Comparison of response models from full, fractionated and small central composite design of experiments in cement-based mortars

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Abstract:

Experimenters often do not have adequate time, resources or budget to carry out large number of experiments. DOE based in factorial designs are a powerful tool for designing and optimizing advanced cement-based materials, such as high performance self-compacting mortars, where many constituents raw-materials are employed (binder phase, granular materials, and admixtures) and several engineering as aesthetics requirements. The objective of this work was to compare response models from full, fractional, and small Central Composite Design of experiments applied to mortars properties.

keywords: cement-based composite; Central Composite Design; design of experiments; response model; six sigma.

MSC2010: 26A##; 34A##; 93B##.

1 Introduction

Design Of Experiments (DOE) is a systematic approach to understanding how process and product parameters affect response variables such as processability, physical properties, or product performance and uses statistical methodology to analyze data and predict product property performance under all possible conditions within limits selected for the experimental design [1]. However, the number of interactions between factors required for factor planning increases dramatically with the number of factors. Consequently, they also increase the chances that the interactions will not significantly affect the response. For this reason, fractional planning is proposed instead of full planning [2]. Because the size of an experiment increases rapidly with increasing number of levels and factors, there is a need to reduce the number of treatments in order to save time and reduce the cost of experimentation [3]. Factorial designs can be a powerful tool for designing and optimizing composite materials, where many constituent raw materials are employed (binder phase, granular materials and admixture(s)). Then, the objective of this work was compare the response models obtained from a full, a fractionated and a small central composite design to described, predict high performance self-compacting mortar properties.

2 Central composite design

The dataset available in Maia (2021) [4] regarded to a design of experiments carried out in mortars with commercial materials through a central composite design with five independent variables (key factors): (i) Vw/Vc - water to cement volume ratio; (ii) Sp/p – superplasticizer to powder mass ratio; (iii) Vw/Vp - water to powder volume ratio; (iv) Vs/Vm - sand to mortar volume ratio; (v) Vfs/Vs - fine sand to total sand volume ratio. The dataset corresponding to a full central composite design (2^5 augmented by 10 axial runs plus 8 central runs) [4], was analyzed previously by the authors. For this current study, the full factorial plan (2^5) was reduced to two half fractions (2^{5-1} augmented by 10 axial runs plus 6 central runs). In addition, a small plan (11 mixtures, augmented by 10 axial runs plus 5 central runs), was analyzed. Four dependent variables, i.e., response variables, were considered: (i) slump-flow diameter (D-flow); (ii) the time in the V-funnel (T-funnel); (iii) flexural strength, at 24 hours, determined by the three-point loading method (F,24h); (iv) compressive strength at 24 hours (Rc,24h) [4].

3 Response models

The statistical procedure will consist namely in:

a) Full quadratic Model fitting (ANOVA);

b) Model adequacy checking and diagnosis (plot analysis, Kolmogorov–Smirnov, Shapiro-Wilk, Durbin-Watson, Cook's distances);

c) Evaluation of significant individual and interaction effects.

Design-Expert software was used to assist in: analyses the results for each response variable by examining summary plots of the data, fitting a model using regression analysis and ANOVA, validating the model by examining the residuals for trends and outliers, leverage points, autocorrelation and violation of statistical assumptions, in general, and interpreting the model graphically. A detailed description of this procedure can be found in a previous publication by the authors [5], [6], [7] and/or in specialized bibliography [2], [8]. The central composite design adopted allows for the estimation of a full quadratic model as presented in Eq. 1.

