International Conference on **Mathematical Analysis and Applications in Science and Engineering** ICMA^{2}SC'22

ISEP **Porto-Portugal, June 27 - 29, 2022**

Response models in experimental studies of fresh properties of cement pastes: Part II – statistical approach based in a central composite design

Lino Maia[1,2*]

[1] CONSTRUCT-LABEST, Faculty of Engineering (FEUP), University of Porto, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal.

[2] Faculty of Exact Sciences and Engineering, University of Madeira, Campus da Penteada, 9020-105 Funchal, Portugal.

[1*]linomaia@fe.up.pt

Abstract: In this work Part II, the fresh properties of cement-based pastes were evaluated through the mini-slump test and the time in Marsh funnel. Several cement-based paste compositions were studied with run of experiments being developed based in a central composite design in order to allow to carry out scientific analyzes and to apply response models. The approach allows changes in multiple input parameters at once. The results shown the ability of this type of approach to detect cross effects, to predict results and to allow composition optimization. The approach used in this Part II is faced with basic and simple approaches wherein only one input parameters is changed at once.

keywords: Approach; Central Composite Design; Response Models.

MSC2020: 49-XX; 34-XX; 92-XX.

1 Introduction

Understanding fresh properties of cement-based materials is one of the key factors for correctly choosing the constituent materials and their mix proportions. That is especially important for specific applications wherein the fresh properties play a major role. Many researches [1-5] have being published on the topic with the overall conclusion being that the as constituent materials are different a characterization is required.

As a part of a major research work to characterize fresh properties of cement-based pastes wherein different experimental approaches were used, the present paper reports the findings of the Part II of the research wherein the design of experiments was developed through a central composite design. Thus, the major goals of this work Part II are (i) to characterize the primary and cross effects due to the constituent materials of cement-based pastes on the fresh properties and parallelly (ii) to analyze and ponder about the type of research methodology applied, namely advantages and disadvantages when compared to typical approaches. Therefore, in this paper only 15 mix compositions are performed for this specific purpose which compared with the 92 mix compositions done in the Part I. The overall research was carried out in cement-based pastes with the mini-cone [6] and the Marsh funnel [7] tests being performed to evaluate fresh properties. Note that DOE of Part I and Part II are for distinct studies. Therefore, they do not compare between themselves. They complement each other for the overall research characterization and are here analyzed to evaluated and compared approaches.

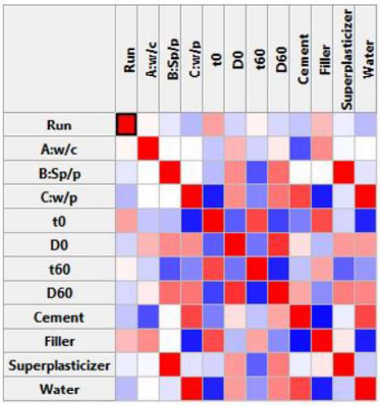
2 Results and discussion

Two powders were used: (i) commercial cement CEM I 42.5R and (ii) limestone filler. A third-generation superplasticizer and distilled water was used in all mixes. Mix compositions were defined by changing the following input parameters: water-to-cement ratio (w/c); superplasticizer-to-powder ratio (Sp/p); water-to-powder ratio (w/p). All mix compositions were prepared in a single batch which was prepared for 1.40 L. Fifteen mixtures were planned based in central composite design with three variables (A, B and C) corresponding to the three-input parameter (w/c, Sp/p, w/p), respectively. The central composition (i.e. the mix with the coded values A=w/c=0, B=Sp/p=0, C=w/p=0) was define with the actual values w/c=0.2886, Sp/p=0.80%, w/p=0.5333. The maximum changes of the input parameters in regarding to the central composition were 6.3%, 25.0% and 10.0% for w/c, Sp/p and w/p, respectively. Table 1 reports the data corresponding to each mix composition of the DOE.

Table 1 – Mix compositions of the central composite design and the corresponding test results.

