BULETINUL INSTITUTULUI POLITEHNIC DIN IAŞI
Tomul LIII (LVII), Fasc. 3–4, 2007
CONSTRUCŢII. ARHITECTURĂ

STATISTICAL INVESTIGATION OF MECHANICAL CHARACTERISTICS OF POLYMER CONCRETE USING MIXTURE DESIGN OF EXPERIMENTS

BY

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The aim of this paper is to present an approach for developing material models, using a statistical investigation, for analysing the mechanical characteristics of polymer concrete. The experimental studies were realized on polymer concrete prepared of epoxy resin, silica fume and aggregates. The mix combinations were designed based on the mixture design of experiments concept. For each polymer concrete combination the mechanical properties were studied. The results are reported for polymer concrete realized of epoxy resin and silica fume (SUF) as filler using Response Surface Methodology (RSM). The effect of each variable on the response was analysed by this advanced method adapted on the study.

1. Introduction

The optimization process is one of the most important problems that appear in the activity of the researchers and engineers for industrial development that has created new building materials with improved performances. The polymeric concrete (PC) [1],...,[3] occurred and developed in construction industry due to its advantages compared with the Portland cement concrete, such as: quick setting characteristics, high mechanical strength, chemical resistance and wear resistance. In the composition of polymeric concrete there are polymer matrix and sand and rocks, as inclusions. Different types, properties and applications of polymer concrete have been reported [5]. The performances and the use domain of polymer concrete depend on the polymer binder, type of filler and aggregates. The mechanical properties and the curing behaviour depend on the selection and the content of the polymer, temperature and aggregate type and dosage. The presence of filler is also important and the use of silica fume in the mix improved the mechanical properties. However, because the PC costs are high, a statistical analyse of her properties is necessary for keeping the costs as low as possible.

The present paper deals with the influence of polymer concrete components on its characteristics. The optimizing of polymer concrete mix was realized by experimental researches on the basis of mixture design of experiments and response surface methodology.

2. Experimental Program

The materials used were: epoxy resin, silica fume (SUF) and crushed aggregates of two grades 0...4 mm (Sort I) and 4...8 mm (Sort II). The epoxy resin in combination with the hardener forms the binder of the polymer concrete. The silica fume is a by-product that results from ferrosilicon production having the following characteristics:

1. particle sizes – 0.01...0.5 μ;
2. shape of particles – spherical;
3. specific surface – between 13,000 and 23,000 m^2/kg;
4. density – between 2.1 and 2.25 g/cm^3 [2].

The aggregates were obtained from river stone by crushing. Polymer concrete of different compositions, as are given in Table 1, was prepared by mixing required quantities of epoxy resin firstly with aggregates, than with the filler (SUF) was added slowly in a mechanical mixer. Than the casting of specimens (cubes of 70.7 mm sides) were prepared (Fig. 1) for determining the densities and the mechanical characteristics such as: Compressive Strength (CS) and Adherence Strength (AS).

Table 1
Mixture Design Combinations for Polymer Concrete

Mixture	Epoxy resin, [%]	SUF %	Aggregate Sort I, [%]	Aggregate Sort II, [%]
PC1	18.8	6.48	37.4	37.4
PC2	12.4	12.8	37.4	37.4
PC3	12.4	6.4	43.8	37.4
PC4	12.4	6.4	37.4	43.8
PC5	15.6	9.6	37.4	37.4
PC6	15.6	6.4	40.6	37.4
PC7	15.6	6.4	37.4	40.6
PC8	12.4	9.6	40.6	37.4
PC9	12.4	9.6	37.4	40.6
PC10	12.4	6.4	40.6	40.6
PC11	16.4	7.2	38.2	38.2
PC12	13.2	10.4	38.2	38.2
PC13	13.2	7.2	41.4	38.2
PC14	13.2	7.2	38.2	41.4
PC15	14.0	8.0	39	39.0

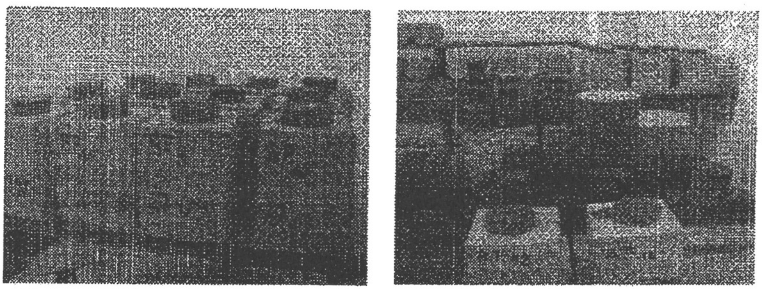

Fig. 1.– Test samples of PC.

