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EXPERIMENTAL PLANS METHOD TO FORMULATE A SELF-COMPACTING CEMENT PASTE

EKSPERIMENTALNO NAČRTOVANA METODA ZA DOLOČITEV SAMOZGOŠČUJOČE CEMENTNE MALTE

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This paper presents a self-compacting cement-paste formulation using Algerian local materials (a binary cement consisting of natural pozzolana and limestone fillers). In this study, simple laboratory instruments were used, i.e., a mini-slump for spreading out diameters and a 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 the components utilizing the multiple combinations of proportioning these materials. From the results given by the ternary diagrams and desirability functions, an optimal self-compacting cement-paste mixture was defined. Experimental checking was performed to validate the obtained results.

Keywords: mixture plans method; pozzolana; limestone fillers; superplasticizer; implicit constraints; desirability functions; ternary diagrams; Algerian local materials

V članku je prikazan razvoj samozgoščujoče cementne malte z uporabo lokalnih alžirskih materialov (binarni cement iz naravnega vulkanskega pepela pozzolane in apnenca kot polnila). V raziskavi so uporabljene enostavne laboratorijske priprave: mini stožec in Marshev stožec za meritev časa iztekanja. Širok razpon variacij je bil uporabljen pri predhodnih preizkusih za izbiro malt s sprejemljivimi lastnostmi. Uporaba metode mešalnih načrtov je pokazala, da je mogoče eksperimentalno opredeliti polje, v katerem je mogoče dobiti optimalne meritve. To polje je bilo formulirano z matematičnimi enačbami, odvisnimi od implicitnih omejitev, ki opredeljujejo cone minimalnih strižnih pragov in maksimalne viskoznosti in je bilo nato rešeno matematično. Merilo optimizacije je bilo preverjeno kot dodatek k interaktivnosti komponent z uporabo multiplih kombinacij proporcioniranja materialov. Iz rezultatov v ternarnih diagramih želelnih funkcij je bila definirana optimalna samozgoščujoča cementna zmes. Dobljeni rezultati so bili tudi eksperimentalno potrjeni.

Ključne besede: metoda mešanja planov, pozzolana, apnenčevo polnilo, superplastizator, implicitne omejitve, želelna funkcija, ternarni diagrami, lokalni alžirski materiali

1 INTRODUCTION

Concrete is a composite material that consists essentially of a) a fluid phase called the cement paste and b) a solid phase of aggregates with a fixed gravel/sand ratio. The self-compacting properties of the concrete depend necessarily on those of the cement paste, which 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 that can be afforded by laboratories on moderate budgets.

Various combinations of paste-mix parameters have been adopted using mini-cone and Marsh-cone measurements, which has made it possible to eliminate all the undesired mixtures, representing segregation, or a lack of capacity to flow, or being poorly proportioned. The remainder of the combinations was used as a database to define an experimental field. From measurements of the spreading out diameters and the 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 criteria of the self-compacting properties.

2 MATERIALS AND METHODS

The choice of materials is based on their abundant availability and their moderate cost. The cement used is a 'CEM II/A 32.5 N', which according to European standard is ENV 197-1, contains less than 20 % of natural pozzolana added during the clinker crushing and fillers consisting of limestone, obtained from a west Algerian quarry. The physical properties of these two powders are shown in **Table 1**.

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

Table 1: Physical characteristics of cement and limestone filler
Tabela 1: Fizikalne značilnosti cementa in apnenčevega polnila

	density	specific surfaces	average diamètre
Cement	3150 kg/m^3	3400 cm^2/g	18.5 µm
Limestone	2800 kg/m^3	2880 cm^2/g	21.2µm

Table 2: Cement chemical analysis
Tabela 2: Kemična sestava cementa

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

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

Table 3: Clinker mineralogical analysis
Tabela 3: Mineraloška analiza klinkerja

C_3S	C_2S	C_3A	C_4AF
58.7	16.4	8.1	9.2

The aspects of limestone fillers and cement are shown in **Figure 1**.

Table 4 presents the chemical analysis of the limestone fillers.

