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A Self-Compacting Cement Paste Formulation using Mixture Design

A. Mebrouki, N. Belas, K. Bendani and N. Bouhamou
Department of Civil Engineering, University of Mostaganem, Algeria

Abstract: This study presents a self compacting cement paste formulation using Algerian local materials (binary cement consisting of natural pozzolana and limestone fillers). In this study, simple laboratory instruments were used i.e., mini-slump for spreading out diameters and Marsh cone for flow times measurements. A wide variation of combinations was used as preliminary tests to select pastes with acceptable properties and the use of the mixture plans method has shown that it is possible to define an experimental field inside which optimal measurements can be obtained. This field has been put mathematically into equation form conditioned by implicit constraints, defining zones of minimal shearing threshold and maximum viscosity and was then solved numerically. The optimization criterion was checked in addition to the interactivity between components utilizing the multiple combinations of proportioning of these materials. From results given by ternary diagrams and desirability functions, an optimal self-compacting cement paste mixture was defined. Experimental checking was performed to validate the obtained results.

Key words: Mixture plans method, limestone fillers, superplasticizer, ternary diagrams, Algerian local materials

INTRODUCTION

Concrete is a composite material which consists essentially of (a) fluid phase called the cement paste and (b) solid phase of aggregates with fixed gravel/sand ratio. Self-Compacting Concrete (SCC) requires both high fluidity to flow under the effect of gravity alone and a good resistance to segregation. Since, controlling the flow properties of cement paste can lead to the control of self-compacting abilities at full concrete scale (Saak *et al.*, 2001; El Barrak *et al.*, 2009), it is necessary to understand the influence of each constituent on the flow behavior at paste scale. Many researchers have studied the influence of constituents on the flow properties of cementitious mixtures (paste, mortar and concrete) using statistical models (Violeta and Yachko, 2005). These approaches make it possible to highlight dominant parameters and to optimize the mixture formulation. When mixtures are optimized on quantitative basis, depending on the objective of the optimization, construction productivity could be improved, durability increased, and both material and construction costs reduced (Yeh, 2007). A multi-objective optimization of cementitious blends of mineral admixtures was studied to achieve an optimal mixture concrete proportioning (Gesoğlu *et al.*, 2009), which means that use of multi-scales optimization avoid an extensive series of tests. This leads to reduction of the costs in product development.

The self-compacting properties of the concrete depend necessarily on those of the cement paste that is why the study carried out on formulations is based primarily on the paste and its different components. The method used on materials from the Building Materials Laboratory (L.M.D.C - INSA-UPS, Toulouse, France) gave very satisfactory results, and it was then applied to Algerian local materials in order to obtain a self compacting cement paste formulation. The experiments were carried out using simple equipments which can be afforded by laboratories of moderate budget.

Various combinations of paste mix parameters has been adopted using mini-cone and Marsh cone measurements, which has allowed to eliminate all undesired mixtures presenting segregation or a lack of capacity to flow or poorly proportioned. The remainder of the combinations was used as a data base to define an experimental field. From measurement of the spreading out diameters and out-flow times, ternary diagrams can be produced in order to delimit zones of low shear threshold and high viscosity. Inside these zones, volumetric proportions of the paste components were retained and then treated numerically to obtain an optimal paste mix satisfying the self-compacting properties criteria.

Corresponding Author: Karim Bendani, Coopérative Seddik Benyahia, No. 28, Chemouma, 27000 Mostaganem, Algeria
Tel: 00213 71 72 38 58 Fax: 00213 45 33 91 57

MATERIALS AND METHODS

The choice of materials is based on their abundant availability and their moderate cost. Cement used is a 'CEM II/A 32.5 N' which according to European standard is ENV 197-1, containing less than 20% of natural pozzolana added during clinker crushing and fillers consisting of limestone, obtained from the west Algerian quarry. It must be mentioned here, that the major part of experiments and chemical analysis have been performed at Building Materials Laboratory of L.M.D.C - INSA-UPS, Toulouse, France and at Mostaganem University, Algeria in the period 2007/2008. The physical properties of these two powders are shown in Table 1.

The cement used contains about 15% natural pozzolana. The results of chemical analysis of this cement are given in Table 2.

The mineralogical composition of Clinker according to Bogue is given in Table 3.

The aspects of limestone fillers and cement are shown in Fig. 1a and b.

The chemical analysis of limestone fillers is shown in Table 4.

Figure 2 shows filler mineralogical analysis, obtained by x-ray diffraction (XRD).

This analysis showed a composition of about 97% calcite, with traces of dolomite and quartz.

The superplasticizer used in this study is 'Viscocrete 20 HE' provided by SIKA-Algeria. It is a no chlorinated product containing acrylic copolymer in liquid form and containing 40% of dry extract with a density of 1,085 kg m^{-3} and a pH of 4,5. It can be shown from several studies (Youjun *et al*., 2002; Naadia *et al*., 2004) that it is possible to prepare self-compacting concretes without using viscosity agent to remain in the context of local materials promotion, so this parameter will not be integrated in the preparation of the cement pastes. For measurements, a mini-cone inspired from the slump test was used and whose dimensions are proportional to it (Kantro, 1980; Cyr, 1999; Roussel *et al*., 2005) (Fig. 3).

The mini-slump cone has a bottom diameter of 38 mm, a top diameter of 19 mm, and a height of 57 mm.