3. Mixture Design of Experiment and Response Surface Methodology

3.1. Mixture Design of Experiment

Research in many disciplines frequently involves bleeding two or more ingredients together. The design factors in a mixture experiment [8], [11], [12] are the proportions of the components of a blend, and the response variables vary as functions of these proportions making the total and not actual quality of each component. The total amount of the mixture is normally fixed in a mixture experiment and the component settings are certain proportions of the total amount. The component proportions in a mixture experiment cannot vary independently as in factorial experiments since they are constrained to sum to a constant (1 or 100% for standard design). Imposing such constraint on the component proportions complicates the design and the analysis of mixture experiments. Although the best-known constraint in a mixture experiment is to set the sum of the components to one (100%) additional constraint such as imposing a maximum or minimum value on each mixture component may also apply.

3.2. Response Surface Methodology

Response Surface Methodology (RSM) provides an approximate relationship between a true response, y, and p design variables, which is based on the observed data from the process or system [4], [6], [7], [10]. The response is, generally, obtained from real experiments or computer simulations, and the true response, y, represents the expected response. Thus, real experiments are performed in this paper. We suppose that the true response, y, can be written as

$$y = F(x_1,\ x_2, ..., x_p), \tag{1}$$

where the variables x_1, x_2,....x_k are expressed in natural units of a measurement. so they are called *natural variables*. Usually, the approximating function, F, of the true response, y, is chosen to be either a first-order or a second-order polynomial model, which is based on a Taylor series expansion. In this study, the second-order model

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{\substack{i=1 \\ i \neq j}}^{k} \sum_{j=1}^{k} \beta_{ij} x_j + e, \tag{2}$$

is utilized, where: β_0, β_i, β_{ii} and β_{ij} are called *regression coefficients*, and e represent the noise or the error observed in the response, y [4].

4. Results and Discussions

Out of number of factors identified by their simplified notation (A, B, C and D), the following ones were considered to be most important and necessary to control:

1. Epoxy resin (A);

2. Silica fume (B);
3. Aggregate sort I, 0...4 (C);
4. Aggregate sort II, 4...8 (D).

The input variables range chosen for the study, their coded value are given in Table 2.

Table 2
Order of Importance of the Variables

Sample	Variable	Lower limit		Upper limit	
		%	coded value	%	coded value
1	A	0	0.124	1	0.188
2	B	0	0.064	1	0.128
3	C	0	0.374	1	0.438
4	D	0	0.374	1	0.438

Each polymer concrete mixture was prepared and tested under identical conditions. The density of hardened concrete and the compressive strength at 14 days were determined experimentally. Table 3 summarizes mixture design and their experimental responses, CS and AS, and for each polymer concrete combination based on the concept of design of experiments. Mixture designs (1...10 runs) are sometimes augmented by adding interior points (11...15 runs). A center points will be added to the design data (Table 3) with five runs making 15 runs total. This addition will change the design from simplex-lattice to simplex-centroid design. The experimentally studied response, based on the observed results at the 14-days, was analysed statistically using Statistica software.

Table 3
Mixture Design Combination and their Experimental Responses

Combination reference (runs)	A, [%]	B, [%]	C, [%]	D, [%]	CS, [N/mm^2]	AS, [N/mm^2]
1	18.8	6.48	37.4	37.4	59.2	7.27
2	12.4	12.8	37.4	37.4	58.79	5.18
3	12.4	6.4	43.8	37.4	59.59	5.67
4	12.4	6.4	37.4	43.8	57.61	6.57
5	15.6	9.6	37.4	37.4	64.08	8.45
6	15.6	6.4	40.6	37.4	58.62	10.25
7	15.6	6.4	37.4	40.6	43.47	9.01
8	12.4	9.6	40.6	37.4	45.95	7.56
9	12.4	9.6	37.4	40.6	55.21	7.66
10	12.4	6.4	40.6	40.6	55.62	9.06
11	16.4	7.2	38.2	38.2	58.8	5.87
12	13.2	10.4	38.2	38.2	63.2	5.47
13	13.2	7.2	41.4	38.2	65.32	5.77
14	13.2	7.2	38.2	41.4	57.61	7.96
15	14.0	8.0	39	39.0	57.75	10.15

Analysing the experimental results (Table 3) we can observe that:

1. The values of compressive strengths varied between 65.32 N/mm^2 (for concrete type BP13) and 43.47 N/mm^2 (for concrete BP7), that characterizes the polymer concrete as a high strength concrete.

2. The epoxy resin dosage varied between 10.8% and 23.6%; the maximum compressive strength was obtained for 12.4%.

3. The values of aderence strenght varied from 10.25 N/mm^2 (for concrete type PC6 with 15.6% polymer) to 5.18 N/mm^2 (for concrete type PC2 with 12.4% polymer).

