2^K FACTORIAL DESIGN APPLIED TO PORCELAIN STONEWARE TILE PROCESSING

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ABSTRACT

Several statistical techniques have been used successfully in recent years in the ceramic tile manufacturing process. One of the most useful of these techniques is 2^* factorial design. 2^3 factorial design was used in this study to quantify the influence of compaction pressure, maximum firing temperature, and duration of the firing cycle on linear shrinkage, water absorption, modulus of rupture, and pyroplastic deformation of four mixtures for the production of porcelain stoneware tiles.

1. INTRODUCTION

The production of ceramic tiles involves several variables in each stage of the productive process, related to raw materials and to equipment adjustments (mills, presses, kilns, etc.). The final product can only be produced efficiently, economically and in a controlled manner, with but minor losses, when proper adjustments are made in each stage. However, when any of the variables requires altering, the professional experience of an individual, which is based on non-statistical methods, this is the most commonly used resource. Factorial design is a highly useful tool for evaluating the influence of one or more factors on one or more properties, since it allows interactions among factors to be assessed. In other words, it allows combinations of the factors to be discovered unlike the combinations used routinely, which can originate the same properties (or improve them) and, at the same time, reduce production time or costs. At the very least, when the objective is not achieved, greater insight is gained into the influence of each evaluated factor on the property in question. Application of the technique requires that the response variables be approximately linear in the range under study, that the factors remain constant at predetermined values and that the experiments be conducted randomly.

2. METHODOLOGY

The technique was tested on four mixtures for porcelain stoneware tiles by means of 2³ design. The three factors of interest analyzed here were compaction pressure, P (500kgf/cm^2 and 667kgf/cm^2), firing cycle, C (55 min and 65 min), and maximum firing temperature, T (1200° C and 1225° C). Table 1 shows the experiments conducted in this study, while Table 2 lists the chemical compositions of the mixtures after firing and the residues on mesh 230 (63μ m) and 325 (45μ m).

Experiment	Pressure (kgf/cm ²)	Firing cycle (minutes)	Firing temperature (°C)
1	500	55	1200
t	500	55	1225
С	500	65	1200
tc	500	65	1225
р	667	55	1200
tp	667	55	1225
ср	667	65	1200
tcp	667	65	1225

Table 1. Experiments conducted.

		Residue									
Body	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	others	#230	#325
1	68.7	22.8	0.9	0.5	0.3	2.0	0.8	3.8	0.2	3.5	9.6
2	60.8	29.7	1.4	0.9	0.1	2.0	4.6	0.2	0.2	0.5	1.1
3	58.0	32.4	2.2	0.7	0.1	2.3	3.8	0.5	0.1	0.5	1.3
4	67.5	24.6	0.6	0.4	0.3	2.0	0.9	3.7	0.1	3.1	9.0

Table 2. Chemical analysis by X-ray fluorescence and residue of the mixtures under study (% in mass).

The mixtures were prepared by granulation with a moisture content of $6.0 \pm 0.1\%$ (dry base) on a 500 μ m sieve, uniaxial pressing of the samples in a 6cm x 2 cm die, and firing in a laboratory kiln, with a five minute soaking time. The fired samples were characterized as to the properties of interest, after which the effects of the variables (P, C and T) and of the interactions (TP, TC, CP and TCP) were calculated (Montgomery, 1984). The pyroplastic deformation was simply classified into three groups: 0 (absent), 1 (slight), and 2 (significant).

3. **RESULTS**

After the pieces made of mixtures 1 to 4 were pressed under 500kgf/cm^2 and 667kgf/cm^2 and dried, their bulk densities were, respectively, $1.96 \pm 0.01 \text{g/cm}^3$ and $1.99 \pm 0.01 \text{g/cm}^3$, $2.02 \pm 0.01 \text{g/cm}^3$ and $2.06 \pm 0.01 \text{g/cm}^3$, $1.97 \pm 0.01 \text{g/cm}^3$ and $2.02 \pm 0.01 \text{g/cm}^3$, and $1.91 \pm 0.02 \text{g/cm}^3$ and $1.99 \pm 0.01 \text{g/cm}^3$. Table 3 lists the results of the characterization of mixtures 1 to 4 with regard to water absorption (WA), linear shrinkage (LS), modulus of rupture (MOR) and pyroplastic deformation (ϵ). Each value listed represents the average of five samples tested under each condition. The effects of the alterations of the factors and of their interactions on the responses of interest are shown in Table 4, representing the average of the effect of the alteration of the factors).