This apparatus will be used primarily for the determination of the spreading out diameters on a horizontal metal plate with respect to the mix parameters variation (water/binder, limestone/binder ratios and superplasticizer). These diameters were measured after 1 min of spreading out, and the same procedure was applied for all the other mixtures. Generally, there is a certain correlation of the test with the threshold of

Table 1: Physical characteristics of cement and limestone filler

	Density (kg m^{-1})	Specific surfaces (cm^3 g^{-1})	Average diamèter (µm)
Cement	3150	3400	18, 5
Limestone	2800	2880	21, 2

Table 2: Cement chemical analysis

SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	Fe_2O_3	SO_3	CaO_{free}	Fire loss
23	5.7	60.9	0.7	0.3	0.4	3.3	3.4	0.09	2.1

Table 3: Clinker mineralogical analysis

C_3S	C_2S	C_3A	C_4AF
58,7	16,4	8,1	9,2

Table 4: Chemical analysis of used limestone fillers

SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	Fe_2O_3	Fire loss
0,7	0,2	56,8	0,5	0,08	0,1	0,9	41,2

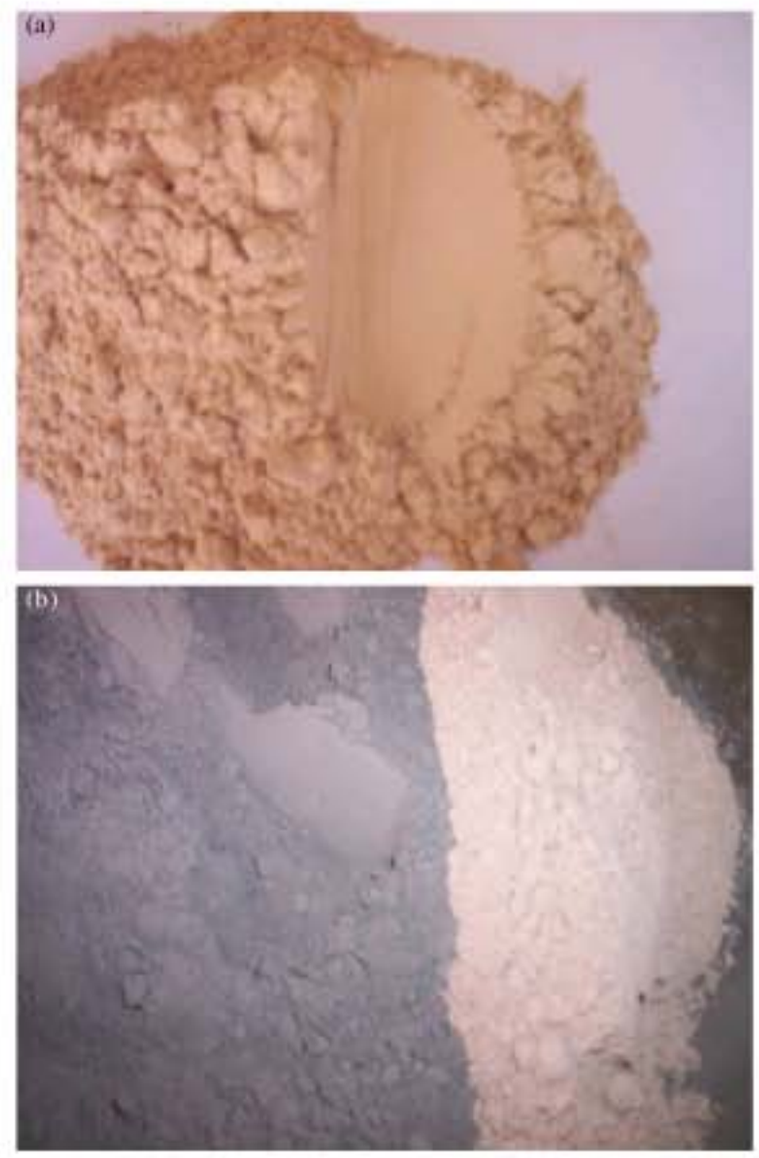

Fig. 1: Aspects of limestone fillers and cement. (a) limestone fillers and (b) Fillers in substitution into cement

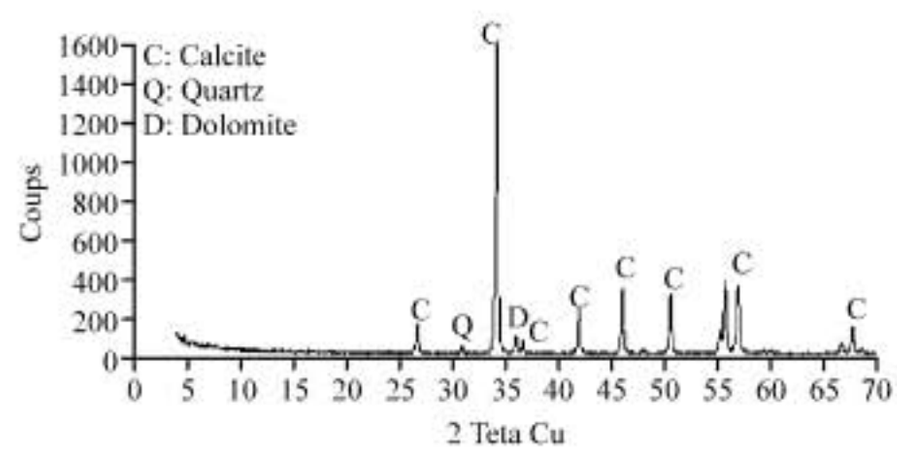

Fig. 2: Limestone filler's mineralogical analysis (XRD)

