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Statistical Modelling of the Compressive Strength of Mortar Based on Cement Blended with Mineral Additions by the Method of Experimental Design

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Abstract: This experimental study aims to study the mechanical behaviour of a mortar based on cement blended with mineral additions (pozzolana, limestone and slag), knowing that the mechanical strength of a mortar is closely related to its composition. The use of the three mineral additions simultaneously, presents a high number of factors affecting the mechanical resistance and requires a very large number of experiments and the obtained data analysis becomes much more complex. In order to optimise the number of tests and to achieve a such satisfactory analysis, a statistical approach known as an "experimental design" was used. The experimental methodology has been established to assess the compressive strength of mortars at 2, 7, 28 and 60 days, by the elaboration of an experimental design for a set of cement mixtures, the level of the three additions (factors), slag, limestone and pozzolana at rates varying from 0% to 35%, provided that a fixed dosage of 35% is maintained for all combinations to form a binary, ternary and quaternary cement in accordance with cement standard requirements CEM II/B. This statistical approach allowed us to evaluate by a numerical analysis the effect of each addition alone as well the meaning of the double or triple interaction resulting from the association of two or three additions at a time. In addition, it has enabled us to establish a representative model that permitted to estimate and predict the mechanical behaviour of any composition in the experimental program with tolerable errors. The obtained results lead to a satisfactory numerical modeling of the compressive strengths, in particular at the age of 28 days, with a trend curve of a an acceptable determined coefficient of R^2 equal to 0.87.

Keywords: Compressive strength, Mortar, Mineral additions, Statistical analysis, Experimental design**.**

Introduction

The cement industry has been transformed by the use of mineral wastes such as silica fumes, fly ash and blast furnace slags as cement-based additions through the economic, environmental and functional benefits (Kerbouche, 2009), whose purpose to value this waste, knowing that these mineral additions have interesting hydraulic and pozzolanic properties.

The properties of a cement are closely related to its composition, therefore the behavior of mortar with mineral additions that are influenced by the nature, hence the diversity of the additions in their chemical, mineralogical composition and their reactivity property, which differs from one to another, either active or not (Paillere et. al., 1977).

This influences the properties of a mortar differently with respect to the effect size of such addition and the significance of the interactions among them according to the type of the binary, ternary or quaternary cement type.

The majority of studies going in one direction pointed a trend towards the use of mineral additions. It is in this development action that we studied binary, ternary and quaternary cements, combining clinker with three additions, when analysis of the results remains very complex. On the other hand, a practical and computerized statistical approach called "experimental design" (Jacques et. al., 2013) which minimizes the number of tests, optimizes the mixtures and allows a modeling of the studied response, it is an experimental method whose use is very advanced in many areas.

In addition, this approach has proved satisfactory in several works in the field of materials (Caré *et al.*, 2000). This method of experimental design has been adopted for the study of the mechanical response of a mortar based on blended cement in the present research work.

Materials

In this experimental program local materials were used. The clinker obtained from Ain Kebira's cement factory (from Sétif is from Algeria). The gypsum from a depository quarry in Djemila (Sétif- Algeria). Granulated blast furnace slag is a by-product of the El-Hadjar (Annaba- Algeria) iron driven from a steel complex. The natural pozzolana from the Beni-Saf deposit (Ain T'emouchent- Algeria) quarry. The limestone used in the study is obtained from the region of Sétif east of Algeria. The standardized sand for mortars confection, was used according to the European standard (NF EN 933-1, 1999; NF EN 933-2, 1999). The chemical composition of the various base materials used is shown in Table 1.

Table 1. Chemical composition of the materials used

	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	SO_3	K_2O	CL	Na_2O
Clinker(C)	21,38	4,25	65,55	5,32	1,72	0,58	0,35	0,005	0.35
Gypsium(G)	10.05	2.99	26.90	1.55	3.86	30.33	0.41	0.007	0.05
Pozzolana(P)	40.00	18.80	15.90	9.00	5.23	2.00	2.26	0.012	0.41
Slag(S)	29.00	11.30	46.00	1.35	8.12	1.98	0.48	0.047	0.67
Limestone(L)	15.20	2.34	78.70	1.73	1.04	0.56	0.32	0.003	0.15

The physical properties of the various materials used are presented in Table 2.

Table 2. Physical properties of materials used

Materials	Specific density (g/cm^3)	Bulk density (g/cm^3)	Fineness (cm^2/g)
Clinker (C)	3.22	1.04	3490
Gypsum (G)	2.50	0.91	3500
Pozzolana(P)	2.62	0.95	3900
Limestone(L)	2.68	0.97	3800
Slag (S)	2.79	0.98	3215

Experimental Procedures

Experimental design

We have chosen a mixture of three quantitative factors (A, B and C) which represent three mineral additions (Pozzolana, Limestone and slag), the delimitation of the range of variation of the chosen factors is limited to a percentage maximum level of 35% in the mixtures as replacement of a CEMII-B cement. Clinker and gypsum remain unchanged at 65 % amount. Thus, a plan of mixture of types I (plan network) is considered in such case, according to the following construction (Cohen, 1989).

