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Model for predicting plastic shrinkage of polypropylene reinforced mortars

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There is increasing interest in the use of concretes reinforced with a low volume of polypropylene fibres added as secondary reinforcement, mainly to control cracking due to plastic shrinkage at an early age. This paper discusses the influence of water–cement ratio, cement–sand ratio and amount of polypropylene fibres in the plastic shrinkage of mortars. A prediction method, based on a factorial design of experiments, is also proposed.

1. Introduction

Fibre-reinforced concrete has been developed to improve several properties of plain concrete [1]. Since polypropylene fibre concrete has a relatively good response to restraint in the early stages of shrinkage [2–4], this paper discusses the shrinkage behaviour of polypropylene fibre-reinforced mortar specimens with different cement–sand and water–cement ratios and fibre contents using a factorial design of experiments.

Design of experiments (DOX) is a topic within the field of statistics [5–7]. It provides an efficient, structured approach to the problem of controlling a process with a large number of variables, such as concrete plastic shrinkage. By enabling one to efficiently explore the process using many variables, designed experiments allow the engineer to determine which of the variables have significant effects on the process. Once they are identified through screening experiments, additional experiments provide mapping of the response surface and lead to efficient process optimization.

In contrast, a traditional method for conducting scientific experiments has been to hold everything constant while changing only one variable at a time. Data variation could then be attributed to the shift in that variable. This method poses two problems: it is very time consuming, and it does not measure the interaction between two variables since they must be varied simultaneously to see the effect. Often these interaction effects are the strongest and most important factors in controlling a process.

2. Experimental procedure

2.1. Materials and mix proportions

A Spanish Portland cement, UNE I/45 A, was used. Its physical properties and chemical analyses are given in Table I. The sand employed followed the Spanish Standard, EH-91 [8].

The commercial polypropylene fibres used in these experiments had a rectangular cross-section and were 14 mm long. The specific gravity, modulus of elasticity and tensile strength of the polypropylene fibres were 0.90, 3.5 kN mm^{-2} and 0.56–0.77 kN mm^{-2}, respectively.

2.2. Apparatus

Experimental equipment was designed to measure plastic state shrinkage in mortars and concretes (Fig. 1). It consists of a chamber, in which air flow speed and temperature are held constant inside by means of a fan (B in Fig. 1), an electrical resistance (C) and a control system (A) connected to a thermometer (D). The relative humidity was also recorded by means of a hygrometer (G). In the interior of the chamber two specimens of $20 \times 150 \times 1200$ mm^3 were placed (H in Fig. 1). Shrinkage was measured with extensometers located on steel plates (F in Fig. 1) and connected to other steel plates by a steel rod (E in Fig. 1).

2.3. Testing procedure

The specimens used for studying shrinkage in the plastic state ($20 \times 150 \times 1200$ mm^3 were fabricated in accordance with the proportions shown in Table II. Once the extensometers are located on these specimens, the chamber is closed and, in the interior, wind speed and temperature conditions are set. The shrinkage reading (mm m^{-1}) is measured over time.

2.4. Factorial design

The designed experiment described here for obtaining the mortar mix, in order to have a reasonably low plastic shrinkage, includes three variables. It allows screening of all three variables with only nine samples. Using the traditional one-variable-at-a-time approach

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TABLE I Physical and chemical properties of Spanish Portland cement, UNE I/45 A

Properties	Results
Chemical properties (wt %)	
SiO_2	18.86
Al_2O_3	3.82
Fe_2O_3	3.88
CaO	63.32
MgO	1.24
SO_3	2.91
Loss on ignition	3.17
Insoluble residue	1.89
Alkali content	0.15
Na_2O	1.04
K_2O	
Physical properties	
Density	3.14 $g cm^{-3}$
Residue on 900 mesh	0.04%
Residue on 4900 mesh	0.35%
Soundness, Le Chatelier	0.3 mm
Compressive and tensile strength	
After 3 days	16/3 MPa
After 7 days	26/5 MPa
After 28 days	43/7 MPa
Time of setting (Vicat test)	
Initial	160 min
Final	280 min

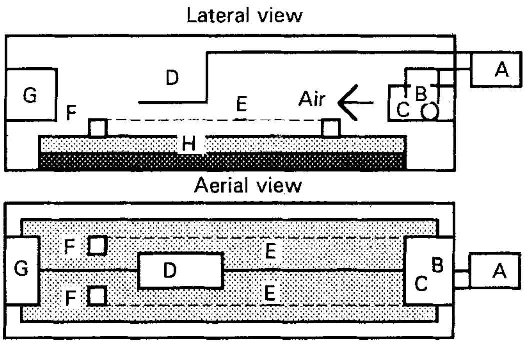

Figure 1 Schematic of experimental apparatus.

would require over 27 samples. In addition, with DOX it is now possible to measure interaction effects which may prove to be very important.