Table 4: Chemical analysis of used limestone fillers
Tabela 4: Kemična sestava uporabljenega apnenčevega polnila

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

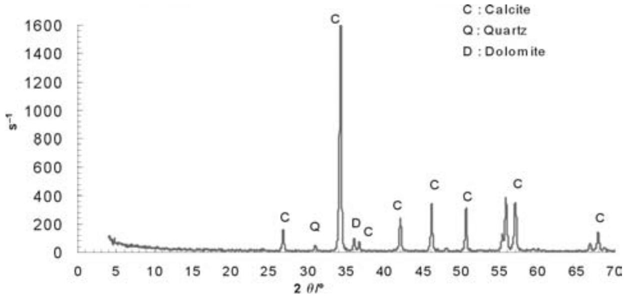


Figure 2: Limestone filler's mineralogical analysis (XRD)
Slika 2: Minaraloška sestava apnenčevega polnila

Figure 2 shows the filler's mineralogical analysis, obtained with 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 non-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 is shown in several studies[1,2] that it is possible to prepare self-compacting concretes without using a viscosity agent to remain in the context of local materials promotion. So this parameter will not be integrated into the preparation of the cement pastes. For measurements, a mini-cone inspired from the slump test was used, whose dimensions are proportional to it[3, 4, 5] **Figure 3**.

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

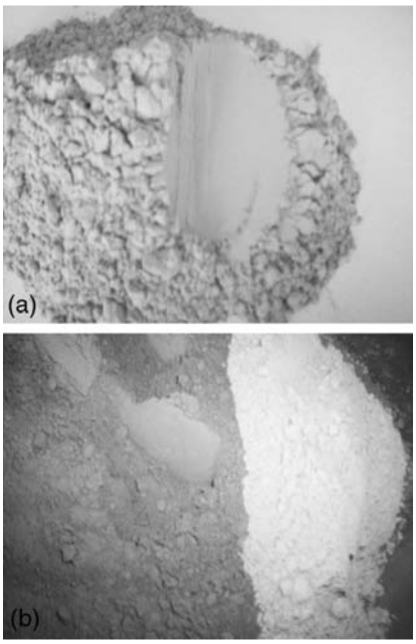


Figure 1: Aspects of limestone fillers and cement. (a) Limestone Fillers, (b) Fillers in substitution into cement
Slika 1: Videz apnenčevega polnila in cementa, (a) polnilo apnenec, (b) polnilo za zamenjavo apnenca

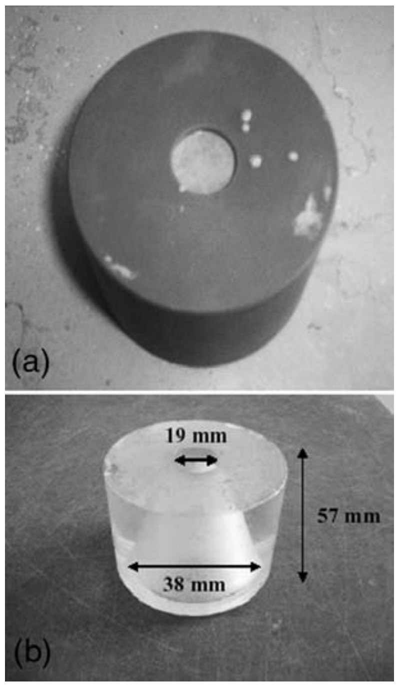


Figure 3: Mini cone
Slika 3: Mini stožec

This apparatus will be used primarily for a 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 shearing or with the apparent viscosity at a low velocity gradient[4], and the mini-slump test results correlate in certain cases with the yield stress[6].

The main advantages of this test are the facility of its implementation, since it requires simple preparation and a small quantity of materials (a volume less than 40 mL). These tests are reproducible and often used in North America for a determination of the superplasticizer's saturation point of a cementing mixture[4]. It has been shown that the paste rheology model is useful to the SCC mix design and reducing the laboratory work testing time and materials used[7]. 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[8].