Table 3.Construction of the experiment plan

Factors	Name	Unit	Type	Minimum	Maximum
A	Pozzolana (P)	%	Mixture	0	35
B	Limestone (L)	%	Mixture	0	35
C	Slag(S)	%	Mixture	0	35
				Total =A+B+C = 35 %	

Mechanical responses

The mechanical properties are to be measured for the variation of the factors mainly, the compressive strength of the mortars tested at the age of 2, 7, 28 and 60 days and designated as R_{C2}, R_{c7}, R_{c28} , and R_{c60}

Mixtures Plan

In order to optimize the number of tests, and consequently the number of mixtures to be prepared, which correctly meet our expectations, we used a practical and computerized statistical approach called "experimental design", carried out using software (Expert Design). the number and the composition of the mixtures are chosen by the experimental plan with arrangement in convenience to our purpose (Table 4).

Table 4.Composition and designation of prepared cements (% by weight of cement).

Designation of cement		Composition of cement mixtures (%)				
Type of cement	Name Mix	Clinker (C)	Gypsium (G)	Pozzolana (P)	Limestone (L)	Slag (S)
Binary	CB1	60	5	35	0	0
	CB2	60	5	0	35	0
	CB3	60	5	0	0	35
Ternary	CT1	60	5	7	0	28
	CT2	60	5	0	7	28
	CT3	60	5	0	28	7
	CT4	60	5	28	7	0
	CT5	60	5	28	0	7
	CT6	60	5	7	28	0
	CT7	60	5	21	14	0
	CT8	60	5	0	21	14
	CT9	60	5	0	14	21
	CT10	60	5	21	0	14
	CT11	60	5	14	0	21
	CT12	60	5	14	21	0
Quaternary	CQ1	60	5	21	7	7
	CQ2	60	5	7	21	7
	CQ3	60	5	7	7	21
	CQ4	60	5	14	14	7
	CQ5	60	5	14	7	14
	CQ6	60	5	7	14	14
	CQ7	60	5	12	12	12

Results and Discussion

Tests of the compressive strength measured at the age of 2.7,28 and 60 days, on prismatic test specimens (4 × 4 × 16) in accordance with European standard (EN 196-1, 2005) were prformed. The results are reported in table 5, below

Table 5. Results observed of the mechanical strengths of mortars tested at the age of 2.7,28 and 60 days

Type of cement	Designation (Mix)	Mineral additions %			Compressive strength			
		P	L	S	RC_{2J}	RC_{7J}	RC_{28J}	RC_{60J}
Binary	CB1	35	0	0	10.16	20.12	31.68	35.89
	CB2	0	35	0	9.83	20.32	28.07	29.99
	CB3	0	0	35	11.01	22.29	39.48	44.78
Ternary	CT1	7	0	28	11.68	24.23	37.81	46.64
	CT2	0	7	28	11.30	23.40	37.82	43.98
	CT3	0	28	7	9.93	20.54	31.03	39.56
	CT4	28	7	0	9.82	20.91	30.91	36.23
	CT5	28	0	7	10.12	21.02	33.58	37.36
	CT6	7	28	0	10.33	23.19	26.57	30.82
	CT7	21	14	0	11.18	23.79	34.13	38.49
	CT8	0	21	14	11.18	21.59	33.49	33.26
	CT9	0	14	21	12.55	24.33	34.95	41.49
	CT10	21	0	14	8.79	20.64	36.33	41.04
	CT11	14	0	21	11.58	21.68	35.27	39.44
	CT12	14	21	0	11.13	21.90	29.89	33.10
Quaternary	CQ1	21	7	7	9.80	20.38	34.51	36.36
	CQ2	7	21	7	11.47	22.87	35.08	34.08
	CQ3	7	7	21	10.37	24.20	36.18	42.46
	CQ4	14	14	7	10.59	22.85	35.11	39.30
	CQ5	14	7	14	10.44	21.83	34.92	43.58
	CQ6	7	14	14	10.55	23.54	36.80	43.00
	CQ7	12	12	12	10.88	24.54	35.55	39.57

The Early Compressive Strength at 2days

Through the analysis of Figure 1, which represents the variation of the compressive strength measured for the mortars studied, the best strength of 12,5 MPa is recorded for ternary composition (slag - limestone) with a dominant percentage due to slag of 21% compared to a rate of 14% for limestone, this is due to the positive interaction of slag and limestone, as long as the limestone acts by its nucleation and condensation role which improves the microstructure at a young age and on the other hand the early reactivity of the slag which positively affects the microstructure and improves the resistance, some authors report that with the addition of limestone, there is an increase in mechanical resistance at young age due to the accelerating effect and the filling effect; the filler effect of limestone. It should be noted that for a thinness of 300 to 350 m²/kg, only the filling effect is considered (Amouri, 2009).