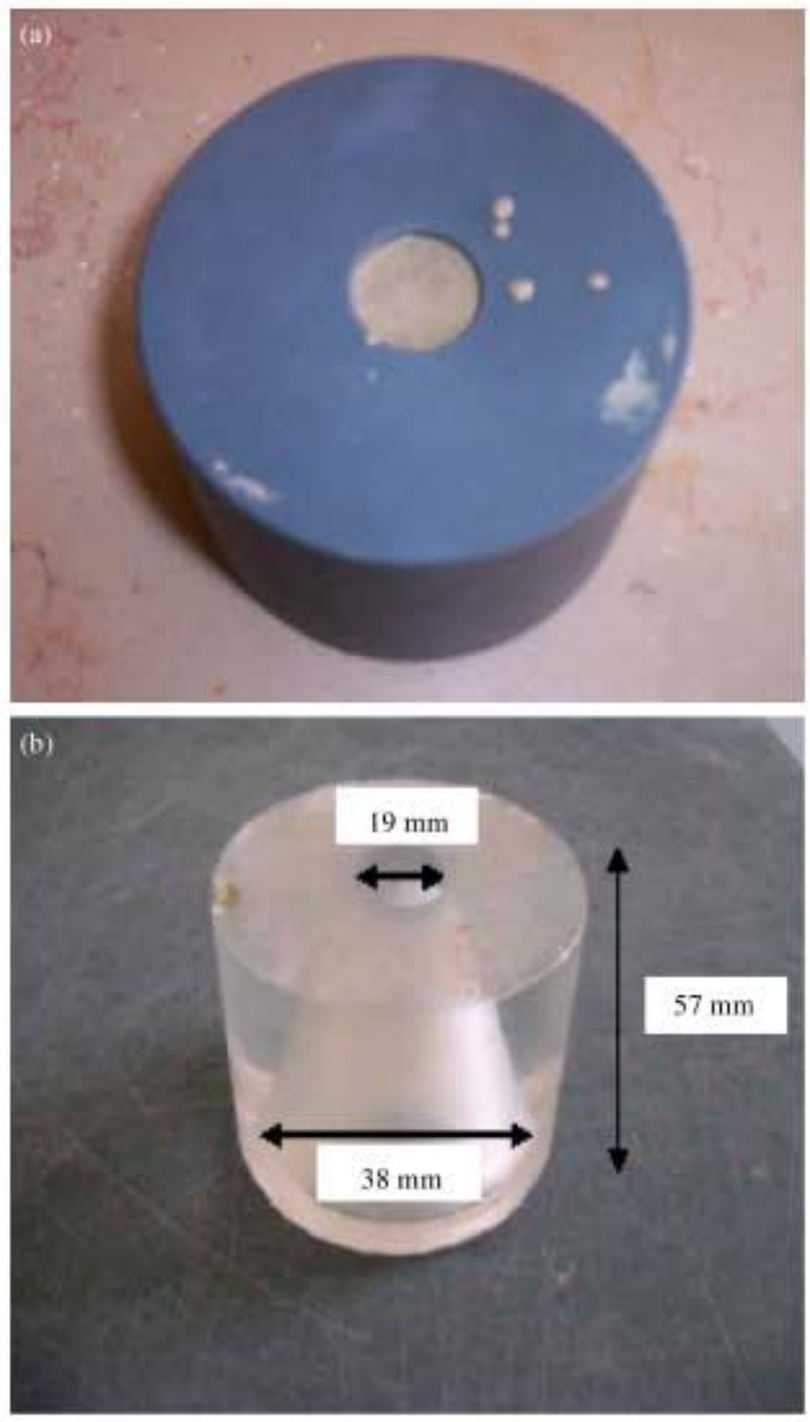

Fig. 3: (a, b) Mini cone

shearing or with apparent viscosity at low velocity gradient (Cyr, 1999), the mini-slump test results correlate in certain cases with the yield stress (Ferraris *et al*., 2001).

The main advantages of this test are the facility of its implementation since it requires simple preparation and small quantity of materials (volume less than 40 mL). America for the determination of superplasticizer saturation point of a cementing mixture (Cyr, 1999). It has been shown that the paste rheology model is useful to SCC mix design and reducing the laboratory work testing time and materials used (Bui *et al*., 2002). It would be more interesting to investigate the flow and workability of the concrete by studying the cement paste which is the main component responsible for these properties (Phan and Chaouche, 2005).

For consistency determination, a Marsh cone (Fig. 4) was used to measure the out-flow times of a reference volume of pastes with different mixtures. It is therefore the measure of the out-flow time of the cement paste flowing

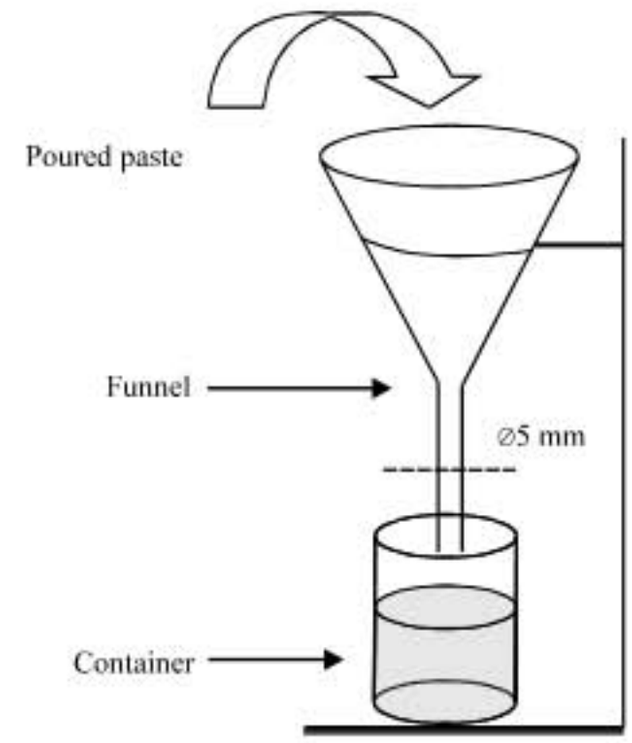

Fig. 4: Marsh cone

Table 5: Compositions of the studied pastes

Composition (%)	Values
Cement	100-95-90-85-80-75-70-60
Limestone in substitution to the cement	0-5-10-15-20-25-30-40
Water/Binder (E/L)	0.22-0.24-0.30-0.40
Superplasticizer (Sp)	0, 0.5, 1, 1.2, 1.3, 1.5, 2 and 3

