A		
d ₄	C ₄ AF		
d ₅	SiO ₂	y ₂	CCS at 2 days
d ₆	Al ₂ O ₃		
d ₇	CaO		
d ₈	CaO _i		
d ₉	Fe ₂ O ₃	y ₇	CCS at 7 days
d ₁₀	SO ₃		
d ₁₁	MgO		
d ₁₂	LOI		
d ₁₃	Refusal at 80µm		
d ₁₄	% Clinker		
d ₁₅	% Fly Ash (FA)	y ₂₈	CCS at 28 days
d ₁₆	% Gypsum		
d ₁₇	% Limestone		

Table 2 shows the average characteristics of the input and output parameters which are used in the models of the multilinear regression and they are corresponding to 148 samples.

Table 2: Average characteristics of input and output data of the MLR models

	Variable	Minimum (%)	Average (%)	Maximum (%)
Input variables	Clinker (KK)	65.80	80.45	97.00
	Limestone	0.00	13.21	23.40
	Gypsum	3.00	4.40	5.40
	Fly Ash (FA)	0.00	1.95	9.00
	MgO	1.00	1.45	2.50
	C ₃ A	6.50	7.39	8.50
	C ₂ S	5.10	17.15	23.60
	SO ₃	0.90	1.64	2.50
	CaO _I	0.60	1.75	4.10
	C ₃ S	49.90	56.68	67.20
	C ₄ AF	8.60	10.82	12.20
	CaO	64.90	65.49	67.10
	SiO ₂	19.40	20.86	21.70
	Al ₂ O ₃	4.80	5.06	5.40
	Fe ₂ O ₃	2.80	3.56	4.00
	LOI	0.10	0.16	0.90
Refusal at 80µm (R ₈₀)	0.40	2.53	6.50	
Output variables	CCS at 2 days	8.80	17.70	32.80
	CCS at 7 days	17.20	28.87	48.50
	CCS at 28 days	27.20	41.95	58.0

Model construction and analysis using MLR

The equation of the multivariate regression which linked the input variables (d₁, d₂, d₃, ..., d₁₆, d₁₇) to the output variable (y₂, y₇ and y₂₈), and which is written in the form:

$$y_n = f(d_1, d_2, d_3, \dots, d_{16}, d_{17})$$

With: n = 2, 7, 28 days.

The functions developed by the MLR will be used to produce compressive strengths of the cements CPJ₃₅ and CPJ₄₅ at 2, 7 and 28 days. The treatment of the stepwise regression data of all the constituents of the cement, clinker and the experimental results of the compressive strength at the different ages, were conducted by SPSS software [19-21].

The execution of the stepwise regression MLR statistical processing of data is allowed to screen all input factors and to select those that have a significant effect on responses. The different combinations of these variables were selected to intuitively take into account all the variables in the global model, in order to eliminate one by one, those variables corresponding to the smallest value of the “t” of the Student test, represented by “p-value” (p-value < 0.05).

Accordingly, the algorithm of the MLR has selected from among the input variables, which one can provide the greatest reduction of the residual variance (unexplained) of the dependent variables. In other words, these variables have the highest partial correlation with the response y (CCS 2, 7 and 28 days).

The coefficients forming the compressive strengths models of the cements CPJ₃₅ and CPJ₄₅ are listed in Table 3.

Table 3: Coefficients forming the three models corresponding to CCS at different ages

Time (days)	Model of the CCS at								
	2			7			28		
Input variables	Coefficients	t	p-value	Coefficients	t	p-value	Coefficients	t	p-value
Constant	-21.50	-0.22	0.83	-36.95	-0.27	0.79	-76.40	-0.64	0.52
FA	0.46	4.31	0.00	0.42	3.09	0.00	0.67	5.18	0.00
KK	0.45	7.09	0.00	0.44	5.97	0.00	0.66	8.48	0.00
R ₈₀	-1.76	-10.39	0.00	-2.69	-12.6	0.00	-3.27	-15.73	0.00
C ₂ S	2.03	2.24	0.03	3.91	3.11	0.00	-5.00	-4.54	0.00
SO ₃	-4.49	-2.56	0.01	-6.34	-2.59	0.01	11.99	5.59	0.00
CaO _I	-6.31	-2.12	0.04	-12.40	-3.00	0.00	15.06	4.13	0.00
CaO	6.40	2.00	0.04	12.06	2.75	0.01	-14.50	-3.78	0.00
SiO ₂	-18.22	-2.59	0.01	-32.61	-3.31	0.00	43.06	4.99	0.00
Al ₂ O ₃	-7.98	-1.78	0.03	-16.97	-2.71	0.01	28.81	5.24	0.00
Fe ₂ O ₃	-3.90	-2.82	0.01	-6.04	-3.12	0.00	3.11	1.83	0.02
Gypsum	1.26	3.23	0.00	-	-	-	1.53	3.21	0.00

