Ceramics-Silikáty 61 (3), 183-187 (2017)
www.ceramics-silikaty.cz
doi: 10.13168/cs.2017.0015

DESIGN OF EXPERIMENTS IN THE STUDY OF BAUXITE REFRACTORY CASTABLE PROPERTIES

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Submitted January 23, 2017; accepted March 9, 2017

Keywords: Plackett-Burman experimental design, bauxite, low-cement refractory castables

This paper presents results of an investigation of the influence of preparation conditions of low-cement refractory castables on their service properties using experiment planning methods. Using the orthogonal Plackett-Burman design, the influence of the amount of water, vibration time, curing conditions and firing conditions on the service properties of the finished refractory castable (apparent density, open porosity, permanent linear changes, Young's modulus, bending strength and compressive strength at ambient temperature) is demonstrated. It is found that, among the investigated properties under the conditions of the conducted experiment, only the mixing time is a negligible factor that has no significant influence on the process of developing service properties of low-cement bauxite castables.

INTRODUCTION

The consumption of unshaped refractory materials is steadily increasing. This increase is connected with the constantly expanding range of applications of refractory castables and the rapid development of new technologies ensuring their high quality [1-3]. It is estimated that the volume of production of monolithic materials in EU countries accounts for approx. 40 % of all produced refractory materials.

Owing to a number of advantages offered by these materials, which are achieved by the reduction of the quantity of cement used and the introduction of nano- and micro-additives as well as appropriate liquefiers, low-cement bauxite refractory castables find application in many industries, such as the power, petrochemical and chemical industries.

The proper installation and heating up of refractory linings to match the operation conditions of a thermal installation are among the key factors that determine their service life [3-4]. Achieving the expected properties of refractory linings is only possible if the installation work is conducted under strictly defined conditions. Any deviations from the manufacturer’s recommendations that might take place in industrial practice, resulting from the wish to reduce the installation operation time, affects the physical and chemical processes occurring in the material, which in turn has an influence on the obtained final properties of the castable refractory lining, and thus on its durability.

The existing literature shows a lack in data on the influence of deviations occurring from the design specifications or the manufacturer’s recommendations on the properties of the finished refractory castable. In view of the above, this study focuses on the determination of the relationships between the preparation conditions and the service properties of bauxite castables.

Work on design of experiment was initiated by Sir Roland Fisher in the 1920s [6] and developed in the 1930s [7], but, in spite of the unquestionable benefits of the results of that work, its use in research has not become common practice to the present day. Nevertheless, in the last few years there is a trend towards the use of design of experimentnot only in natural sciences [8] but also in studies of ceramic [9-11], metallic and polymeric materials [12].

It is clear that the common practice of conducting tests by the single-factor method, consisting in changing only one parameter, with the others remaining unchanged, is highly ineffective in cases where many factors have to be examined. It involves a large number of experiments to be carried out, which will yield an excessive number of results, whose interpretation will be difficult [13-14].

Examining many factors at the same time is possible if appropriate experiment designs are used.

The orthogonal Plackett-Burman design [15] belongs to the group of selective factor designs that enable the examination of a larger number of factors simultaneously, the determination of the significance of their influence on individual properties and the selection of significant factors for subsequent model or optimization studies. The outcome of the Plackett-Burman experiment does not specify a relationship between the variable and the result; however, it does enable a possible influence, or its lack, on the examined function to be shown.

The aim of the present study was to determine, using experiment planning methods, the relationship between the conditions of making and firing low-cement bauxite castables and their service properties, and to select variables that significantly influence the process of developing the properties under investigation, i.e. the apparent density, open porosity, permanent linear changes, as well as Young's modulus, compressive strength and bending strength at ambient temperature.

EXPERIMENTAL

Test material

A commercially available low-cement bauxite castable mix was used for testing. The physical properties of the castable after firing at 800°C, as declared by the manufacturer, are as follows: apparent density 2.82 $g \cdot cm^{-3}$, compressive strength 100 $N \cdot mm^{-2}$, permanent linear changes −0.30 % and open porosity 22 %. The chemical and phase composition of the commercial bauxite castable mix used in this study is summarized in Table 1.

For the purpose of carrying out the experiments, eight types of 230×64×64 mm samples were made. Variable conditions of castable mix preparation, curing and heating were used. The experimental design considered six independent variables: amount of water X_1, mixing time X_2, mixer rotational speed X_3, vibration time X_4, curing conditions X_5, and the conditions of firing up to the working temperature X_6. The eight types of specimens were prepared under variable conditions defined at two levels, a lower level [–] and a higher level [+] relative to the reference value (Table 2), according to the experiment planning matrix for the orthogonal Plackett-Burman saturated design (Table 3). The value levels defined in the test, as shown in Table 2, correspond to the conditions prescribed by the manufacturer or by the standards (PN-EN 1927-5: 2013) [16], as well as the conditions that might occur during monolithic lining installation work not complying with the recommendations.