out through the cone by gravity to fill up a given reference volume i.e., 150 mL. Since the tested volume was small, the test was simple and short in duration (Roussel *et al*., 2005). The rheological properties of the concrete (Steel Fiber Reinforced Self Compacting Concrete) can be deduced from those of the paste which constitutes it (Ferrara *et al*., 2007).

Sonebi and McKendry (2008) have concluded in their study that the Marsh cone with an orifice of 5 mm was not suitable to measure the out flow time and so, bigger orifices of 8 or 10 mm may be used.

The time required for a paste sample to flow through the cone is proportional to the paste viscosity. The flow time increases with the increase in viscosity, therefore, it will be considered as an index of fluidity.

Table 5 presents various pastes mixtures (mass proportions) for which spreading out and out-flow times measurements were carried out. The compositions shown below, as broad as they are, take into account all possible mix parameters variations which can contribute to elaborate the cement pastes. The quantity of the binder (cement+filler) was maintained constant and for superplasticizer, a maximum proportioning of 3% was recommended by manufacturer.

The substitution percentage is calculated in mass terms.

The experimental procedure used for pastes preparation is shown in Table 6.

Table 6: Experimental procedure of preparation of a standard paste mixture

Step	Moment	Duration	Measured parameter	Result
Materials Preparation and weighing	-	10 min	Materials masses	Components proportion
Materials Mixture	-	5 min	-	-
Visual aspect with the trowel	-	During mixing	-	Paste aspect
Measure with the min-cone	t_0: end of mixing	2 min	Flow	Spreading out (cm)
Visual aspect with glass tube	t_0	5 min In parallel with the previous one	-	Consistency/sedimentation
Measure with Marsh cone.	t_0 + 2 min	1 min	Flow time	Time (sec)

Fig. 5: (a-d) Visual aspects of the elaborate pastes

RESULTS

The selected results are based on the standard deviations calculated for a chosen paste mixture and was repeated three times under the same experimental conditions. The standard deviation will have the same value for all mixtures.

Consistency: Visual aspect: The visual aspect is a preliminary but very important stage, which allows checking the validity of the mixture visually. Paste mixture can be dry or very firm state (Fig. 5a), when it is made with insufficient water/binder ratio (Water/Binder = 0.22) and with an important filler substitution quantity (F = 30%). In Fig. 5b, it can be shown that the paste formed was plastic and not capable to flow (F = 10%, W/B = 0.23 and Sp = 1%). Contrary to the previous case, it was noticed that a paste can be capable to flow but presents a white layer (F = 15%, Water/Binder = 0.24 and Sp = 1.5%) (Fig. 5c), which is synonymous of segregation between the solid and the liquid phases of the paste. For other cases, the paste was well formed but segregation was noticed at the time of the measurement of the spreading out diameter: bubbles appeared on the surface of the wafer with a liquid halo around it. Figure 5d shows a well formed and homogeneous wafer, which was kept to measure the spreading out diameters and the out-flow time (F = 15%, Water/Binder = 0.24 and Sp = 1.2%). The same procedure was followed during the experiment and only mixtures without any abnormality were retained for measurements.

Experimental plans for cement pastes

Experimental field: Several mixtures of normal consistencies were useful for rheological measurements and have contributed to delimit the experimental field inside which the measurements give the required results. This concerns a spreading out diameter within the interval (14.4-16 mm) in accordance to what was found in the literature (El-Barrak *et al*., 2004) and in

accordance to a flow without rupture of paste volume, which is a characteristic of good fluidity.

The parametric analysis allows the understanding of the influence of each mix parameter on the fluid suspensions and on the prepared paste. However, an important parameter was introduced, it is the solid volumetric concentration (Γ) defined by the ratio of volume of solids on total volume (solid particles coming from cement, limestone fillers and superplasticizer in dry extract form). The use of the experimental plans method contributes to collect maximum information about components, their influences taken separately and about their possible interactions. It makes possible to reduce considerably the number of experiments, to plan and facilitate the study. The main objective of the study is then to obtain mixtures having optimal responses, or satisfying certain requirements fixed on departure (Mathieu *et al*., 2000). To achieve this, a wide variation of combinations between mixing parameters was used as preliminary tests to select pastes with acceptable characteristics and the use of the experimental plans method showed that it is possible to delimit an experimental field bounded by the volumetric proportions of materials composing the paste. The field was transformed mathematically into equations conditioned by implicit constraints, defining zones of minimal shearing threshold and maximum viscosity. The required response depends on the volumetric proportions of the components used. Thus, for an experimental plan with 4 factors C (cement), F (limestone filler), W (water) and Sp (superplasticizer)] taken in volumetric proportions, with a total volume equal to unity, implies that there is a dependence and an interaction between each component. The experimental field was constrained by the following expression:

$$C + F + w + Sp = 1 \tag{1}$$

Considering a complete mixture plan, taking account of the required accuracy of the response and the number of admitted experiments, the choice of a mathematical model has converged towards a polynomial of degree 2, relating the response Y (Y1 for spreading out diameter or Y2 for out-flow time) to the proportions of the components, which can be written in the following form:

$$Y = \sum_{i=j}^{k} \beta_i \times X_j + \sum_{i<j}^{k}\sum^{k} \beta_{ij} \times X_i \times X_j \tag{2}$$