Moreover, the counting of the results in the table 3 by estimation of the parameters by the maximum likelihood, reveals that there is eleven variables (11) truly significant in the multivariate models for predicting CCS at 2 and 28 days also there is ten variables (10) in the model predicting of CCS at 7 days, according to the values of probability (p-value < 0,05). Similarly, the SPSS software tells in its 4th step of treatment that "no other variable can be deleted or added to the current model". Therefore, the algorithm of the model MLR is systematically removed the variables whose its significance is too low, compared to the resistance of 2 to 28 days at each stage. And for each 4 steps, the non-selected variables in the three models are shown in Table 4.

Table 4: Variables excluded in the three models MLR

Time (days)	Model of CCS at								
	2			7			28		
Step	variables deleted	t	p-value	variables deleted	t	p-value	variables deleted	t	p-value
1	C ₃ A	.	>0.1	C ₃ A	.	>0.1	C ₃ A	.	>0.1
	C ₃ S	.	>0.1	C ₃ S	.	>0.1	C ₃ S	.	>0.1
	C ₄ AF	.	>0.1	C ₄ AF	.	>0.1	C ₄ AF	.	>0.1
2	Limestone	0.12	0.90	limestone	0.08	0.94	MgO	0.46	0.64
3	LOI	0.14	0.89	MgO	0.21	0.83	Limestone	0.54	0.59
4	MgO	1.25	0.21	LOI	-0.36	0.72	LOI	0.69	0.49
	-	-	-	gypsum	1.33	0.18	-	-	-

Statistical model validation tests

Table 5: MANOVA data

Time (days)	Models	Model of CCS at					
		2		7		28	
		F	p-value	F	p-value	F	p-value
1	Regression	84	0.000 ^b	76	0.000 ^b	160	0.000 ^b
2	Regression	91	0.000 ^c	82	0.000 ^c	174	0.000 ^c
3	Regression	100	0.000 ^d	90	0.000 ^d	189	0.000 ^d
4	Regression	108	0.000 ^e	98	0.000 ^e	207.5	0.000 ^e
	Regression			107	0.000 ^f		

The model validation was carried out by the coefficients of multiple determination test R², Fisher and Durbin-Waston test, which were calculated from the data indicated in the table of the Multivariate Analysis Of Variance (MANOVA) (Table 5). The data results of the three tests are significant at the 4th stage because that the R-squared (R²) values is 0.95; 0.94 and 0.97 for CCS at 2, 7 and 28 days, respectively. So we conclude that

the global significance of the models is good. Thus, the resulting models have excellent predictive qualities (Table 6).

The variation of Fisher test associated to the three models is significant (p-value < 0,001). Therefore, these models explain a significant proportion of the variables variance of the compressive cement strength of the cements CPJ₃₅ and CPJ₄₅ at 2, 7 and 28 days.

Table 6: Statistical models validation data

Measurement time of the CS	Model	R ²	Standard error of the estimate	Variation of F	Sig. Variation of F	Durbin-Watson
2 days	1	0.95 ^a	1.96	84.13	0.000	2.005
	2	0.95 ^b	1.96	0.014	0.000	
	3	0.95 ^c	1.95	0.018	0.000	
	4	0.95 ^d	1.95	1.57	0.000	
7 days	1	0.943 ^a	2.76	75.75	0.000	1.840
	2	0.943 ^b	2.75	0.007	0.000	
	3	0.943 ^c	2.74	0.044	0.000	
	4	0.943 ^d	2.73	0.128	0.000	
		0.942 ^e	2.74	1.78	0.000	
28 days	1	0.972 ^a	2.41	160.61	0.000	1.600
	2	0.972 ^b	2.40	0.214	0.000	
	3	0.972 ^c	2.40	0.281	0.000	
	4	0.972 ^d	2.39	0.474	0.000	

a. Dependent: 28J

b. Predictors: (constant), Fe₂O₃, FA, C₂S, SO₃, Al₂O₃, R₈₀, gypsum, CaO_i, MgO, LOI, limestone CaO, KK, SiO₂

c. Predictors: (constant), Fe₂O₃, FA, C₂S, SO₃, Al₂O₃, R₈₀, gypsum, CaO_i, LOI, limestone CaO, KK, SiO₂