For a consistency determination, a Marsh cone (**Figure 4**) was used to measure the out-flow times of a reference volume of pastes with different mixtures, and is a measure of the out-flow time of the cement paste flowing out through the cone by gravity to fill up a given reference volume (150 mL in this study). Since the tested volume was small, the test was simple and short in duration[5]. The rheological properties of the concrete (Steel Fiber Reinforced Self-Compacting Concrete) can be deduced from those of the paste which constitutes it[9].

It was concluded[10] in their study that the Marsh cone with an orifice of 5 mm was not suitable for measuring the out-flow time and bigger orifices of 8 mm 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.

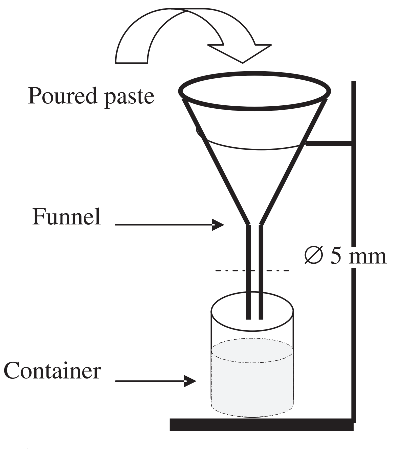


Figure 4: Marsh cone
Slika 4: Marsh stožec

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 the possible mix parameters variations that can contribute to elaborate the cement pastes. The quantity of the binder (cement + filler) was maintained as a constant and for superplasticizer, a maximum proportioning of 3 % was recommended by the manufacturer.

Table 5: Compositions of the studied pastes
Tabela 5: Sestava preiskanih muljev

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

The substitution percentage is calculated in mass terms.

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

Table 6: Experimental procedure of preparation of a standard paste mixture
Tabela 6: Eksperimentalna procedura priprave standardne zmesi mulja

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 (s)

3 RESULTS AND DISCUSSION

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

3.1 Consistency: Visual aspect

The visual aspect is a preliminary but very important stage, which allows checking the validity of the mixture visually. The paste mixture can be dry or of very firm

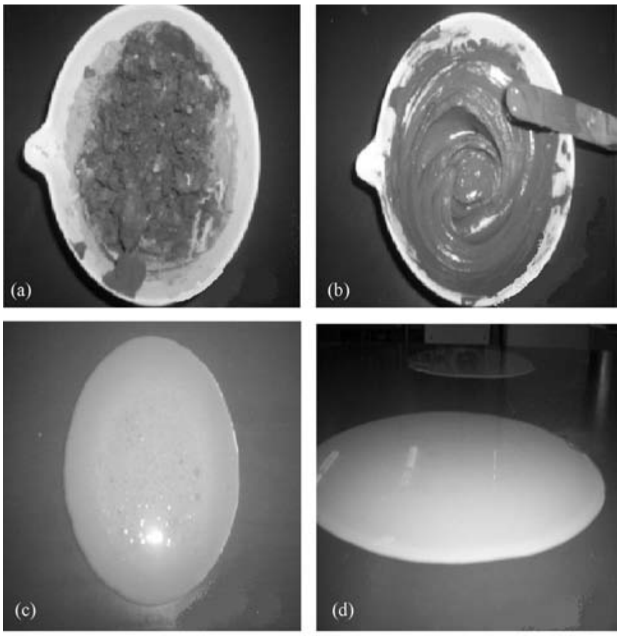


Figure 5: Visual aspects of the elaborate pastes
Slika 5: Videz pripravljenih zmesi

state, **Figure 5a**, when it is prepared with insufficient water/binder ratio (Water/Binder m(W)/m(B) = 0.22) and with an important filler substitution quantity φ(F) = 30 %). It is shown in **Figure 5b** that the paste formed was plastic and not able to flow φ(F) = 10 %, m(W)/m(B) = 0.23 and φ(Sp) = 1 %). Contrary to the previous case, it was noticed that a paste can be capable of flowing but presents a white layer (φ(F) = 15 %, m(W)/m(B) = 0.24 and φ(Sp) = 1.5 %) **Figure 5c**, which is synonymous with a 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 φ(F) = 15 %, m(W)/m(B) = 0.24 and φ(Sp) = 1.2 %). The same procedure was followed during the experiment, and only mixtures without any abnormality were retained for measurements.