In the mixture design approach the total amount of the input variables was fixed and constrained to sum 100. For each statistical combination all properties of interest were measured and empirical models for each property as function of the input variables were determined with regression analysis. The advantage of the mixture approach is that the experimental region of interest is more naturally defined. To simplify calculation and analysis, the actual variables ranges were transformed to dimensionless coded variables with a range between 0 and 1. Intermediate values were also translated similarly. The variables A, B, C, and D were codified using the following formula:

$$\text{Pseudo} = \frac{R_i - L_i}{1 - L}, \tag{3}$$

where: $R_i = A_i / \sum A_i$; L_i – the lower constraint in real value, L – the sum of lower constraint in real value, A – the actual value, and A_i – the total of actual values. The second-order polynomial relation with special cubic interactions can approximate the mathematical relationship between four independent variables and the response

$$Y = \beta_0 + \sum_{j=1}^{p} \beta_j x_j + \sum_{j=1}^{p} \beta_{jj} x_j^2 + \sum_{i<j}^{p} \beta_{ij} x_i x_j + \sum_{i<j}^{p} \sum_{\substack{k=1 \\ k \neq i,j}}^{p} \beta_{ijk} x_i x_j x_k + \varepsilon, \tag{4}$$

where: β_i are linear coefficients, β_{ii} – quadratic coefficients, β_{ij} – cross-product coefficients, β_{ijk} – the special cubic coefficients and ε – the random error which includes measurement error regarding the response and is inherent in the process or system. These coefficients are unknown ones, usually estimated to minimize the sum of the squares of the error term, which is a process known as regression.

A standard statistical technique to carry it out is the analysis of variance (ANOVA); it is routinely used to provide a measure of confidence. ANOVA results for 14-day strength are shown in Tables 4 and 5. By this way we can observe the importance of interaction effect of the three leading factors (A, B, C), which is expressed by the coefficient $R^2 = 0.99987$ (Table 4) and $R^2 = 0.97619$. This coefficient shows an adequate fit for the predictive response surface model of the responses (CS, AS) investigated.

Table 4
Summary of ANOVA for CS

	SS effect	df effect	MS effect	SS error	df error	MS error	F	p	R^2
Linear	10.4285	3	3.4761	228.504	11	20.77316	0.16734	0.916175	0.043646
Quadratic	155.595	6	25.932	72.9095	5	14.58191	1.778405	0.272058	0.694854
Special cubic	72.8799	4	18.219	0.02963	1	0.02963	614.9242	0.030235	0.999876
Total adjusted	238.933	14	17.066						

Table 5
Summary of ANOVA for AS

	SS effect	df effect	MS effect	SS error	df error	MS error	F	p	R^2
Linear	22.57143	3	7.52381	257.4286	11	23.4026	0.321495	0.809785	0.080612
Quadratic	212.2476	6	35.3745	45.18102	5	9.036204	3.914762	0.077884	0.838639
Special cubic	38.51435	4	9.62858	6.666667	1	6.666667	1.444288	0.547853	0.97619
Total adjusted	280	14	20						

Pareto chart, obtained from the statistical analysis, is presented in Figs. 2 and 3, and shows the importance order of the variables. This chart shows the variables effects on CS (Fig. 2) or AS (Fig. 3) variation of the polymer concrete.

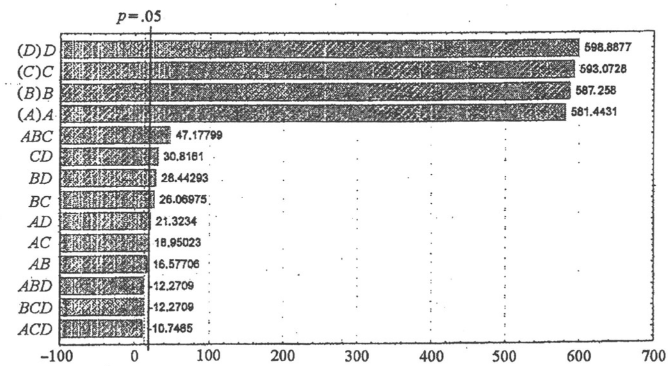

Fig. 2.– Pareto chart of standardized effect of CS.

The Pareto diagrams for the two investigated response (CS and AS) show that the four leading factors, epoxy resin (A), silica fume (B) and crushed aggregates sort I and II (C and D) are in inverse importance order.