d. Predictors: (constant), Fe₂O₃, FA, C₂S, SO₃, Al₂O₃, R₈₀, gypsum, CaO_i, LOI, CaO, KK, SiO₂

e. Predictors: (constant), Fe₂O₃, FA, C₂S, SO₃, Al₂O₃, R₈₀, gypsum, CaO_i, CaO, KK, SiO₂

f. Predictors: (constant), Fe₂O₃, FA, C₂S, SO₃, Al₂O₃, R₈₀, CaO_i, CaO, KK, SiO₂

In this study, the acceptable value of the Durbin-Watson test, are respectively 2.005; 1.84 and 1.6 for the prevention models of CCS at 2, 7 and 28 days because they are between 1 and 3 [15] and they are closed to the value of 2. This saves that there are fewer problems in terms of the independence errors [22,23].

The analysis of the results of the Fisher test “F” (Table 6) showed that the developed models are very significant. Indeed, the “F” values of the CCS models at 2 to 28 days, equal to 108, 107 and 207.5, respectively and they are significant at p-value < 0,001. This result indicates that we have less than 0,1% chance of being wrong in claiming that the models contribute better to predict the CS and the level of significance is the best. Accordingly, the three equations of the regression MLR are very good and record that the variables forming the prediction equation of the CS at 2, 7 and 28 days contribute in a very reproducible way in the CCS variable score at these tree ages, while providing a significant an amount of the information to models with a maximum error of the data which is about 1.94; 2.7 and 2.4. The functions generated by the algorithm MLR presenting the best outcome of predicting CCS of CPJ₃₅ and CPJ₄₅ at 2, 7 and 28 days according to the chemical and mineralogical composition of the clinker, are given by equations (1), (2) and (3).

Time (days)	Equations compressive strengths of cements CPJ ₃₅ and CPJ ₄₅ at different ages
2	$Y_{2days} = -21,49 + 0,46FA + 0,45KK - 1,76R_{80} + 2,03C_2S - 4,5SO_3 - 6,31CaO_1 + 6,4CaO - 18,22SiO_2 - 7,98Al_2O_3 + 1,26Gypsum - 3,9Fe_2O_3$ (1)
7	$Y_{7days} = -36,96 + 0,42FA + 0,44KK - 2,7R_{80} + 3,91C_2S - 6,34SO_3 - 12,5CaO_1 + 12,06CaO - 32,61SiO_2 - 16,97 Al_2O_3 - 6,04Fe_2O_3$ (2)
28	$Y_{28days} = -76,38 + 12SO_3 + 15,06 CaO_1 - 5,00 C_2S - 14,50 CaO + 43,06 SiO_2 + 0,67 FA + 0,66kk - 3,27R_{80} + 28,81Al_2O_3 + 1,53 Gypsum + 3,11Fe_2O_3$ (3)

Experimental validation of the models

Model validation of the compressive strengths of the cements CPJ₃₅ and CPJ₄₅ at 2, 7 and 28 days was conducted by established experimental development of 25 mortars cements according to the standards

NM.10.01.004 [17] and the European standard EN 196-1. Those cements were taken directly from the cement Dozer of the cements CPJ₃₅ and CPJ₄₅. The physico-chemical and mineralogical composition of the various components has been performed in the laboratory of the company Asment Temara according to standard NM.10.01.005 [13].

The results of the validation test of the three functions are shown in figures 1 to 3. The compressive strengths of the two types of cements CPJ₃₅ and CPJ₄₅ at the different ages were measured experimentally for 25 days corroborate to those calculated and theoretically predicted from the three models established by multiple regression. So the three functions of the MLR are able to present the real value of the compressive strength with the minimal error.