3.2 Experimental plans for the cement pastes

3.2. 1 Experimental field

Several mixtures of normal consistencies were useful for the rheological measurements and have contributed to delimit the experimental field inside of which the measurements give the required results. This concerns the spreading out diameter within the interval 14.4–16 mm, in accordance with what was found,[11] and in accordance to the flow without rupture of the 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, the important parameter introduced is the solid volumetric concentration (Γ) defined by the ratio of the volume of solids on the total volume (solid particles coming from the cement, limestone fillers and superplasticizer in dry extract form). The use of the experimental plans method contributes to collecting the maximum amount of information about components, their influences taken separately and about their possible interactions. It makes it 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[12]. To achieve this, a wide variation of combinations between the mixing parameters was used as preliminary tests to select the pastes with acceptable characteristics and the use of the experimental plans method showed that it is possible to delimit an experimental field bound by the volumetric proportions of the 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 φ/% of the components used. Thus, for an experimental plan with four factors, φ(C) (cement), φ(F) (limestone filler), φ(W) (water) and φ(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:

$$\varphi(\mathrm{C}) + \varphi(\mathrm{F}) + \varphi(\mathrm{W}) + \varphi(\mathrm{Sp}) \qquad (1)$$

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 converged towards a polynomial of degree 2, relating the response Y (Y1 for the spreading out diameter or Y2 for the out-flow time) to the proportions of the components, which can be written in the following form:

$$Y = \sum_{i=j}^{k} \beta_i \times X_j + \sum_{\mathrm{i<j}}^{k} \sum^{k} \beta_{\mathrm{ij}} \times X_{\mathrm{i}} \times X_{\mathrm{j}} \qquad (2)$$

The polynomial coefficients β_{i} and β_{ij} have to be determined and are expected to be different for each response. The parameters X_{i} and X_{j} correspond to the volumetric proportions of the components. Equation (2) can be expanded to give:

$$\begin{aligned} Y = {} & \beta_1 \cdot \varphi(\mathrm{C}) + \beta_2 \cdot \varphi(\mathrm{F}) + \beta_3 \cdot \varphi(\mathrm{W}) + \beta_4 \cdot \varphi(\mathrm{Sp}) + \\ & + \beta_{12} \cdot \varphi(\mathrm{C}) \cdot \varphi(\mathrm{F}) + \beta_{13} \cdot \varphi(\mathrm{C}) \cdot \varphi(\mathrm{W}) + \beta_{23} \cdot \varphi(\mathrm{F}) \cdot \varphi(\mathrm{W}) + \\ & + \beta_{14} \cdot \varphi(\mathrm{C}) \cdot \varphi(\mathrm{Sp}) + \beta_{24} \cdot \varphi(\mathrm{F}) \cdot \varphi(\mathrm{Sp}) + \beta_{34} \cdot \varphi(\mathrm{F}) \cdot \varphi(\mathrm{Sp}) \qquad (3) \end{aligned}$$

Equation (3) can be rewritten in matrix form as follows:

$$[Y] = [X] \cdot [\beta] + [\varepsilon] \qquad (4)$$

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

After preliminary tests, a parametric analysis was used to define zones that 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:

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

Where (Γ) is the volumetric concentration in the solids.

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

$$\begin{aligned} \varphi(\mathrm{C}) + \varphi(\mathrm{F}) + \varphi(\mathrm{Sp}) &= 1 \\ -0.1125\ \varphi(\mathrm{C}) + \varphi(\mathrm{F}) &\geq 0 \\ 0.2250\ \varphi(\mathrm{C}) - \varphi(\mathrm{F}) &\geq 0 \\ -0.0290\ \varphi(\mathrm{C}) - 0.0258\ \varphi(\mathrm{F}) + \varphi(\mathrm{Sp}) &\geq 0 \\ 0.0435\ \varphi(\mathrm{C}) + 0.0387\ \varphi(\mathrm{F}) - \varphi(\mathrm{Sp}) &\geq 0 \\ 0.43\ \varphi(\mathrm{C}) + 0.43\ \varphi(\mathrm{F}) - 0.17\ \varphi(\mathrm{Sp}) - 0.57\ \varphi(\mathrm{W}) &\geq 0 \\ -0.41\ \varphi(\mathrm{C}) - 0.41\ \varphi(\mathrm{F}) + 0.19\ \varphi(\mathrm{Sp}) + 0.59\ \varphi(\mathrm{W}) &\geq 0 \end{aligned} \tag{6}$$