The polynomial coefficients β_i and β_{ij} are to be determined and are expected to be different for each response. Parameters X_i and X_j correspond to the volumetric proportions of the components. Equation 2 can be expanded to give:

$$\begin{aligned} Y = {} & \beta_1.C + \beta_2.F + \beta_3.W + \beta_4.Sp + \beta_{12}.C.F + \beta_{13}.C.W \\ & + \beta_{23}.F.W + \beta_{14}.C.Sp + \beta_{24}.F.Sp + \beta_{34}.F.Sp \end{aligned} \tag{3}$$

Equation 3 can be rewritten in matrix form as follows:

$$[Y] = [X].[\beta] + [\varepsilon] \tag{4}$$

where, [X] is the experiment matrix, [β] is the vector of the model coefficients and [ε] the vector of the experimental errors.

After preliminary tests, a parametric analysis was used to define zones which are checked at the same time for a high viscosity and a minimal shearing threshold and also to define the experimental field bounded by the lower and higher constraints (in mass proportion) given as follows:

$$\begin{aligned} 10\% \le \text{limestone} \le 20\% \\ 1\% \le \text{Superplasticizer} \le 1.5\% \\ 0.24\% \le \text{water ratio} \le 0.3\% \\ 0.57\% \le \Gamma \le 0.59\ \% \end{aligned} \tag{5}$$

where, (Γ) is the volumetric concentration in solids.

The transformation of these constraints to a system of Eq. 6 allowed modeling the experimental problem which can be solved numerically.

$$\left.\begin{aligned} C + F + W + SP &= 1 \\ -0.1125\ C + F &\ge 0 \\ 0.2250\ C - F &\ge 0 \\ -0.0290\ C - 0.0258\ F + Sp &\ge 0 \\ 0.0435\ C + 0.0387\ F - Sp &\ge 0 \\ 0.43\ C + 0.43\ F - 0.17\ Sp - 0.57\ W &\ge 0 \end{aligned}\right\} \tag{6}$$

Here, a numerical example is presented to show the way of obtaining the equations system.

$$\text{(Limestone's mass proportion)} \le 20\% \Rightarrow \frac{MF}{MC} \le 0.2 \quad \Rightarrow \frac{MF}{MC} - 0.2 \le 0$$

$$\frac{VF}{VC} = \frac{\frac{MF}{2800}}{\frac{MC}{3150}} = \frac{MF}{MC} \times \frac{3150}{2800} \rightarrow \frac{MF}{MC} = \frac{VF}{VC} \times \frac{2800}{3150} = \frac{VF}{VC} \times 0.889$$

$$\frac{VF}{VC} \times 0.889 - 0.2 \le 0 \Rightarrow VF - 0.225 \times VC \le 0 \ \Rightarrow \ -VF + 0.225 \times VC \ge 0$$

Which gives 0.225×C-F≥0, this represents the third equation of system (6).

Where VF and VC are limestone's filler and cement volume proportions in the paste, respectively, MF and MC are limestone's filler and cement masses, respectively.

Table 7 gives the solutions computed by excess and default and are illustrated in the form of higher and lower constraints, respectively

For an experimental mix plan with 4 factors, the field formed was a space with 4 dimensions. The model calculation points and the obtained experiment matrix have produced a geometric form of a hyper polyhedral. These points are located at the tops, at the mid-sides, at the middle of the faces and at the gravity centre. An analytical solution for this complex problem is almost impossible, numerous solutions were obtained by using softwares for experimental mixture plans processing and the one used in this study among others is called NemrodW, developed for the design and the analysis of experimental plan.

Experiment matrix: The determination of the experiment matrix was carried out by the analysis of the exchange algorithm generated by the software, which is a procedure, applied to N = 10 as a number of variables (number of polynomial coefficients) until $N = N_{max}$ satisfying the following optimization criterion:

- **Criterion D:** Optimization of information quality
- **Criterion A:** Optimization of model coefficients quality
- **Criterion G:** Optimization of model predicting quality

Once determined, the basic matrix was used to calculate the model polynomial coefficients which will be different for each response. Table 8 gives the necessary information about experiment matrix and the main characteristics of the studied problem generated by the software.

The characteristics of the experiment matrix for volumetric proportions of the components to be used in the preparation of the cement pastes are shown in Table 9. These values were generated by the software and taken inside the experimental field.

The volumetric values of composition parameters provided by the software were used to prepare the pastes and followed by measurements. In order to define the optimal proportion values of cement, limestone fillers, superplasticizer and water, the measured responses of spreading out diameters and out-flow times were again input into the software as new data. Figure 6 shows ternary diagrams in space and in the plan illustrating the influence of each parameter on the paste mixture. Indeed, by fixing one parameter and while varying the three others, their sum should always be equal to unity. For example, if the water parameter is fixed at 0.305 and while varying the volumetric proportions of the others components, the parameter Sp will be dominating i.e., responses are more sensitive to the variations of this parameter than to those of "cement" or those of the filler. There would be then interactions between "Sp" and cement according to the site of the influence field which is closer to cement than to filler (Fig. 6a). It should be noted here, that the same work has been carried out for the out-flow time response.