Table 1. Chemical and phase composition of the tested bauxite castable mix (producer's data).

Chemical composition		Phase composition of castable mix
Oxide	Declared content	
Al_2O_3	84 %	α-A, β-A, A_3S_2, AS,
SiO_2	8 %	CA, CA_2, $C_{12}A_{17}$, TA*
CaO	2.3 %	Amorphous phase
P_2O_5	1 %	

** A ≡ Al_2O_3, S ≡ SiO_2, C ≡ CaO, T ≡ TiO_2*

Table 3. Experimental design matrix (Plackett-Burman).

Experiment	Factor (as per Table 2)						
i	X_0	X_1	X_2	X_3	X_4	X_5	X_6
1	+	+	–	–	+	+	+
2	+	–	+	–	–	+	+
3	+	+	–	+	–	–	+
4	+	+	+	-	+	–	–
5	+	+	+	+	-	+	–
6	+	–	+	+	+	-	+
7	+	–	–	+	+	+	–
8	+	–	–	–	–	–	–

Testing methods

For each experiment the 3 samples (230×64×64 mm) were measured i.e. the total number of the tested samples was 24**.**

The determination of the apparent density and the open porosity was performed by the hydrostatic weighing method in accordance with PN-EN ISO 1927-6:2013 [17]. The bending strength at ambient temperature was determined in accordance with EN ISO 1927-6:2013 [17]. Three point bending test was conducted with a

Table 2. Values of levels of the investigated factors in the Plackett-Burman design.

Factor *j*	Factor (X_j)	Level [–]	Level [+]
1	Amount of water [%] (distilled water)	7.6	8.4
2	Mixing time [min]	2	4
3	Mixer rotational speed [rpm]	65	120
4	Vibration time [min] (for an amplitude of 0.5 mm)	3	5
5	Curing conditions 48 h in a mould and 24 h without a mould (drying after curing at 110°C to constant mass)	20°C, humidity 90 %	16°C, humidity 60 %
6	Conditions of firing up to the firing temperature	5°C·min^{-1} to 350°C, 2 h dwell time, 5°C·min^{-1} to 800°C, 2 h dwell time	10°C·min^{-1} to 350°C, 1 h dwell time, 10°C·min^{-1} to 800°C 2 h dwell time

loading rate of 0.15 MPa·s^{-1} and stress perpendicular to direction of fabrication was applied. The compressive strength determination was carried out in accordance with EN ISO 1927-6:2013 [17]. As a test pieces the broken halves produced from the three point bending test were used. To fulfill the requirements of EN ISO 1927-6:2013 a pressure perpendicular to the direction of casting with a loading rate of 1.0 MPa·s^{-1} was applied. Young's modulus was determined using a dynamic method, where reso-nance vibrations were induced by impulse excitation using the Resonant Frequency and Damping Analyzer RFDA (IMCE/ Belgium) (ISO 12680-1:2005) [18]. The examination of permanent linear changes was performed in accordance with PN-EN ISO 1927-6 2013 [17].

RESULTS AND DISCUSSION

The examined system response functions providing a basis for the quality assessment of the produced castable were: apparent density, open porosity, permanent linear changes after firing at a temperature of 800°C, as well as Young's modulus, compressive strength and bending strength at ambient temperature.

The average determination values, as summarized in Table 6, were obtained from three repetitions for each planned experiment

Based on the obtained results, the regression equation

$$y = b_0\,x_0 + b_1\,x_1 + b_2\,x_2 + ... + b_k\,x_k \tag{1}$$

was determined, the individual factors of the regression equation being determined from the formula

$$b_j = \frac{\sum_{i=1}^{N} x_{ji}\,y_i}{N}\ , \tag{2}$$

where i is the experiment number, j the factor number, N the number of experiments, x the variable value and y the experimental result.

To verify the significance level of individual effects, Student's t-test was used according to the formula

$$t_{calc_j} = \frac{|b_j|}{s_{b_j}}\ , \tag{3}$$

where

$$s_{b_j} = \frac{s_{rep}}{\sqrt{N}}\ , \tag{4}$$

where s_{rep} is the average standard deviation from all the determinations.

The regression equations obtained from the performed analysis of variance are summarized in Figures 1-6, where the significance level of individual factors, as expressed by $t_{calc.}$, are represented in graphical form. The significance test was performed based on Student's t-test. The critical value $t_{crit} = 2.11$ was read out for the significance level of 0.05 and the number of degrees of freedom equal to 17.