Here, a numerical example is presented, which shows a way of obtaining the system of equations:
φ(F) is the limestone's filler volume rate = V(F).
M(F) is the limestone's filler mass.
(Limestone's mass proportion)

$$\leq 20\ \% \Leftrightarrow \frac{M(\mathrm{F})}{M(\mathrm{C})} \leq 0.2 \Leftrightarrow \frac{M(\mathrm{F})}{M(\mathrm{C})} - 0.2 \leq 0$$

$$\frac{V(\mathrm{F})}{V(\mathrm{C})} = \frac{\frac{M(\mathrm{F})}{2800}}{\frac{M(\mathrm{C})}{3150}} = \frac{M(\mathrm{F})}{M(\mathrm{C})} \times \frac{3150}{2800} \rightarrow \frac{M(\mathrm{F})}{M(\mathrm{C})} = \frac{V(\mathrm{F})}{V(\mathrm{C})} \times \frac{2800}{3150} =$$

$$= \frac{V(\mathrm{F})}{V(\mathrm{C})} \times 0.889$$

$$\frac{V(\mathrm{F})}{V(\mathrm{C})} \times 0.889 - 0.2 \leq 0 \Leftrightarrow V(\mathrm{F}) - 0.225 \times V(\mathrm{C}) \leq 0 \rightarrow$$

$$-\varphi(\mathrm{F}) + 0.225 \times V(\mathrm{C}) \geq 0$$

Thus: $0.225 \times \varphi(\mathrm{C}) - \varphi(\mathrm{F}) \geq 0$ 3rd equation of system (6).

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

Table 7: Implicit constraints
Tabela 7: Implicitne omejitve

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

For an experimental mix plan with four factors, the field formed was a space with four 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 gravitational centre. An analytical solution for this complex problem is almost impossible; numerous solutions were obtained by using software packages 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 an experimental plan.

3.2.2 Experiment matrix

The determination of the experiment matrix was carried out with 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 $N = N_{max}$ satisfies the following optimization criteria:

- Criterion D: optimization of the information quality.
- Criterion A: optimization of the model coefficients' quality.
- Criterion G: optimization of the model prediction quality.

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

Table 8: Characteristics of the problem
Tabela 8: Karakteristike problema

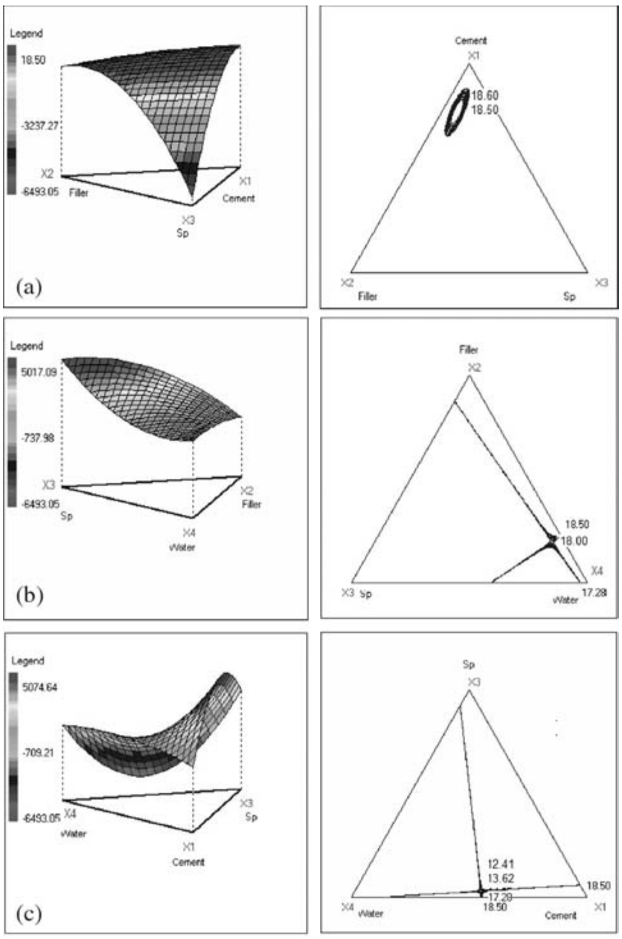
Aim of the study	Mixing study
Variables number	4
Experiments number	35
Coefficients number	10
Responses number	2