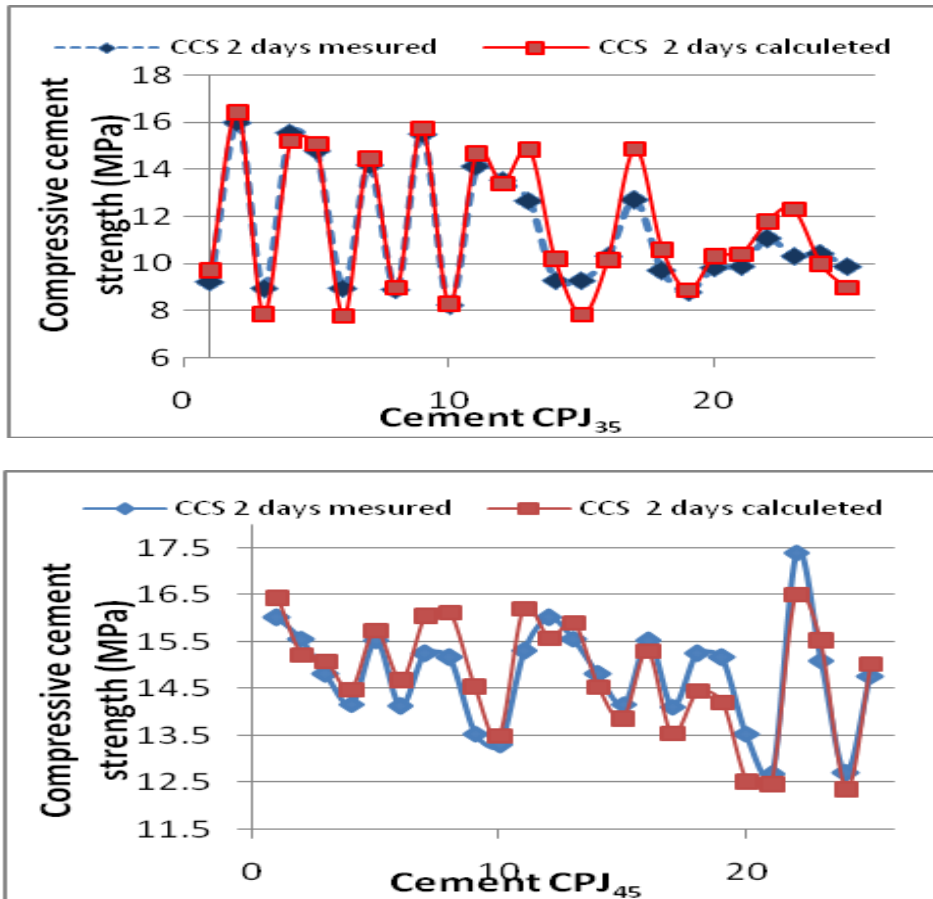
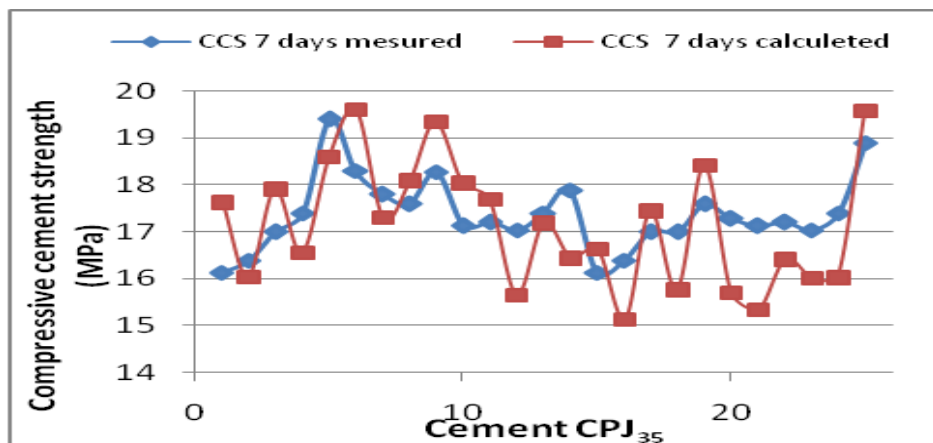


Figure 1: Evaluation of the MLR model for predicting resistance of 25 trying of the cements CPJ₃₅ and CPJ₄₅ at 2 days



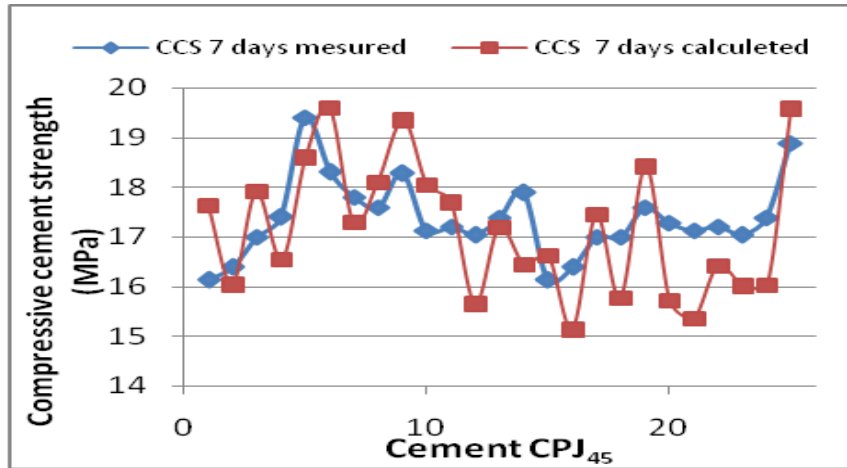


Figure 2: Evaluation of MLR model for predicting resistance of 25 trying of the cements CPJ₃₅ and CPJ₄₅ at 7 days