std	w/c	Sp/p	w/p	cem	Filler	Sp	water	D0	t0	D60	t60
1	-1	-1	-1	1690.3	1046.7	19.159	461.15	19.95	82.50	19.18	140.12
2	1	-1	-1	1587.9	1136.0	19.067	461.21	20.28	80.06	19.18	138.69
3	-1	1	-1	1690.3	1046.7	24.634	457.87	20.25	78.50	19.35	107.75
4	1	1	-1	1587.9	1136.0	24.515	457.94	19.93	76.09	19.45	100.03
5	-1	-1	1	1804.4	861.3	18.660	493.35	18.13	67.50	18.43	112.72
6	1	-1	1	1695.0	956.5	18.561	493.41	19.63	63.03	19.68	90.44
7	-1	1	1	1804.4	861.3	23.991	490.15	21.35	61.56	21.25	87.28
8	1	1	1	1695.0	956.5	23.864	490.23	21.90	59.34	20.78	75.78
9	-1.6818	0	0	1806.6	901.6	21.666	475.88	19.98	68.46	20.20	109.87
10	1.6818	0	0	1594.1	1086.8	21.447	476.01	21.18	57.82	20.43	85.78
11	0	-1.6818	0	1693.7	1000.0	16.162	479.18	19.53	78.34	16.85	215.80
12	0	1.6818	0	1693.7	1000.0	26.937	472.71	20.35	67.37	19.93	86.56
13	0	0	-1.6818	1579.3	1188.9	22.145	442.55	18.80	87.97	18.43	144.03
14	0	0	1.6818	1800.5	823.8	20.994	507.09	21.93	53.03	21.88	74.34
15	0	0	0	1693.7	1000.0	21.550	475.95	20.30	70.97	20.00	105.25

The testing results of Table 1 – wherein the D0 and t0 are the flow diameter and the Marsh funnel time, respectively, immediately mixing and the D60 and t60 are the flow diameter and the Marsh funnel time, respectively, 60 minutes after mixing – were then analyzed in term of analysis to detect correlations between all the variables (either input or response). Those correlations might be expressed in a correlation matrix – Figure 1. This allowed a primary perception of the effect the input variable in the response variables.



	Run	A:w/c	B:Sp/p	C:w/p	t0	D0	t60	D60	Cement	Filler	Superplasticizer	Water
Run	1,000	0,030	-0,090	-0,269	0,364	-0,161	0,041	-0,152	-0,223	0,260	-0,061	-0,263
A:w/c		1,000			-0,216	0,283	-0,167	0,072				
B:Sp/p			1,000		-0,260	0,452	-0,677	0,570				
C:w/p				1,000	-0,892	0,436	-0,475	0,535				
t0					1,000	-0,631	0,716	-0,746	-0,501	0,703	-0,159	-0,863
D0						1,000	-0,542	0,803	0,118	-0,261	0,393	0,389
t60							1,000	-0,870	-0,231	0,350	-0,620	-0,404
D60								1,000	0,339	-0,445	0,507	0,472

Figure 1 – Correlation matrix of the variables.

After that, assisted by the software Design-Expert 13 Annual Network (Serial Number: 0964-0841-3719-3394) the statistical procedure ANOVA was applied to the Reduced Quadratic Model fitting for the D0, t0, D60 and t60. The regression models were found significant after several analyses as the ones presented in the Figure 2 for the t0. With the response models obtained, analyses of the results were carried out to understand the influence of each input variable in the response variable (Figure 3). Especial attention was paid on cross effects.

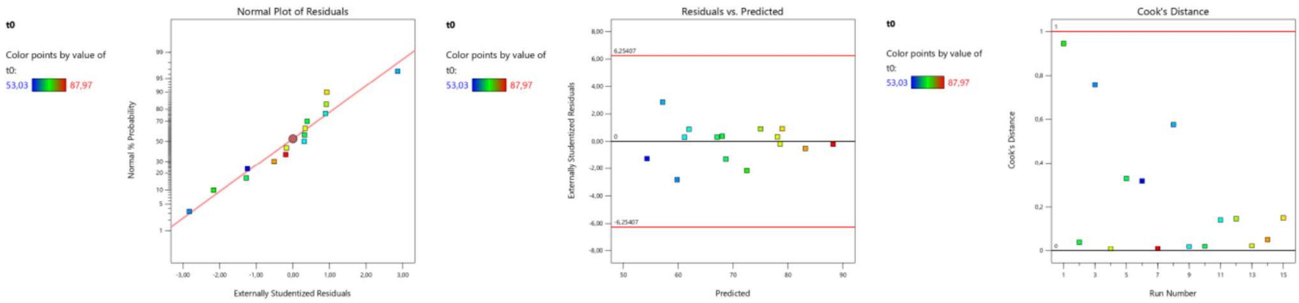


Figure 2 – Analysis of the quadratic model applied: left) normal plot of residuals, center) the Externally Studentized Residuals distribution vs. predicted, and right) the Cook's Distance.