The characteristics of the experiment matrix for the 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.

Table 9: Characteristics of the experiment matrix
Tabela 9: Značilnosti eksperimentalne matrice

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

The volumetric values of the composition parameters provided by the software were used to prepare the pastes and followed by the measurements. In order to define the optimal proportion values of the cement, limestone fillers, superplasticizer and water, the measured responses of the spreading out diameters and the out-flow times were again input into the software as new data. **Figure 6** shows the ternary diagrams in space and in the plane 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 "φ(Sp)" will be dominating i.e., the responses are more sensitive to the variations of this parameter than to those of the "cement" or those of the "filler". There would then be interactions between the "φ(Sp)" and "cement" according to the site of the influence field, which is closer to the cement than to the filler, **Figure 6**. It should be noted here, that the same work has been carried out for the out-flow time response.



(a) Variation of the spreading out response in the plan Cement-Filler-Sp. Fixed component: Water, φ(W) = 0.305
(b) Variation of the spreading out response in the plan Filler-Sp-Water. Fixed component: Cement, φ(C) = 0.511
(c) Variation of the spreading out response in the plan Sp-Water-Cement. Fixed component: Filler, φ(F) = 0.112

Figure 6: Ternary diagrams for the spreading out response
Slika 6: Ternarni diagram odgovora pri širjenju

3.2.3 Experimental responses

Figure 7a and 7b show the curves obtained for Γ = 0.58 at the end of the processing. **Figure 7a** shows that the substitution of 15 % of limestone fillers decreases the shearing threshold, whereas **Figure 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's property. The values of φ(Sp) and φ(F)/φ(C), satisfying these two properties, are surrounded by circles in the graphs of the two responses.

As shown[13], the two essential properties "*high flowability and segregation resistance*" have been obtained with the use of the superplasticizer and fine particles (limestone fillers) and without use of a viscosity-modifying admixture.

Table 10 presents the statistical characteristics of these two responses.

Table 10: Characteristics of the responses
Tabela 10: Značilnosti odgovorov

Response	*Average*	*Type – gap*	*Minimum*	*Maximum*	*Center*
Spreading out (cm)	18.377	2.275	13.600	23.000	18.300
Time flow (s)	12.217	2.444	8.000	18.000	13.000

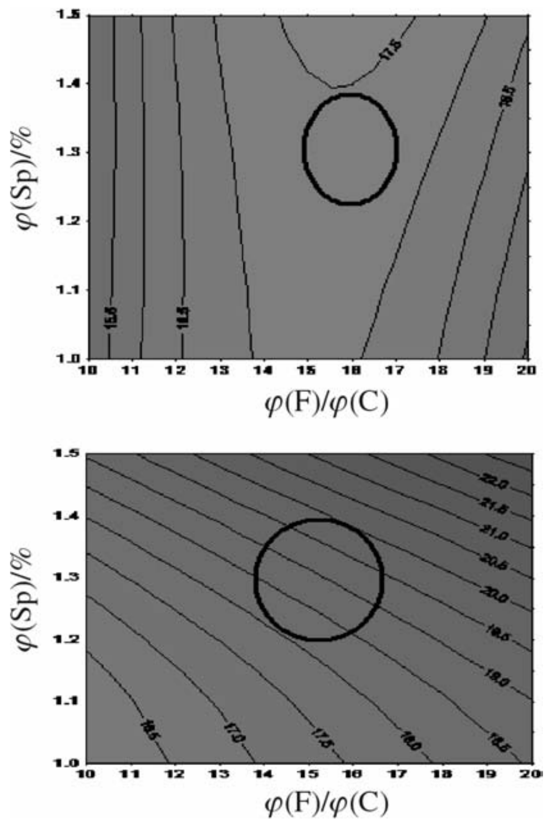


Figure 7: (a) Spreading out for Γ = 0.58, (b) Flow Time for Γ = 0.58
Slika 7: (a) Širjenje za Γ = 0.58, (b) čas iztekanja za Γ = 0.59