The level (setting) of each variable in an experiment can cover a large range. However, the intention is not to test the extremes of the process, but to sample a reasonable range of the values of interest. If a variable is significant, and the results show that extreme values are desirable, subsequent experiments can explore this new range.

To illustrate factorial designs, let us represent a simple version by the corners of a cube (Fig. 2). The corner points (cells) include all of the possible factorial combinations of three variables at two levels each. The four corners shown as solid balls, are the ones that would be required for a half fraction experiment. The fractional designs select a subset of cells that optimize the validity of the most important statistics (mean, main effects and two level interactions), while sacrificing the validity of higher level interactions (three level and above). For most engineering work this represents a reasonable compromise.

To obtain the optimum matrix composition and polypropylene fibre content of the composite, the Box–Hunter statistical method was applied to the experimental results of the plastic shrinkage property, in a wide range of three variables and with a small number of experiences. In this special case the three variable method was used.

A design of this type, with eight experiences on the cube corners and three in the centre, is shown in Fig. 2. This design is formed by 11 experimental combinations distributed as follows:

(a) Eight combinations corresponding to the factorial design 2^3.

(b) Three experiences in the centre in order to calculate the experimental error.

The compositions of the samples utilized in this study are shown in Table III, being the variables studied where: X_1 is the water–cement ratio (W/C), X_2 the cement–sand ratio (C/S) and X_3 the percentage of polypropylene fibres (PP).

To establish the experimental levels it is necessary to code all the variables in order to obtain the three levels of 1, 0 and +1. The levels or values of the coded variables X_1, X_2 and X_3 were obtained from Equation 1

$$X_i = \frac{X_i' - X_0}{\delta} \quad (1)$$

where X_i is the value or level to code, X_i' is the uncoded variable, X_0 is the central value (mean value of the matrix composition) and δ is the distance between the central value and the +1 and −1 levels.

TABLE II Details of specimens used for studying shrinkage in the plastic state

Experiment no.	1	2	3	4	5	6	7	8	9
Cement–sand	1:3	1:3	1:1	1:1	1:2	1:3	1:3	1:1	1:1
Water–cement	0.45	0.45	0.45	0.45	0.5	0.55	0.55	0.55	0.55
Polypropylene fibre content (VOL %)	0.00	0.20	0.00	0.20	0.10	0.00	0.20	0.00	0.20

Experiment no.	10	11	12	13	14	15
Cement–sand	1:2	1:2	1:9	1:0.8	1:2	1:2
Water–cement	0.42	0.58	0.50	0.50	0.50	0.50
Polypropylene fibre content (VOL %)	0.10	0.10	0.10	0.10	0.27	0.00

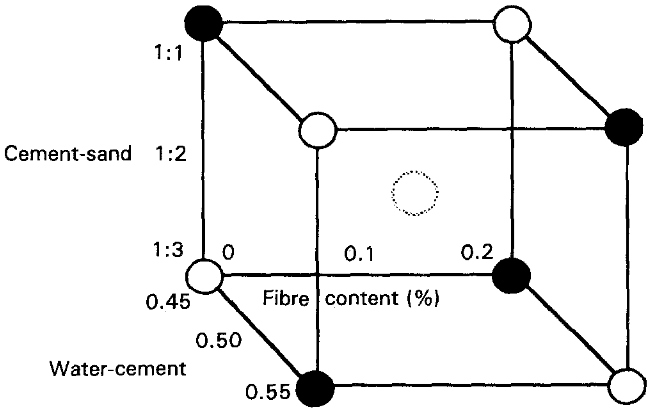

Figure 2 Example of a simple factorial design.

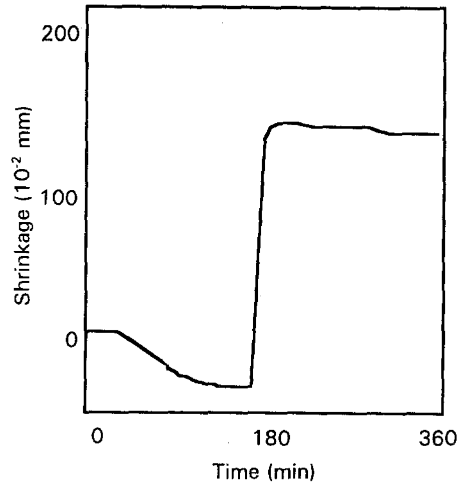

Figure 3 Shrinkage versus duration of testing.