$$y = \beta_0 + \sum_{i=1}^{k}\beta_i x_i + \sum_{i=1}^{k}\beta_{ii} x_i^2 + \sum_{i<j}\sum \beta_{ij} x_i x_j + \varepsilon \qquad \text{Eq. 1}$$

where y represents the response variable; xi correspond to the design variables considered; the letter β is used for model parameters (β0 is the independent term, βi represents the linear effect of x, βii represents the quadratic effect of xi and βij represents the linear-by-linear interaction between xi and xj); and ε is the fitting error. The model parameters (β0, βi, βij) can be estimated by means of a multilinear regression analysis. In the course of the analysis, it may happen that some of the terms in Eq. (1) may not be significant. The regression of Dflow is presented in Table 1 only for response variable Dflow due to length limitations

The models generated with reduced values for D-flow and T-funnel, initially, resulted in a significant Lack of Fit. This fact led to the withdrawal some values. In case of fractional plans (A and B) for the D-flow, in order to reach the non-significant Lack of Fit, fewer points were excluded. However, the small model did not respond well to treatment, and they were excluded as initially proposed for the full plan. For the T-funnel Small plan, just applying the inverse transform, as recommended for full plan, adjusted the model and obtained Lack of Fit not significant. In the case of the Fractional A, 2 values were excluded based on the linearity of the normal plot of the residuals. In the case of the Fractional A, no was excluded, but the lack of was significant if no transform of variable is applied. The F,24h and Rc,24h considered all values and resulted in a not significant Lack of Fit for all types of plans. The **Error! Reference source not found.** shows the fitted models for D-flow for full, fractionated and small central composite design (an example for comparison among plans), and the respective linear correlation factors (R^2). For a direct comparison, Fractionated B is shown with no transform of variable (i.e. with significant lack of fit).

Table 1 – Fitted models for D-flow for full, fractionated and small central composite design (design variables in coded values).

	FULL	**Fractionated A**	**Fractionated B**	**Small**
Model Terms	**D-flow (mm)**	**D-flow (mm)**	**D-flow (mm)**	**D-flow (mm)**
Independent	343.75	338.36	337.66	343.90
Vw/Vc	8.92	4.50	7.45	6.22
Sp/p	4.58	*NS*	*NS*	*5.69*
Vw/Vp	**19.42**	**16.28**	**17.60**	**18.69**
Vs/Vm	**<u>-34.92</u>**	**<u>-37.37</u>**	**<u>-35.83</u>**	**<u>-34.70</u>**
Vfs/Vs	-1.42	*NS*	*NS*	*NS*
(Vw/Vc) x (Vw/Vp)	-5.36	*NS*	*-5.87*	*NS*
(Vw/Vc) x (Sp/p)	-2.89	*NS*	*NS*	*NS*
(Vw/Vc) x (Vs/Vm)	*NS*	4.51	4.56	*5.16*
(Vw/Vp) x (Vs/Vm)	3.11	*NS*	*NS*	*NS*
(Vw/Vp) x (Vfs/Vs)	2.98	*NS*	*NS*	*NS*
(Sp/p) x (Vfs/Vs)	1.89	*NS*	*NS*	*NS*
(Vs/Vm) x (Vfs/Vs)	*NS*	*NS*	*NS*	*NS*
$(Vw/Vc)^2$	**-20.67**	*NS*	*NS*	***-19.79***
$(Vw/Vp)^2$	*NS*	*NS*	*NS*	*NS*
$(Vs/Vm)^2$	*NS*	***-13.86***	***-14.35***	*NS*
$(Vfs/Vs)^2$	*NS*	*NS*	*NS*	*NS*
Error term (ε)				
R^2	0.9858	0.9830	0.9759	0.9885
Adj-R^2	0.9802	0.9793	0.9696	0.9808

NS: non-significant term. The three most significant parameters are typed **bold** and the most significant term is also <u>underlined</u>.

4 Conclusions

DOE permit examine factors simultaneously in one experiment, in order to obtain a sound knowledge of physical and chemical mechanisms concerned. The generated models for Fractional and Small plans allowed to find answers with values close to the results of the samples obtained with Full plan. All models found high linear correlation R^2. It possible to estimate a relatively large number of main effects using a small number of experimental units.

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