Experimental responses: Figure 7a and b show the curves obtained for $\Gamma = 0.58$ at the end of processing. Figure 7a shows that substitution of 15% of limestone fillers decreases the shearing threshold, whereas Fig. 7b shows that a proportioning of 1.5% of Superplasticizer increases the viscosity. These two complementary rheological aspects define the required self-compacting cement paste property. The values of Sp and F/C satisfying these two properties are surrounded by circles in the graphs of the two responses.

Table 10 presents statistical characteristics of these two responses.

Optimal paste: Digital processing of the experimental plans has allowed to optimize simultaneously two responses, it is a purely numerical procedure which consists mathematically to find a formulation or a combination of parameters for which the desired responses are either of optimal values or belonging to an interval of optimal values. This is called a multi-criteria

Table 7: Implicit constraints

Component	C	F	Sp	E
Implicit higher constraints	0.5159	0.1150	0.0246	0.4198
Implicit lower constraints	0.4491	0.0568	0.0160	0.4033

Table 8: Characteristics of the problem

Aim of the study	Mixing study
Variables number	4
Experiments number	35
Coefficients number	10
Responses number	2

Table 9: Characteristics of the experiment matrix

Properties	Values
Determinant (X'X) **1/p	3.9588
Determinant (M)	5.5578975E-0017
Determinant (M) **1/p	0.082476
Function of maximum variance	0.333333
Trace $(X'X)^{-1}$	6.0948
Effectiveness G (%)	93.75
Index of khuri (%)	97.85

Table 10: Characteristics of the responses

Response	Average	Type-gap	Minimum	Maximum	Center
Spreading out (cm)	18.377	2.275	13.60	23.0	18.30
Time flow (sec)	12.217	2.444	8.00	18.0	13.00

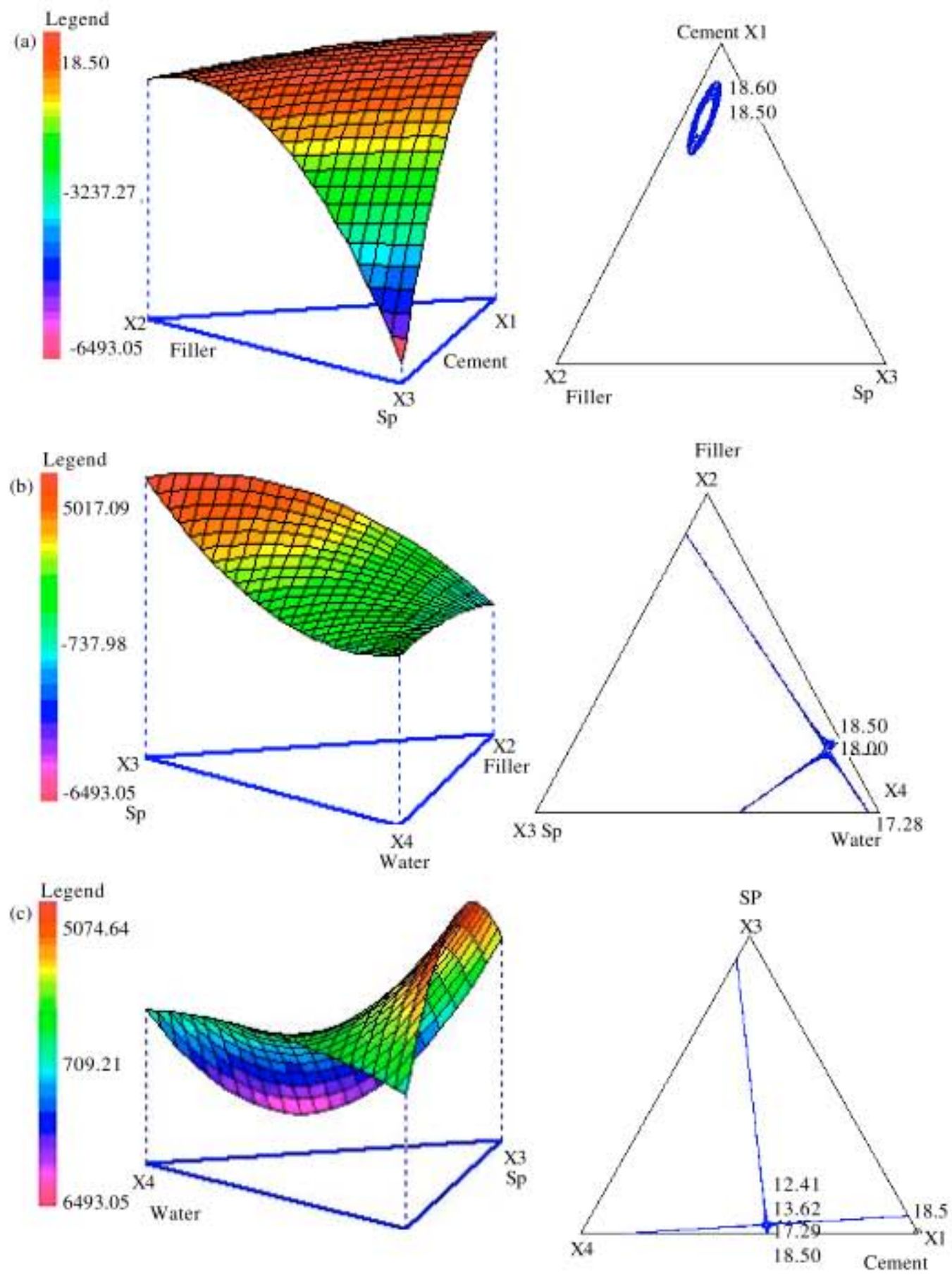

Fig. 6: Ternary diagrams for the spreading out response. (a) Variation of the spreading out response in the plan Cement-Filler-Sp Fixed component: Water = 0.305(b) Variation of the spreading out response in the plan Filler-Sp-Water Fixed component: Cement = 0.511 and (c) Variation of the spreading out response in the plan Sp-Water-Cement Fixed component: Filler = 0.112