TABLE III Variable levels

Parameter	Lower value (−1)	Central value (0)	Higher value (+1)
X_1 (W/C)	0.45	0.50	0.55
X_2 (C/S)	1:3	1:2	1:1
X_3 (PP)	0.00	0.10	0.20

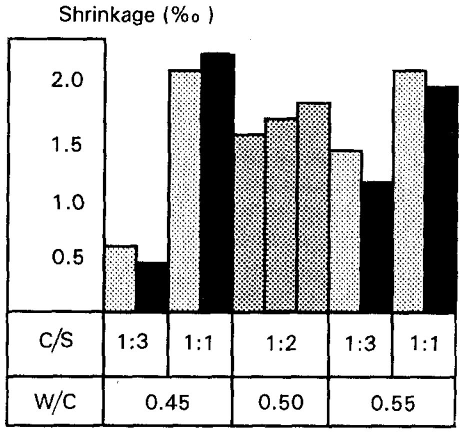

Figure 4 Plastic shrinkage results: ▨ 0% pp; ▨ 0.1% pp; ■ 0.2% pp.

The corresponding values of both variables, compiled in Table III, were calculated bearing in mınd that the percentages of fibres vary from 0 to 0.2 % by volume of the mortar matrix composition, the C/S ratio varies between 1:1 and 1:3 and the W/C content may vary from 0 to 0.6 % in the mortar.

Nine mortar mixes were made, with different cement–sand (C/S) and water–cement ratios (W/C) and polypropylene fibre content in percentage by volume of mortar (PP), that cover the working range proposed to study the effect of the mix proportions on plastic shrinkage. Then, there is a 2^3 factorial design experiment. The coding values for the different parameters are shown in Table III.

3. Results and discussion

Fig. 3 shows a characteristic trend obtained in the shrinkage tests. Three different stages are observed: (1) slight expansion due to the settling of layers on the mortar; (2) strong mortar shrinkage; and (3) loss of plastic state.

All the mortar mixes were tested by the method described in the experimental part, obtaining the plastic shrinkage results shown in Fig. 4.

The results obtained at the central point had a standard deviation of 0.1021, which confirms that the system considered for measurement of plastic shrinkage in mortars and concretes is accurate. The results are summed up in Fig. 4. It can be observed that higher shrinkage due to a higher amount of cement is more evident in "dry" mortars (water–cement = 0.45) than in the more fluid ones (water–cement = 0.55). This occurs because of the effect of the polypropylene fibres in less porous mortars (W/C = 0.45 and C/S = 1:3), is more significant than in the more porous ones.

Shrinkage reduction when the fibre content is increased in C/S of 1:3 mortars is influenced little by the water–cement ratio (W/C = 0.45 and 0.55), this being practically constant.

Summing up, it can be said that the fibre addition leads to less shrinkage, except in mortars having C/S = 1:4 and W/C = 0.45. This increase is less significant and may be produced by the increase in capillary pore content due to fibres addition in very porous mortar. This fact leads to more intense bleeding of the pore solution through the capillary pores.

In order to determine the experimental error and the curvature effect, a confidence level of 95% has been selected. It means that there exists a 95% possibility of the measure being the most accurate data obtained, with an infinte number of experiments.

Table IV shows the influence calculation. It shows that significant influences, which are bigger than experimental error, are: (a) the water–cement ratio, (b) the cement–sand ratio, and (c) the polyprolylene fibre content. The curvature value is bigger than the confidance interval. This fact does not allow us to consider a linear model to determine the influence between variables.

3.1. Full design

A star design was made in order to complement (fulfil) the pure factorial design and take into account the

TABLE IV Main effects and interactions

Parameters	Main effects and interactions
W/C	0.3650
C/S	1.2000
PP	− 0.1700
W/C C/S	− 0.5500
C/S PP	− 0.1000
W/C PP	0.1050
W/C C/S PP	− 0.1150
Standard error	0.1624
Curvature effect	− 0.2240
Confidance interval of the curvature effect	0.1980

TABLE V Variable levels

Parameter	Lower value ($-\alpha$)	Central value (0)	Higher value ($+\alpha$)
X_1 (W/C)	0.42	0.50	0.58
X_2 (C/S)	1:9	1:2	1:0.8
X_3 (PP)	0.00	0.10	0.28

curvature effect [9]. These two designs form a Box–Wilson factorial design, which needed the complementary tests shown in Table II (experiments 10–15).