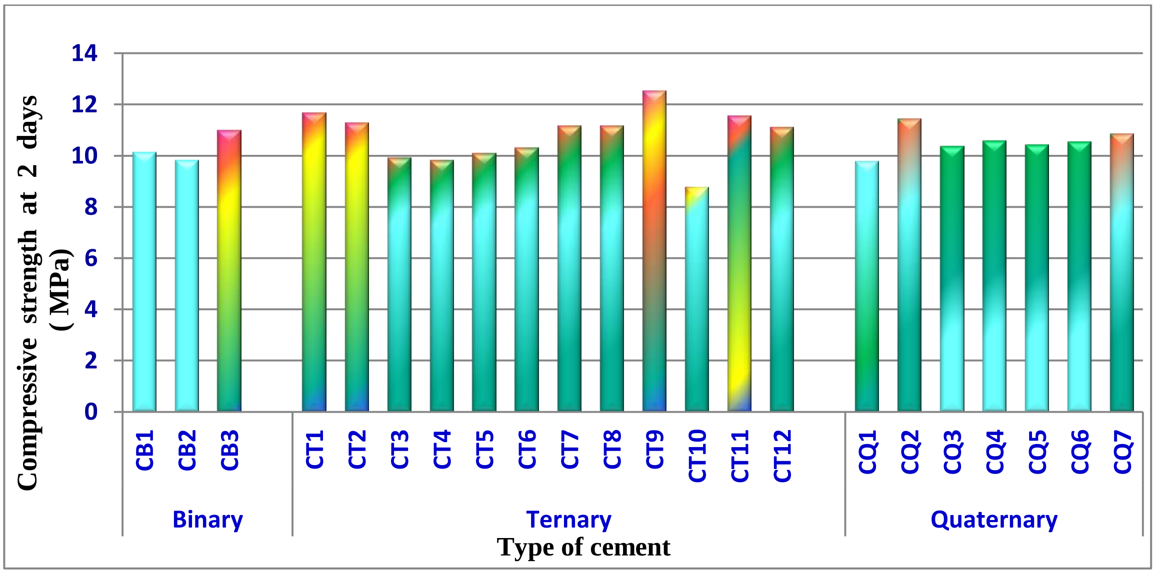

Figure 1. Variation of the compressive strength results of the mortars at the age of 2 days.

Nevertheless, the limestone becomes less beneficial beyond a rate of 14% and plays a detrimental role with a rate higher than 21% for all types of ternary and quaternary compositions. Noting that combinations (slag-pozzolane) are acceptable in all compositions. Overall the slag which remains the best addition that has a considerable effect at young age compared to pozzolana and limestone which are less beneficial in binary compositions with rates up to35%.