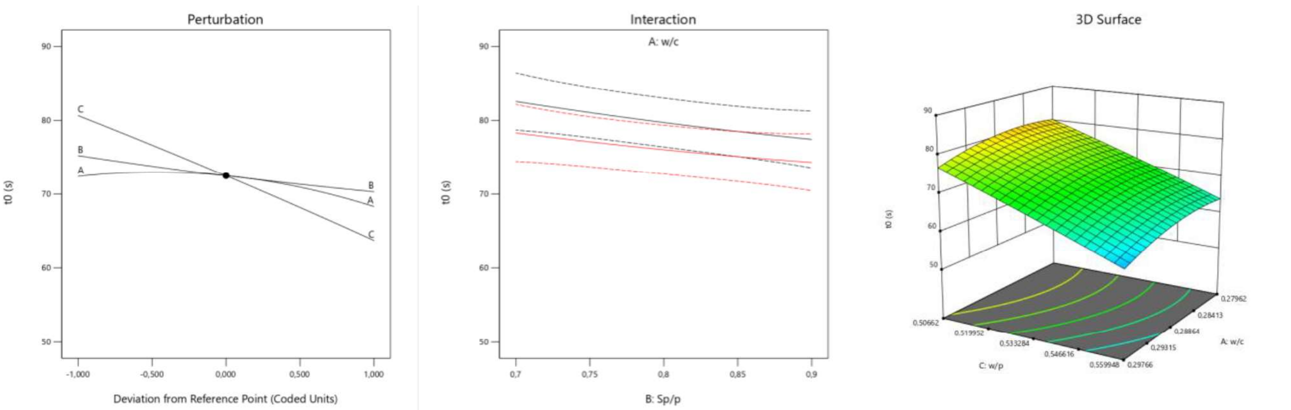


Figure 3 – Plotted results: left) Primary effect of the input parameters, center) Interaction effect of the w/c and S/p, and right) 3D model visualization.

3 Conclusions and Future work

In this paper a central composite design of experiments wherein the input parameters were changed all at once to characterize their effect on the fresh state of cement-based pastes. A vision of the advantages and disadvantages of the approach based in factorial design to apply response models when compared with the one applied in the Part II is portrayed below.

- Advantages

- It allows complex studies with many factors' interactions with a reduced number of experiments.
- As DOE is well-defined, it allows determining response models for more scientifically understand effects.
- Besides the primary effects, models can also detect the crossed effects of two factor interaction and quadratic effects (or even higher terms).
- Software assists the user and provides statistical analysis and several graphs.
- It is easy to predict results for other mix compositions.
- It allows to find out optimized compositions.

- Disadvantages

- The range of the mix compositions (input parameters) is reduced and well-defined with the response models being suitable only nearby that range.
- Previous trial-error testes are normally necessary to define the range of the input parameter to defined DOE.
- DOE has be complete, i.e. does not allow basic studies.
- It is functional only when a software is available.
- Models must be careful interpretated and effects and tends may not be easily understood.
- If advanced models are not necessary or understood, results from DOE are difficult to interpretate.

Acknowledgments

This work is financially supported by: Base Funding – UIDB/04708/2020 of the CONSTRUCT – Instituto de I&D em Estruturas e Construções - funded by national funds through the FCT/MCTES (PIDDAC). This work is funded by national funds through FCT – Fundação para a Ciência e a Tecnologia, I.P., under the Scientific Employment Stimulus – Institutional Call – CEECINST/00049/2018.

References

[1] I. Galobardes, R. P. Salvador, S. H. P. Cavalaro, A. Figueiredo, and C. I. Goodier, "Adaptation of the standard EN 196-1 for mortar with accelerator," Constr. Build. Mater., vol. 127, pp. 125–136, 2016, doi: 10.1016/j.conbuildmat.2016.09.147.

[2] B. Yogitha, M. Karthikeyan, and M. G. Muni Reddy, "Progress of sugarcane bagasse ash applications in production of Eco-Friendly concrete - Review," Mater. Today Proc., vol. 33, pp. 695–699, 2020, doi: https://doi.org/10.1016/j.matpr.2020.05.814.

[3] R. Gettu, R. G. Pillai, M. Santhanam, and B.S. Dhanya, "Ways of Improving the Sustainability of Concrete Technology Through the Effective Use of Admixtures," Indian Inst. Technol. Madras, Chennai, no. April, p. 21, 2018.

[4] Douglas C. Montgomery, Introduction to Statistical Quality Control, 7.ed. John Wiley and Sons Inc., 2013.

[5] A. M. Matos, S. Nunes, C. Costa, and J. L. Barroso-Aguiar, "Spent equilibrium catalyst as internal curing agent in UHPFRC," Cem. Concr. Compos., vol. 104, 2019, doi: 10.1016/j.cemconcomp.2019.103362.

[6] Kantro, D. L. Influence of water-reducing admixtures on properties of cement paste - a miniature slump test. Cem. Concr. Aggregates 2, 95–102 (1980).

[7] EN 445:2008 - Grout for prestressing tendons - Test methods.