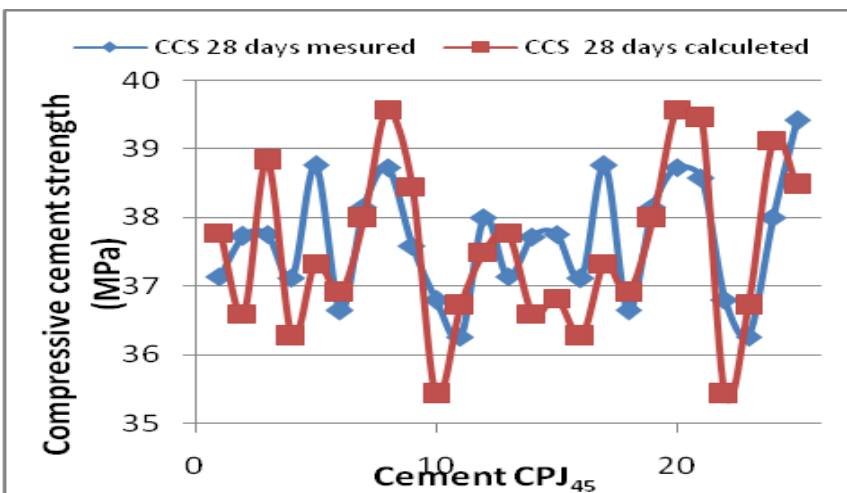
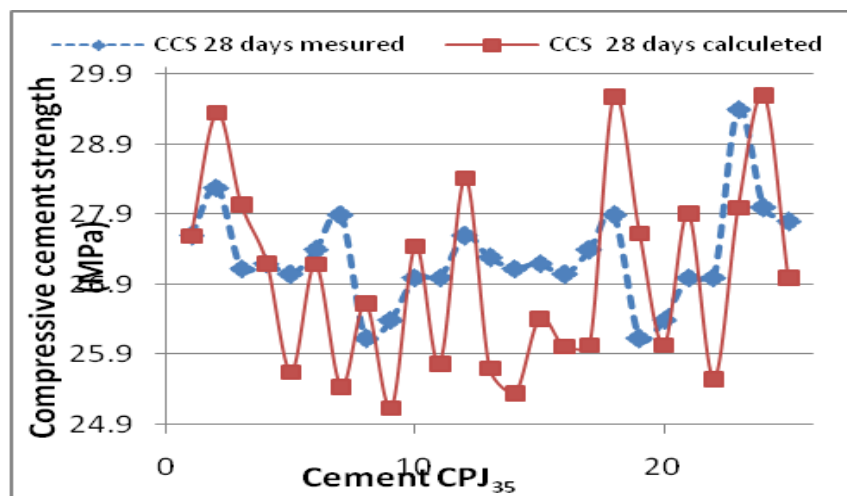


Figure 3: Evaluation of MLR model for predicting resistance of 25 trying of the cements CPJ₃₅ and CPJ₄₅ at 28 days

The results presented in Figures 1, 2 and 3 show that the variability explained by models 1, 2 and 3 is good, since the calculated differences between the compressive cement strength calculated from the established mathematical equations and those measured by the traditional method is always less than the error related to the established model and which is equal to 1.96; 2.74 and 2.39 for the model of 2, 7 and 28 days, respectively. So, the three models are experimentally reliable and predictive.

CONCLUSION

In this study, three multilinear regression models were developed for prediction the compressive strength of the cements CPJ₃₅ and CPJ₄₅ at 2, 7 and 28 days, according to the percentage of its constituents (limestone, clinker, gypsum and fly ash), the chemical and mineralogical composition of the clinker. The analysis of the MLR has shown that these models have a high predictive power of the compressive cement strength from the combination of effects of the selected factors. Similarly, the effects of the factors: Al₂O₃, Fe₂O₃, CaO, C₂S, SiO₂, SO₃, CaO, % fly ash, % clinker, % gypsum, including the size distribution of the cement grains, were modelled to have the models with the least errors.

Feasibility tests of these industrial-scale models revealed that the exploited models are very promoters and they are useful tools to prevent the compressive cement strength at any age.

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