The Compressive strength results at 7 days

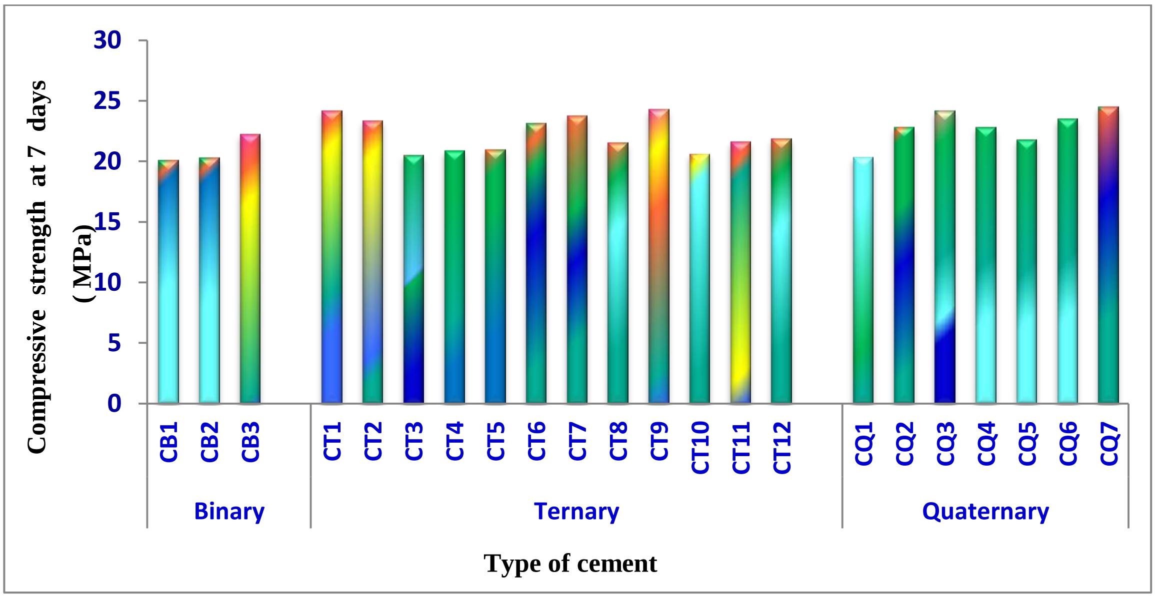

Figure 2. Variation of the compressive strength measured of the mortars at the age of 7 days.

The results of the compressive strength measured at the age of 7 days shown in Figure 2 show that the variation of the compressive resistances follows the same trend as that of the 2-day resistances, preserving the dominance of the slag effect, either in the binary compositions, or by double interaction with pozzolana or limestone with rates lower than 14% in ternary and quaternary compositions. Noting that the evolution of resistance is important and resistances are evolved from 100 to 80% compared to resistances at the age of 2 days.

The Compressive Strength Results at 28 Days

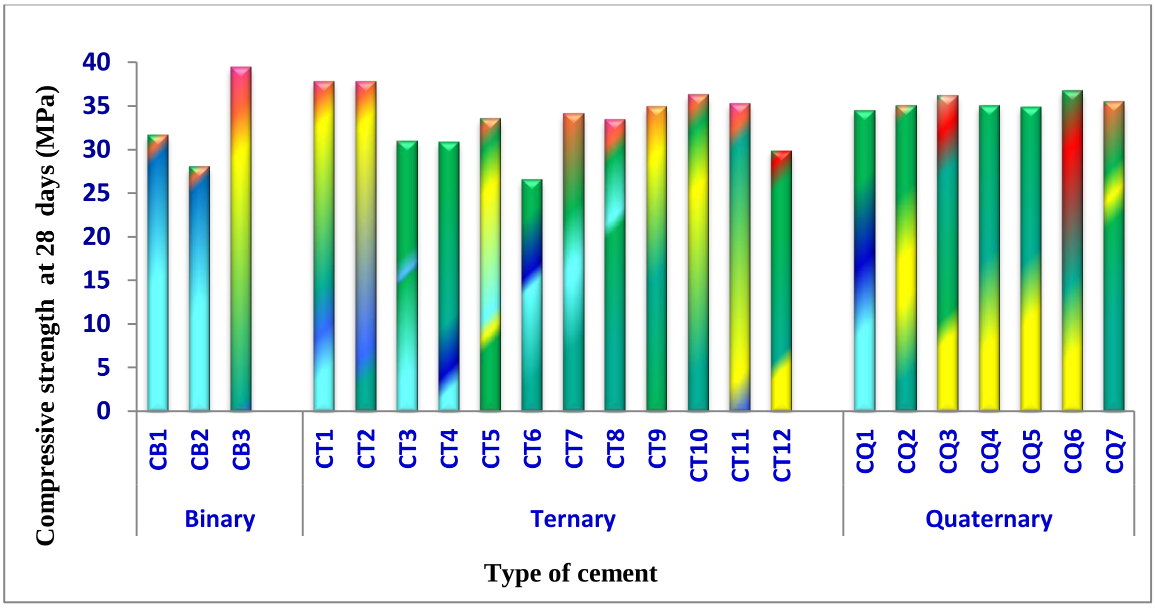

Figure 3. Variation of the compressive strength measured of the mortars at the age of 28 days.

Figure 3, which represents the variation of the compressive strength measured at 28 days, shows the best resistance of 39.48 MPa for binary composition with 35% of the slag replacement. This shows that the reactivity of the slag is in its advanced state for better participation in the formation of hydrates and their beneficial arrangement in the microstructure. Typically, between 7 and 28 days, resistance–is similat to that of control (Tokyay, 2016).

On the other hand, the lowest resistances are recorded for limestone with a rate of 35% for binary composition and 21 and 28% for ternary compositions with values of (28.07, 26.57 and 29.89 Mpa) respectively, which represents values below the guaranteed strength for cement II/B type.It should be noted that the reactivity of pozzolana begins to increase at the age of 28 days and manifests binding properties (Miller, 1993). . Noting the significant effect of the interaction (pozzolane-limestone) with limestone levels less than 21% and the effect of the double interaction (pozzolana-Slag) which sows better performances for all compositions**.** For the quaternary compositions it is noted that all the studied combinations ensure a guaranteed resistance greater than 30 MPa.