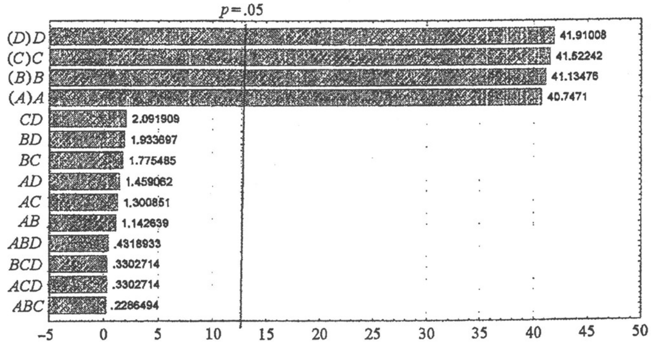

Fig. 3.– Pareto chart of standardized effect of AS.

Analysis of variance gives the non-linear response surface with the significant interactions: ABC (epoxy resin (A), silica fume (B) and crushed aggregates sort I (C), Fig. 4).

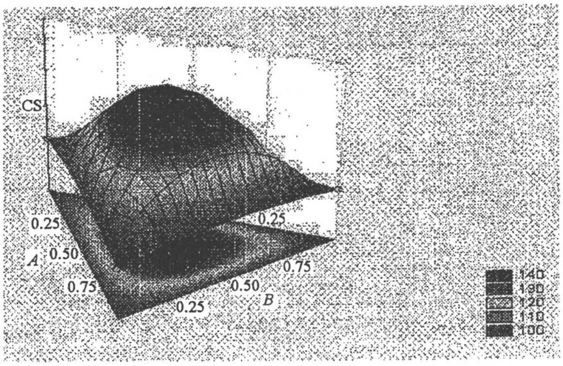

Fig. 4.– Effect of silica fume, epoxy resin and aggregate sort I on CS.

Fig. 5 shows the effect of silica fume, epoxy resin and aggregate sort I on AS.

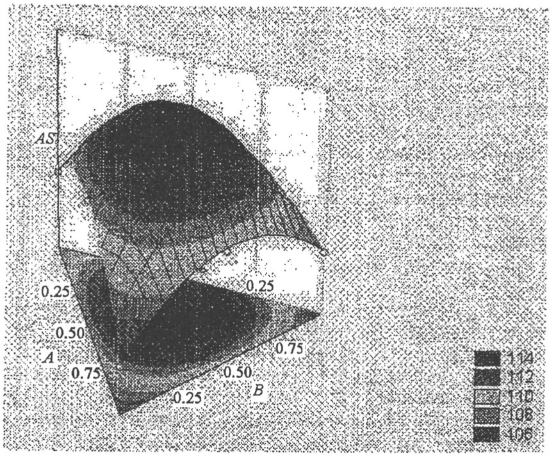

Fig. 5.– Effect of silica fume, epoxy resin and aggregate sort I on AS.

The obtained results with the performed experiment indicate that the factors have very significant effects for CS and AS. The all second-order interactions have an important role for CS. The special cubic interaction (ABC) has very significant effects (for CS) and all others interactions have very weak effects, whereas the other interactions are barely noticeable at all.

5. Conclusions

Polymer concretes were made with epoxy resin, silica fume and aggregates. Response surface method has been used for a better understanding of the influence of the deviation of the polymer concrete parameters on the Compressive Strength evolution and on Adhesion Stress between PC and ordinary concrete. For compressive strength were obtained high values for reduced dosage of resin, that show that there is a high strength concrete. The optimizing of compressive strength as maximum strength (CS = 65.32 N/mm^2) was obtained corresponding to a resin dosage of 12.4%, which is the same with that obtained experimentally. The adherence of PC to support layer is very good, for all dosages of resin, so that PC of low costs can be used for rehabilitations. The properties that were statistically predicted were verified by experimental researches.

Received, January 31, 2008

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INVESTIGAREA STATISTICĂ A PROPRIETĂŢILOR MECANICE ALE BETONULUI POLIMERIC UTILIZÂND PLANURILE DE EXPERIENŢE PENTRU AMESTECURI

(Rezumat)

Scopul acestei lucrări îl constituie prezentarea unei metode de determinare a unui model de material utilizând interpretarea statistică pentru analizarea proprietăţilor mecanice ale betonului polimeric. Studiile experimentale au fost realizate pe betonul polimeric obţinut cu răşină epoxidică, silice ultrafină şi agregate. Reţetele preparate au fost obţinute cu ajutorul metodei planurilor de experienţe pentru amestecuri. Pentru fiecare combinaţie a planului experimental au fost studiate proprietăţile mecanice ale betonului polimeric. Rezultatele au fost comparate cu cele obţinute analitic prin metoda suprafeţelor de răspuns. Efectele fiecărei variabile asupra raspunsului au fost analizate cu această tehnică avansată şi adaptată acestui tip de studiu.