The α value for orthogonal designs is calculated using Equation 2, where K is the number of factors. In this case three independent variables exist.

$$\alpha = 2^{K/4} \quad (2)$$

Six more combinations are needed to obtain central design rotability; these are called "star" combinations. Now, the coding values for the three parameters are shown in Table V.

The first experiment found in the matrix was not possible to achieve, due to the impossibility of having a negative polypropylene value. This problem was solved by considering a value for polypropylene fibre content equal to zero.

Fig. 5 shows the experiments corresponding to the star design. Their results are summarized in Fig. 6, in which it is possible to observe the lowest plastic shrinkage in mortars having a low cement content (C/S = 1:9), and the highest in rich mortars (C/S = 1:0.8). These results are in accordance with the influence calculation made above, where the parameter most affecting the plastic shrinkage of mortars is the cement–sand ratio.

The results obtained for cement–sand ratios equal to 1:2, and water–cement ratios of 0.5 without fibres and 0.58 with 0.1 vol % polypropylene fibres, are similar. This data suggests that, sometimes, the positive effect of fibre addition might compensate for an excess of mixing water. The results of other dry mortars (C/S = 1:2; PP = 0.1 vol %; W/C = 0.42 and 0.58) also confirm the trend explained above, with shrinkage in these mortars being less.

Data obtained in the plastic shrinkage of mortar tests, were fitted to a polynomial function of the type

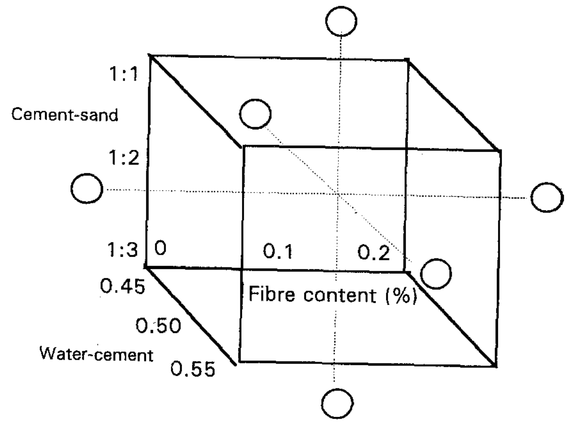

Figure 5 Schematic of star design experiment.

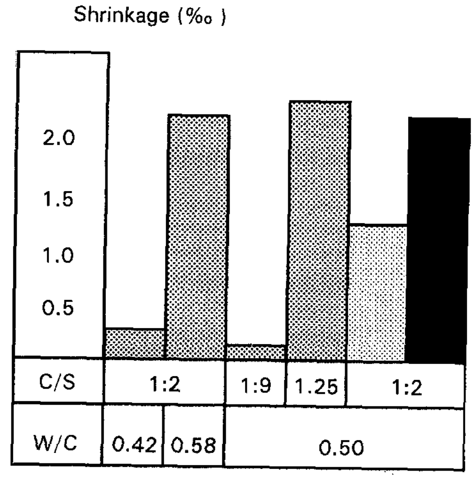

Figure 6 Star design plastic shrinkage results: ▨ 0% pp; ▨ 0.1% pp; ■ 0.2% pp.

shown in Equation 3, using the Marquard algorithm (non-linear regression method): while R is the plastic shrinkage in millimetres, $B_0, B_1, B_2, \ldots, B_n$ are constants; W/C is the water–cement ratio; C/S the cement–sand ratio; and PP the polypropylene fibre content by mortar volume.

$$\begin{aligned} R = {} & B_0 + B_1 \times \mathrm{C/S} + B_2 \times \mathrm{W/C} + B_3 \times \mathrm{PP} \\ & + B_4 \times \mathrm{W/C} \times \mathrm{C/S} + B_5 \times \mathrm{W/C} \times \mathrm{PP} \\ & + B_6 \times \mathrm{C/S} \times \mathrm{PP} + B_7 \times \mathrm{W/C} \\ & \times \mathrm{C/S} \times \mathrm{PP} + B_8 \times \mathrm{C/S}^2 + \cdots \end{aligned} \quad (3)$$

Finally, the result obtained is

$$\begin{aligned} R = {} & -4.15 + 3.80 \times \mathrm{C/S} + 7.93 \\ & \times \mathrm{W/C} - 0.76 \times \mathrm{PP} - 11.28 \times \mathrm{PP}^3 \\ & - 10.71 \times (\mathrm{C/S} \times \mathrm{W/C})^3 \end{aligned} \quad (4)$$

The polynomial equation obtained (Equation 4) permitted accurate reproduction of the experimental data, having an average error of 19.70 % (Table VI). This value was only higher in dry mortar experiments. The rest of the experiments gave noticeably smaller errors.