4 OPTIMAL PASTE

Digital processing of the experimental plans has allowed us to optimize simultaneously two responses. It is a purely numerical procedure that 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 the multi-criteria case of an optimization, based on desirability functions. Using these functions to solve the postulated problem for each response, a profile curve of the desirability function was selected, **Figure 8**. The desirability is null for an unsuitable response and is a maximum when the response given is very satisfactory, and it takes intermediate values to a lesser extent than the 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 the spreading out and out-flow time, respectively, and it is defined by the following relationship:

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

The graphs of the choice of desirability functions are shown in **Figure 8**. It is of the right unilateral type, without tolerance for the Y1 responses (spreading out) and of the 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).

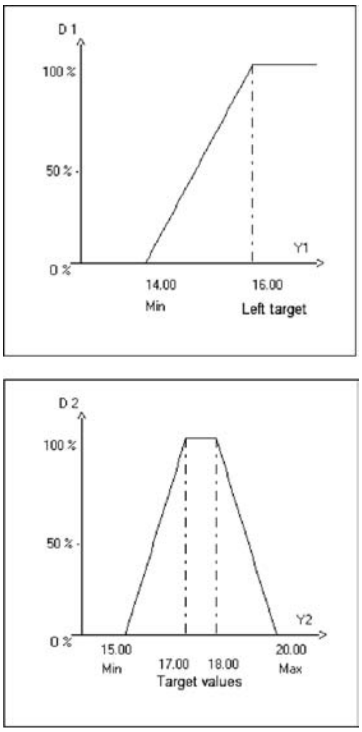


Figure 8: Graphs of the desirability functions
Slika 8: Graf želelnih funkcij

Table 11: Characteristics of desirability functions
Tabela 11: Značilnosti želelnih funkcij

Response	Value	*di* /%	*di*(min) /%	*di*(max) /%
Spreading out diameter	17.15 cm	100.00	65.31	100.00
Out-Flow time	15.75 s	100.00	100.00	100.00
Desirability (Dg)		100.00	80.82	100.00

The formulation of the optimal paste mix was obtained by satisfying the desirability criterion relating to the paste's homogeneity and fluidity. The volume and proportions of each component of the optimal paste are given in **Table 12**.

Table 12: Composition of the optimal paste
Tabela 12: Sestava optimalne mešanice

Component	Volume proportions	Dosage, g/L
Cement	0.538	1694.7
Limestone	0.116	324.8
Sp	0.014	15.19
Water	0.332	3320

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

Table 13: Comparison of the theoretical and experimental results
Tabela 13: Primerjava teoretičnih in eksperimentalnih rezultatov

	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 small difference between the theoretical and experimental values, which is about 0.05 cm for the spreading out diameter and 0.11 s 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 G/S ratio) 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, checking its characteristics in its fresh state according to the recommendation of the French Association of Civil Engineering.

5 CONCLUSION

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

The optimal paste mix was produced simply with the measurements of the spreading out diameters and the out-flow times. This was achieved by combining the criteria for a low shearing threshold, a high viscosity and the 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 the experimental measurements. The proposed numerical model has yielded a total desirability of 100 %, which is proof of a satisfactory formulation of a self-compacting cement paste within the experimental field.

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Used symbols:

C: Cement. **F:** Limestone fillers. **Sp**: Superplasticizer. **W**: Water.

D1 = Y1: First response: Spreading-out diameter (measured by mini-slump).

D2 = Y2: Second response: Flow time (measured by Marsh cone).

φ(C), φ(Sp), φ(F) and φ(W): volumetric proportions of cement, superplasticizer, limestone filler and water.