case of an optimization which is based on desirability functions. Using these functions to solve postulated problem for each response, a profile curve of the desirability function was selected (Fig. 8). The desirability is null for an unsuitable response and is maximum when the response given is very satisfactory and it takes intermediate values to a lesser extent of satisfactory responses. The total required desirability Dg (paste) for the required optimal paste is a function of the elementary desirability's 'd(spread)' and 'd(flow)' necessary for spreading out and out-flow time respectively, and it is defined by the following relationship:

$$\text{Dg (paste)} = \sqrt{\text{d(spread)} \times \text{d(flow)}} \tag{7}$$

The graphs of the choice of desirability functions are shown in Fig. 8, it is of right unilateral type without

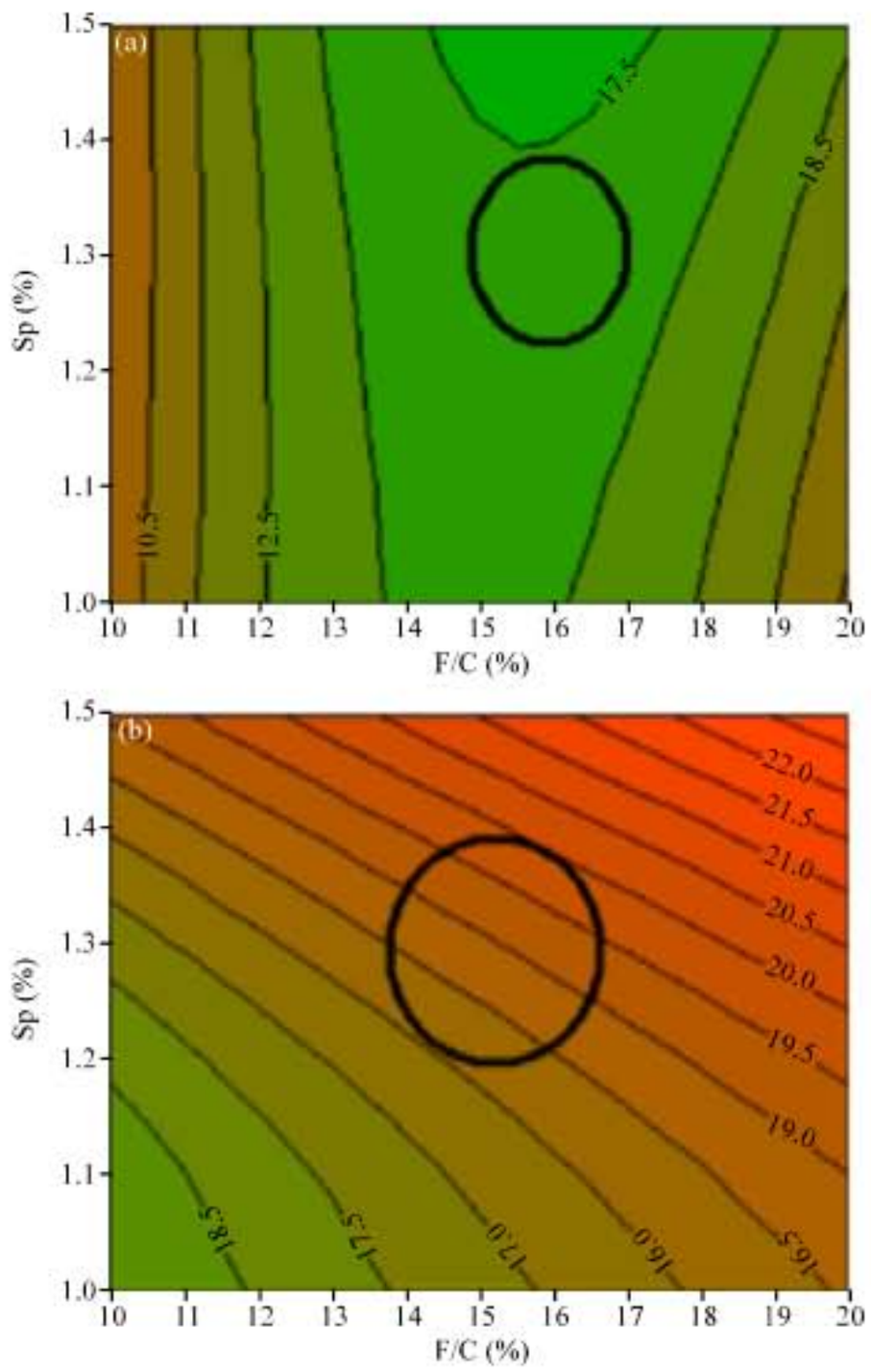

Fig. 7: (a) Spreading out for $\Gamma = 0.58$ (b) Flow Time for $\Gamma = 0.58$

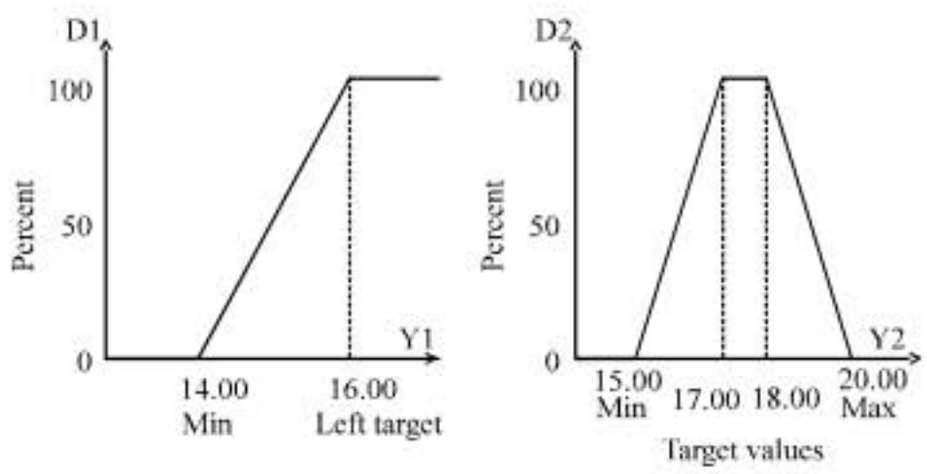

Fig. 8: Graphs of desirability functions

tolerance for the Y1 responses (spreading out) and of bilateral type with tolerance for Y2 responses (out-flow time). On the basis of this choice, Table 11 presents the characteristics of the elementary functions of desirability and the function of total desirability defined by the relationship (7).

The formulation of the optimal paste mix was obtained by satisfying the desirability criterion relating to

Table 11: Characteristics of desirability functions

Response	Value	di (%)	di min (%)	di max (%)
Spreading out diameter	17.15 cm	100	65.31	100
Out-flow time	15.75 sec	100	100.00	100
Desirability (Dg)		100	80.82	100

Table 12: Composition of the optimal paste

Component	Volume proportions	Dosage (g L^{-1})
Cement	0.538	1694.70
Limestone	0.116	324.80
Sp	0.014	15.19
Water	0.332	3320.00

Table 13: Theoretical-experimental results comparison

Values	Spreading out diameter (cm)	Out-flow time
Target values	≥14.00	15.00≤time≤20.00
Theoretical values	17.15	15.75
Experimental values	17.20	15.86

the paste homogeneity and fluidity. The volume and proportions of each component of the optimal paste are given in Table 12.

Using the proportions obtained, a cement paste was produced in order to check, to compare and then to validate theoretical results. Visually, the paste aspect was acceptable without any apparent segregation. Table 13 gives the experimental measurements of spreading out diameter and out-flow time.

The small difference between theoretical and experimental values which is about 0.05 cm for the spreading out diameter and 0.11 seconds for the out-flow time, means that the proposed model gives satisfactory results and can then be validated.

The remaining part of the work consisted of injecting aggregates (sand and gravel for a fixed ratio G/S) at the same time and balancing with water to reach the desired fluidity. At the end of the experimental procedure, a self consolidating concrete could be achieved on checking its characteristics in its fresh state according to the recommendation of the French association of civil engineering.

DISCUSSION