Figure 1 illustrates the effect of castable mix preparation conditions on the apparent density. Factors that significantly influence the material density are the amount of water X_1, the vibration time X_4, the curing conditions X_5 and the conditions of firing up to the working temperature X_6. Similarly as for the apparent density, the amount of water X_1, strongly influences the open porosity of the examined castable, as illustrated by Figure 2. A further factor that strongly affects the properties of the material is the type of curing conditions X_5. The significance values of the factors X_3 and X_4 are close to the critical value determined from Student's t-distribution. Figure 3 illustrates the influence of castable mix preparation conditions on the permanent linear changes. Factors significantly influencing the permanent linear changes of the examined castable are the type of curing conditions X_5 and the vibration time X_4, while the remaining factors are below the critical value. As can be seen from Figure 4, the value of Young's modulus is strongly determined by the amount of water X_1, the vibration time X_4, and firing conditions X_6. A significant factor in this case turns out to be also the mixer rotational speed X_3. The result of the performed analysis of the

Table 4. The results of apparent density, open porosity, permanent linear change, Young's modulus, bending strength and compressive strength determination.

Exp.	Apparent density [g·cm^{-3}]	Open porosity [%]	Young's modulus [GPa]	Bending strength [MPa]	Compressive strength [MPa]	Permanent linear changes after firing [%]
1	2.74 ± 0.01	22.3 ± 0.4	48.1 ± 0.9	15.0 ± 2.6	60.1 ± 5.5	0.19 ± 0.10
2	2.77 ± 0.00	21.2 ± 0.2	53.2 ± 3.9	18.1 ± 1.5	66.6 ± 4.0	0.29 ± 0.03
3	2.71 ± 0.01	23.2 ± 0.2	51.2 ± 0.6	15.8 ± 1.7	99.3 ± 7.3	0.17 ± 0.16
4	2.71 ± 0.01	23.4 ± 0.2	42.8 ± 1.3	10.6 ± 0.4	85.1 ± 1.4	0.15 ± 0.13
5	2.72 ± 0.01	22.4 ± 0.2	50.1 ± 1.3	15.1 ± 0.7	61.2 ± 5.9	0.31 ± 0.04
6	2.78 ± 0.01	21.6 ± 0.3	54.5 ± 2.0	14.6 ± 2.4	103.2 ± 5.0	0.07 ± 0.03
7	2.78 ± 0.01	20.8 ± 0.5	48.1 ± 2.3	12.7 ± 0.7	55.4 ± 4.9	0.17 ± 0.08
8	2.75 ± 0.01	22.2 ± 0.2	52.4 ± 1.2	15.2 ± 0.1	109.0 ± 3.8	0.09 ± 0.04

influence of the examined factors X_1-X_6 on bending strength at ambient temperature shows that it has been significantly influenced by two factors, namely the vibration time X_4, and the firing conditions X_6 (Figure 5). The factors X_1 and X_5 are situated practically at the significance borderline. A factor that definitely most heavily influences the compressive strength is the type of curing conditions X_5. A slight, though statistically significant, effect has also been noted for the amount of water X_1, the vibration time X_4, and the firing conditions X_6 (Figure 6).

SUMMARY AND CONCLUSIONS

Experiment planning methods have been used in this study for determining the relationship between the conditions of preparation, curing and heating of low-cement bauxite castable and its service properties.

Based on the obtained results, the significance of the following factors and their effect on the examined service properties has been established as follows:

- The amount of water is a factor that most significantly determines the apparent density, open porosity, Young's

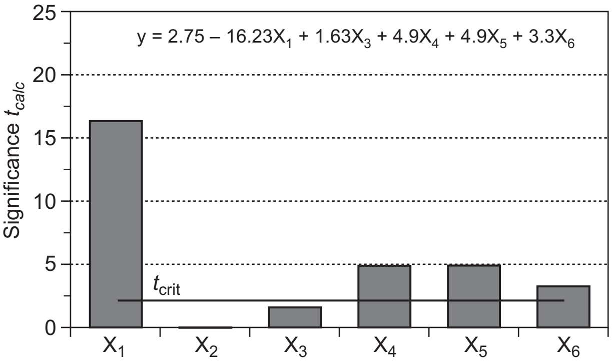

Figure 1. Factorial experiment results for apparent density with calculated regression equation.