EXP.	N	ліхт	URE 1		MIXTURE 2					TURE 3	MIXTURE 4					
	WA (%)	LS (%)	MOR (MPa)	ε	WA (%)	LS (%)	MOR (MPa)	ε	WA (%)		MOR (MPa)	ε	WA (%)		MOR (MPa)	8
1	1.47	7.8	39.3	0	0.82	8.1	62.8	0	1.09	7.9	55.4	0	1.86	6.4	29.4	0
t	0.69	7.9	36.4	1	0.37	8.3	53.2	0	0.47	8.9	101.4	1	1.20	7.8	62.2	2
с	0.86	7.2	36.8	0	0.74	7.8	60.6	0	1.61	7.7	41.9	0	2.82	6.5	51.4	0
tc	0.41	7.6	37.2	2	0.44	8.3	55.8	1	0.49	9.0	112.3	1	1.09	7.6	54.3	2
р	1.10	7.1	39.8	0	0.59	7.6	68.1	0	0.85	7.5	57.2	0	1.77	6.0	32.2	0
tp	0.69	7.2	37.3	1	0.29	7.7	58.4	0	0.42	8.1	105.2	1	0.99	6.9	65.1	2
ср	0.69	6.7	34.5	Ü	0.37	7.2	46.4	0	1.05	7.3	54.6	0	2.49	5.9	31.9	0
tcp	0.41	6.9	38.8	2	0.42	7.5	47.8	1	0.37	7.9	105.2	1	0.93	6.8	65.1	2

Table 3. Water absorption, linear shrinkage, modulus of rupture and pyroplastic deformation of mixtures 1 to 4.

F / I		EFFECTS														
	Mixture 1				Mixture 2					ture 3	Mixture 4					
	WA	LS	MOR	ε	WA	LS	MOR	ε	WA	LS	MOR	ε	WA	LS	MOR	ε
Р	-0.14	-0.7	0.2	0.0	-0.18	-0.6	-2.9	0.0	-0.24	-0.7	0.8	0.0	-0.20	-0.7	4.3	0.0
Т	-0.48	0.2	-0.2	1.5	-0.25	0.3	-5.7	0.5	-0.71	0.9	51.7	1.0	-1.18	1.1	30.5	2.0
С	-0.40	-0.4	-1.4	0.5	-0.03	-0.2	-8.0	0.5	0.17	-0.1	0.8	0.0	0.38	-0.1	-1.6	0.0
TP	0.14	-0.1	1.1	0.0	0.13	-0.1	1.5	0.0	0.16	-0.3	-1.8	0.0	0.01	-0.2	2.9	0.0
ТС	0.12	0.1	2.5	0.5	0.13	0.1	4.0	0.5	-0.19	0.1	4.1	0.0	-0.46	-0.1	-2.7	0.0
СР	0.05	0.1	-0.5	0.0	-0.02	-0.1	-8.2	0.0	-0.10	-0.1	-2.7	0.0	-0.05	0.0	1.1	0.0
ТСР	-0.05	-0.1	0.9	0.0	0.05	0.0	1.6	0.0	0.06	-0.1	-3.4	0.0	0.07	0.1	2.5	0.0

 Table 4. Effects of the factors (F) and the interactions (I) on water absorption, linear shrinkage, modulus of rupture and pyroplastic deformation of mixtures 1 to 4.

The results of the characterization of the mixtures and the calculation of the factors allow for the quantification of the effects of variations in the factors and interactions on the responses evaluated. Behaviours such as that displayed by mixture 4, which did not reach the necessary water absorption, could be optimized by increasing the compaction pressure (effect P), which reduces water absorption and linear contraction, in addition to increasing the mechanical strength without causing pyroplastic deformation. For this same mixture, a conventional solution of adjustment in the process would be to increase the firing temperature (effect T), which abruptly reduces water absorption and increases the tendency for pyroplastic deformation, reducing the mixture's workability.

4. CONCLUSIONS

This study based on factorial design shed new light on the behaviour of mixtures when subjected to changes in compaction pressure, duration of the firing cycle, and maximum firing temperature, requiring a minimum of experiments. In other words, in practical situations, the most convenient factor to be altered in order to achieve the desired objective is known, and the magnitude of the effect indicates the mixture's response to the adjustment of each factor.

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