The Compressive Strength Results at 60 Days

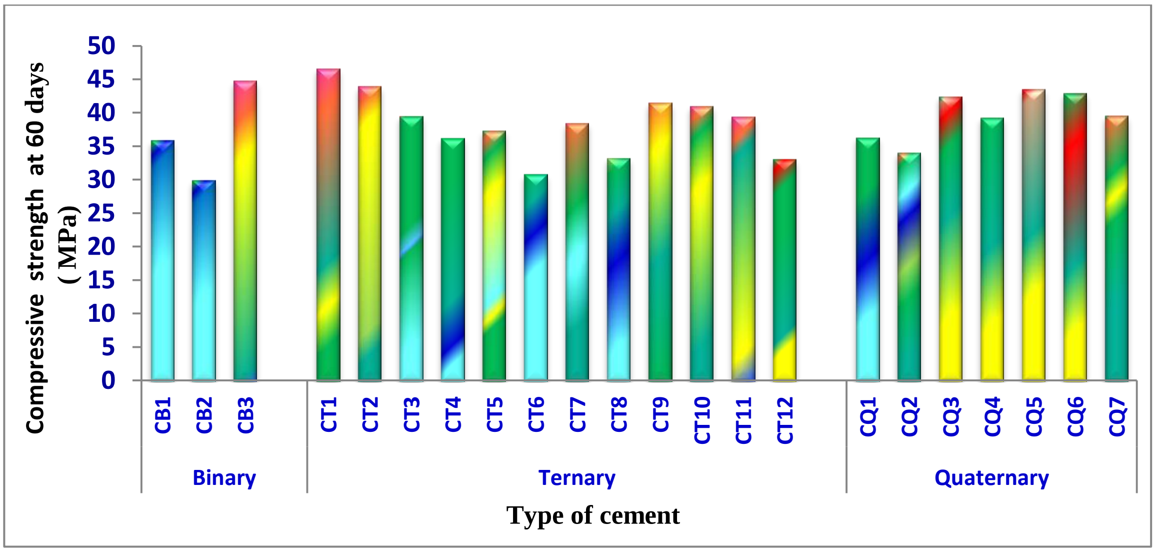

Figure 4. Variation of the compressive strength measured of the mortars at the age of 60 days.

Through the analysis of Figure 4, which represents the variation in compressive strength measured for the mortars studied at age 60 days, the best resistance values of 44.78 and 46.64 Mpa are recorded respectively for a binary composition of 35% of the slag and a ternary composition (slag – pozzolane) with a dominant percentage of the slag of 28% compared to a rate of 7% for the pozzolane, this is due to the positive interaction of the (Pouzzolane-slag), which reflected the reactivity of the two additions and the maturity of the microstructure. This is the case for a study (Kerbouche *et al.*, 2009) which concluded that the natural beni-saf pozzolane contributes positively in the long term.

At the age of 60 days the evolution is less important, nevertheless that all the registered resistances exceed 30 MPa which represents the threshold of the resistance guaranteed for a Cement II/B, with the exception of the binary composition with 35% of the limestone which remains below the guaranteed resistance.

Modeling Of The Compressive Strength

Model Summary Statistics for Compressive strength

The modeling of the mechanical behavior relative to the results measured by the experiment program consists in the choice of a model which represents a trend in the curve with a correlative coefficient of (R^2) better and close to 1. This reflects good convergence with minimal deviations between the results predicted by the model and those measured experimentally. The statistical results are reported in the following Table 6.

Table 6. Model Summary Statistics for Compressive strength

Model	R-Squared (R^2)			
	$\mathbf{RC_{2j}}$	$\mathbf{R_{C7j}}$	$\mathbf{RC_{28j}}$	$\mathbf{RC_{28j}}$
Linear	0.252	0.226	0.755	0.704
Quadratic	0.43	0.575	0.843	0.744
Special Cubic	0.483	0.576	0.868	0.754
Cubic	0.599	0.731	0.893	0.780

Modeling of the Compressive Strength at 2 Days

Table 7. Equation of the Compressive strength model at 2 days of age (Équation 1)

$\mathbf{RC_{2j}}$ =	0.29	* P
	+ 0.27	* L
	+ 0.316	* S
	+ 0.00399	* P * L
	- 0.000757	* P * S
	+ 0.0042	* L * S
	- 0.000428	* P * L * S
	- 0.000118	* P * L * (P-L)
	- 0.000177	* P * S * (P-S)
	- 1.76E-05	* L * S * (L-S)