In this study, the method used at L.M.D.C.- INSA-UPS, Toulouse to obtain a self compacting cement paste formulation was applied to Algerian local materials due to their satisfactory results. The use of experimental plans method has contributed to give maximum information about components, their influences and possible interactions. This method and with a parametric analysis has permitted us to define zones of high viscosity and minimal shearing threshold and also to define an experimental field bounded by lower and higher constraints. The experimental mix plan with four factors i.e., C (cement), F (limestone filler), W (water) and Sp

(superplasticizer), has produced a hyper polyhedral form. The analysis of the exchanged algorithm generated by the software NemrodW was used to determine the experiment matrix satisfying a number of optimization criteria.

The volumetric values of composition parameters provided by the software were used to prepare the paste followed by measurements. The measured responses of the spreading out diameters and out-flow times were input into the software as a new data in order to define the optimal proportion values of the four components of the paste. The influence of each parameter on the paste mixture was shown via ternary diagrams obtained in space and in plan.

The experimental responses has shown that substitution of 15% of limestone fillers decreases the shearing threshold, whereas that a proportioning of 1.5% of Superplasticizer increases the viscosity both for $\Gamma = 0.58$. These two complementary rheological aspects define the required self-compacting cement paste property. In Boel *et al*. (2006) study, it was shown that the two essential properties high flowability and segregation resistance have been obtained with the use of superplasticizer and fine particles (limestone fillers) and without use of a viscosity modifying admixture.

Finally, an optimal paste was obtained by the use of multi-criteria optimization based on desirability functions and it was shown that the theoretical results were in good agreement with experimental values.

CONCLUSION

In this study, an extensive experimental program has been performed, adopting a new paste mix concept for Self-Compacting Concrete (SCC), which considers that fresh concrete self-compacting properties come from those of the cement paste. Experimental mixture method was applied to the cement pastes to get maximum information about components, their influences taken separately and their possible interactions. This method has allowed defining an experimental field in which all mixtures can show measurable characteristics and can reduce considerably the number of experiments. Indeed, it has allowed to build an experiment matrix and to propose a formulation according to fixed target values.

The optimal paste mix was produced simply by the measurements of the spreading out diameters and out-flow times. This was done by combining the criteria for low shearing threshold, high viscosity and optimum flow-viscosity ratio of the paste.

The NemrodW software used has produced ternary diagrams which showed interactions between components taken two by two. The proposed model has produced satisfactory results when compared to experimental measurements. The proposed numerical model has yielded a total desirability of 100 %, which is a proof of a satisfactory formulation of a self compacting cement paste within the experimental field.

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