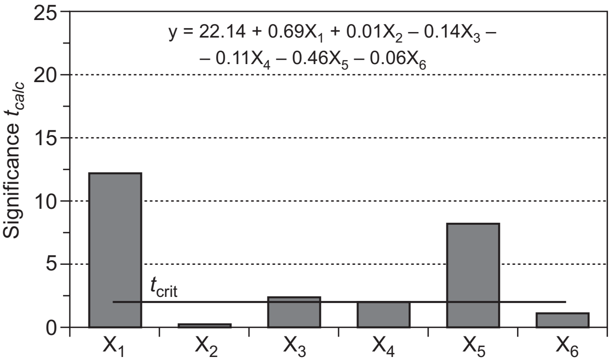

Figure 2. Factorial experiment results for open porosity with calculated regression equation.

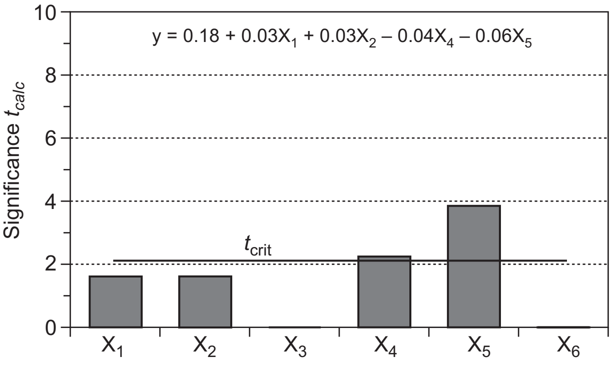

Figure 3. Factorial experiment results for permanent linear change with calculated regression equation.

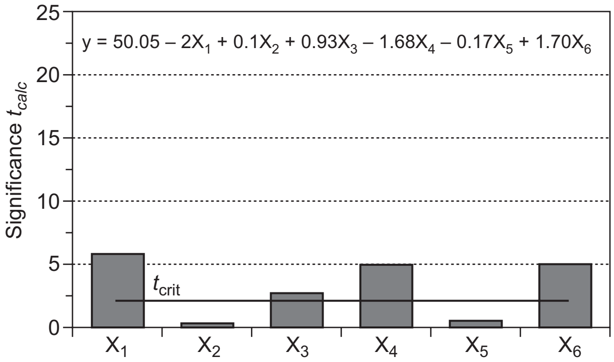

Figure 4. Factorial experiment results for Young's modulus with calculated regression equation.

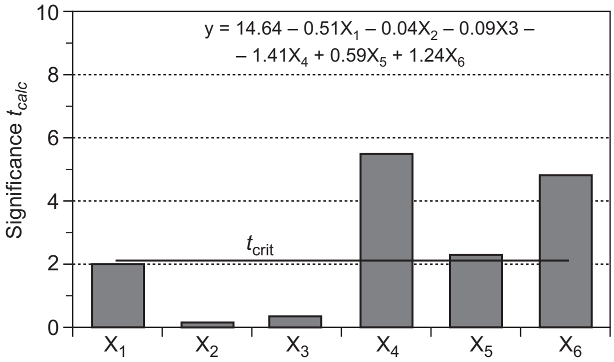

Figure 5. Factorial experiment results for bending strength with calculated regression equation.

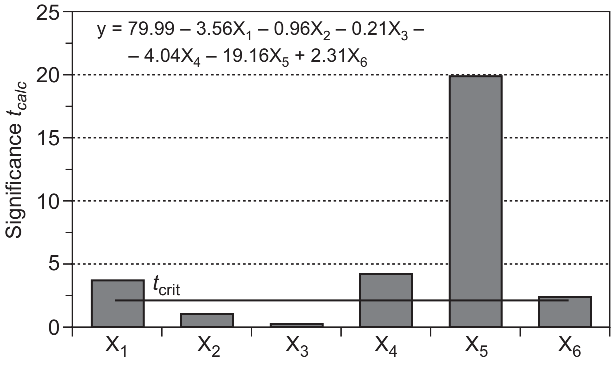

Figure 6. Factorial experiment results for compressive strength with calculated regression equation.

modulus and, to a small extent, the compressive strength at ambient temperature.

- The mixer rotational speed influences the open porosity of the investigated castable; however, this influence, similarly as that of vibration time, was at the significance borderline, influencing mainly Young's modulus.
- The vibration time influences the apparent density of the material equally significantly as the curing conditions. It is a factor that most significantly determined the bending strength at ambient temperature.

The curing conditions influence significantly the apparent density and the open porosity; they are a key factor for the permanent linear changes and the compressive strength at ambient temperature.

- The conditions of firing up to the temperature of 800°C influences the apparent density, Young's modulus and the mechanical properties at ambient temperature.

The mixing time is the only of the examined factors, whose change within the examined range did not have any significant influence on the determined service properties of the investigated castable.

Acknowledgements

The study was supported by the Institute of Ceramics and Building Materials, the Refractory Materials Division in Gliwice.

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