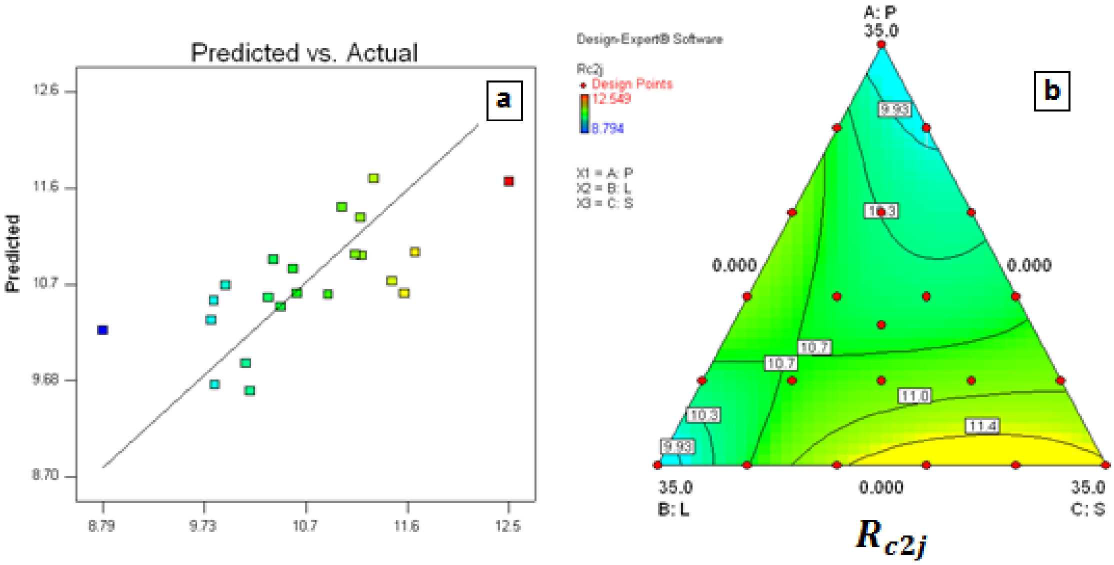

Figure 5. Compressive strength at 2 days a) Correlation between observed values and the model, b) Variation of compressive strength according to the model.

The suggested model records a coefficient of determination (R^2 = 0.599) for the cubic trend curve Figure 2. (a), but this coefficient remains unsatisfactory and the suggested model remains unrepresentative, no good correlation between the expected results and those actually achieved and which are very distant, with very large differences, given these results, in this case we cannot adopt this modelling which proves to be unsatisfactory.

Based on the results presented in a triangular diagram (Figure 5). It is noted that the 2-day compressive strength is relatively greater for binary cement with slag and ternary and quaternary cement with slag dominance, which is decisive in short-term resistance compared to the lowest results recorded at the level of binary cements; limestone and pozzolane). Since the correlation coefficient is quite low, the interpretation and analysis of the results at this age of 2 days can only be considered as indicative and not predictive.

Modeling of the Compressive Strength (7 Days)

Table 8. Equation of the Compressive strength model at 7 days of age (Équation 2)

RC_{7j} =	0.586	* P
	+0.578	* L
	+0.64	* S
	+0.00833	* P * L
	+0.000584	* P * S
	+0.00517	* L * S
	+0.00012	* P * L * S
	-0.000221	* P * L * (P-L)
	-0.00029	* P * S * (P-S)
	-0.000247	* L * S * (L-S)

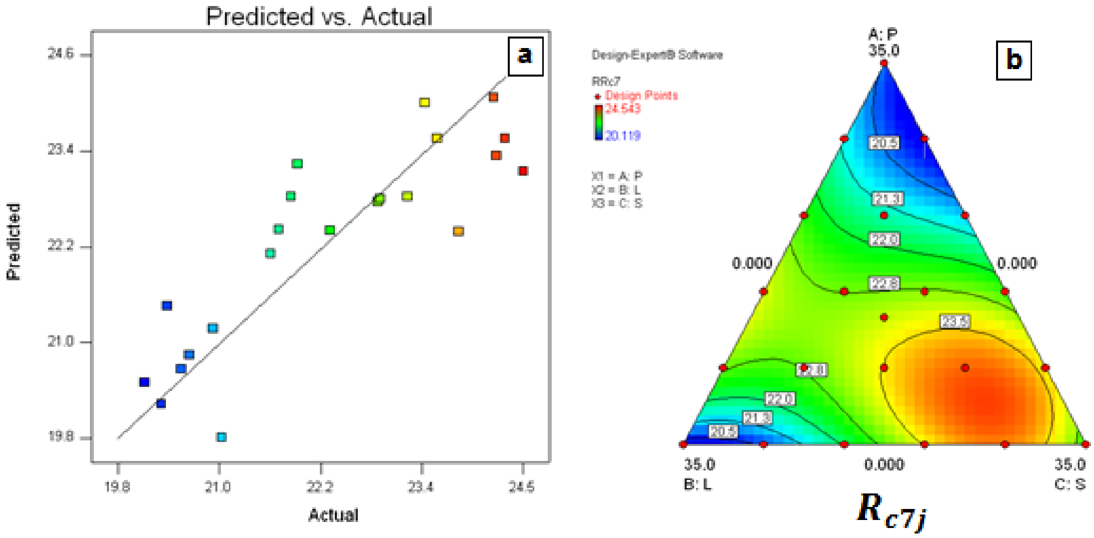

Figure 6- Compressive strength at 7 days: a) Correlation between observed values and the model, b) Variation of compressive strength according to the model.