The proposed model permits semiquantitative calculation of the influence of the parameters studied.

From the polynomial function obtained, it can be deduced that the parameters most positively affecting

TABLE VI Experimental results and prediction errors

Test number	C/S	W/C	PP	Result (mm)	Prediction (mm)	Error (%)
1	1.00	0.45	0.00	2.05	2.24	– 9.19
2	1.00	0.45	0.20	2.20	2.00	9.27
3	1.00	0.55	0.00	2.08	2.23	– 6.97
4	1.00	0.55	0.20	1.80	1.98	– 10.15
5	0.33	0.45	0.00	0.52	0.65	– 24.25
6	0.33	0.45	0.20	0.23	0.40	– 75.52
7	0.33	0.55	0.00	1.42	1.41	0.77
8	0.33	0.55	0.20	1.16	1.17	– 0.57
9	0.50	0.50	0.10	1.62	1.46	10.01
10	0.50	0.50	0.10	1.59	1.46	8.31
11	0.50	0.50	0.10	1.78	1.46	18.10
12	0.50	0.58	0.10	1.86	2.00	– 7.42
13	0.50	0.42	0.10	0.42	0.89	– 112.34
14	0.50	0.50	0.27	1.18	1.12	5.28
15	0.11	0.50	0.10	0.16	0.15	8.20
16	1.25	0.50	0.10	2.05	1.86	9.39
17	0.50	0.50	0.00	1.91	1.55	19.10

plastic shrinkage of mortars are the cement/sand ratio and the water/cement ratio, according to the main effects and interactions studied. However, it is noticeable that these parameters working together act negatively, although this effect is less than when the parameters act separately. With regard to the amount of fibres, they have a negative affect, and have less influence on shrinkage.

4. Conclusions

The optimization of a composite, based on plain and polypropylene mortars, has been studied. Experimental design techniques enable investigation of fibre reinforced mortar plastic shrinkage, obtaining results that corroborate theoretical and instrumental test results. This produces great savings in time, resources and investigation costs. The validity of the results is limited to the experimental range considered, due to the high heterogeneity of the material studied.

Working at high PP and low values of C/S and W/C gives the lower plastic shrinkage result. However, it is necessary to take into account further parameters, such as workability, in order to obtain a good quality mortar.

A polynomial function has been obtained for predicting plastic shrinkage of polypropylene reinforced mortars, in which it can be deduced that the parameters most affecting the plastic shrinkage of mortars positively are the cement–sand and water–cement ratios. However, both parameters working together negatively influence plastic shrinkage. The amount of fibres also has a negative affect, but its effect is of less significance.

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References

1. A. L. LANDAU and T. E. WEBSTER, in Proceedings of the International Conference on Evaluation and Rehabilitation of Concrete Structures and Innovations in Design, Hong Kong, edited by V. M. Malhotra (American Concrete Institute, Detroit, 1991) pp. 653–676.
2. P. A. DAHL, in Proceedings of the European Mechanics Colloquium 204 edited by A. M. Brandt and I. H. Marshall (Elsevier Applied Science, London, 1986) pp. 435–441.
3. R. N. SWAMY and H. STAVRIDES, *J. Amer. Concr. Inst.* **76** (3) (1979) 443.
4. H. KRENCHEL and S. P. SHAH, in "Fibre Reinforced Concrete Properties and Applications", edited by S. P. Shah and G. B. Botson (American Concrete Institute, Detroit, 1987) pp. 141–158.
5. G. E. P. BOX and J. H. HUNTER, " Multifactor Experimental Designs", (Oliver and Boyd, London, 1956).
6. K. C. PENG, "The Design and Analysis of Scientific Experiments", (Addison Wesley, Reading, MA, 1966).
7. G. E. P. BOX, W. G. HUNTER and J. S. HUNTER, "Statistics for Experimenters", John Wiley, New York, 1978).
8. Comisión Permanente del Hormigón, EH-91. Instrucción para el proyecto y la ejecución de obras de hormigón armado. (Ministerio de Obras Públicas y Urbanismo, Madrid, 1991).
9. O. L. DAVIES, in "The Design and Analysis of Industrial Experiments", (ICI, London, 1978).

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