The proposed model is a cubic model that takes into consideration the triple order interactions with side effects, this model is represented by equation 2, with an adjusted trend curve with a satisfactory coefficient of determination R^2 = 0,731 Figure 6 (a), which reflects a good correlation between expected and observed results with non-significant differences, which supports the effectiveness of the model which can be adopted with satisfaction.

Figure 6 (b) explains the variation in compressive strength at the age 7 days for the results obtained and expected. It is noted that through the predictions of the model it is possible to develop better values of compressive strength for quaternary cements with slag dominance. For binary compositions, pozzolana and limestone do not develop as good resistance to young age compared to Slag. The interaction is better between the (slag-pozzolana) and (slag-limestone) with low levels of pozzolana and limestone.

Modeling of the Compressive Strength (28 Days)

Table 9. Equation of the compressive strength model at 28 days of age (Équation 3)

RC_{28j} =	0.916	* P
	+0.781	* L
	+1.11	* S
	+0.00477	* P * L
	-0.000179	* P * S
	+0.00503	* L * S
	+0.00114	* P * L * S

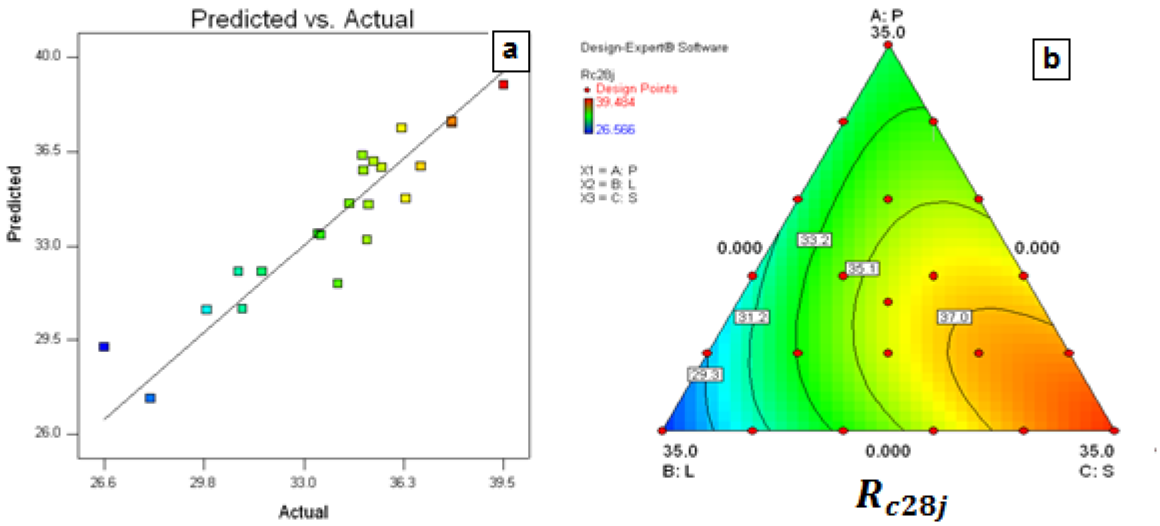

Figure 7. Compressive strength at 28 days : a) Correlation between observed values and the model, b) Variation of compressive strength according to the model.

The model proposed and represented by Equation 3 is a cubic model, which is limited to the consideration of triple simple order interactions (P-L-S). This model shows a satisfactory correlation between the expected results and those actually obtained illustrated by a trend curve Figure 7 (a) with a coefficient of determination (R^2 = 0.868), the model can be adopted with good satisfaction.

Figure 7 (b) shows the variation of compressive strength at the age of 28 days for the results obtained and those expected. The analysis of the expected and proposed results of the model is similar to the analysis made for the actually measured results, it is noted that the resistance becomes very important around the top (c) corresponding to the slag addition, which develops the best resistance the case of a binary cement and a significant interaction with limestone and pozzolane where the slag is dominant the case of ternary or quaternary cements, which explains the significance of the effect of the slag at this age. In addition, it can be noted that resistance in the vicinity of the pozzolane is acceptable for binary, ternary and quaternary cements. On the other hand, near the limestone tip, low resistance develops for ternary and quaternary cements, in particular a resistance lower than the resistance guaranteed for an CEM II/B in the case of binary cement with limestone.

Modeling of the Compressive Strength (60 Days)

The suggested model for 60-day compressive strength is represented by equation 4, it is a specific cubic model, which takes into account triple interactions, this model shows a satisfactory correlation illustrated by a trend curve Figure 8 (a) with a coefficient of determination (R^2=0.754), the model can be adopted with good satisfaction. Figure 8 (b) shows the variation in compressive strength at the age 60 days for both the results obtained and those expected. The analysis of the expected and proposed results of the model is similar to the analysis made for the actually measured results.

Similarly, the reading of the results and the analysis of the variation of the compressive strength at the age of 60 days, the significance of the effects of each addition as well as the double and triple interaction between different additions, remains similar to reading done for compression resistance at the age of 28 days with a regular and less important evolution of resistance at the age of 60 days.

Table 10. Equation of the Compressive strength model at 60 days of age (Équation 4)

RC_{60j} =	1.03	* P
	+0.865	* L
	+1.31	* S
	+0.00538	* P * L
	+4.16E-05	* P * S
	+0.00415	* L * S
	+0.00107	* P * L * S

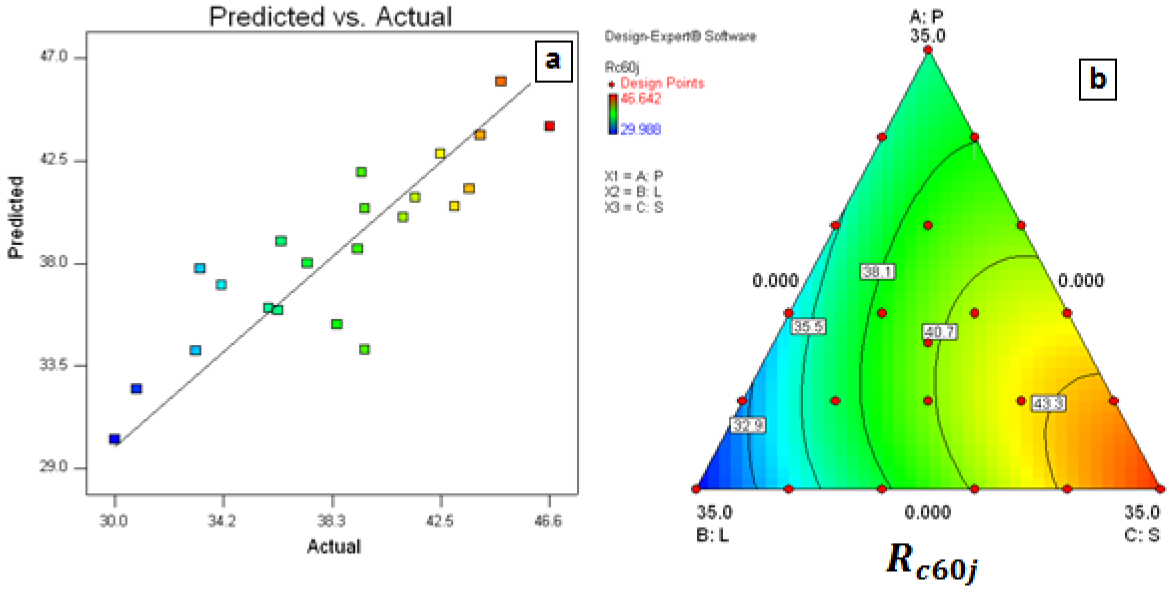

Figure 8. Compressive strength at 60 days a) Correlation between observed values and the model, b) Variation of compressive strength according to the model.

Conclusion

The results obtained from this study allow the following conclusions to be drawn:

The experimental design analysis could lead to a modelling which would give a relatively acceptable satisfaction to the analytical design and the explanation of the phenomenon studied in this case the mechanical response particularly in terms of estimation of the effects of the additions and the quantification of interactions, thus the delimitation and localization of the zones of amplification of the resistance which allows a good understanding of the mechanical behavior of a quaternary and ternary cement which resides complex without this tool.

With regard to the prediction of the results, it is found that the models estimated values close to those of the experiment, in particular for the characteristic compressive strength at 28 days, when the model was very close to the real phenomenon with very small differences.

However, it cannot be denied that there is a divergence in the results expected by the model, resulting in large differences and a very low coefficient of determination in the case of the 2-day compressive strength, This leads us to carry out complementary experiments for the refinement of the results.

This confrontation allowed us to estimate with tolerance the degree of conformity of these models with the real phenomenon (the mechanical response) of a quaternary cement, ternary and binary compositions. Thus, the model obtained will always be imperfect but it does not prevent that it can serve as an indicator in terms of analysis and prediction.

Finally, it could be concluded all compositions belonging to the field of study limited to a rate of addition of 35%, are acceptable in regards to mechanical performances with the exception made for the compositions with limestone percentage higher than 21% that show lower strength in comparison with almost tested mixtures.

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Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

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