REDUCING PORCELAIN TILE THICKNESS BY OPTIMIZATION OF MASS FORMULATION USING MIXTURE DESIGN

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This study aimed at reducing the thickness of porcelain tiles by optimizing the mass formulation methodology usinga simplex mixture design. Three raw materials (K-feldspar, Na-feldspar, plastic clay) were used as factors (independent variables) in order to determine their effect on each property under analysis (independent variables). The raw materials were characterized by XRF, XRD and particle size distribution (laser diffraction). The formulations were characterized by their water absorption, tensile strength by diametrical compression and pyroplasticity index. The results were analysed by ANOVA and were graphed in response surfaces. As a result, it was possible to obtain porcelain tileswith 50% reduction in thickness by optimizing the mass formulation methodology using mixture design, while maintaining the desirable characteristics of the final product.

1. INTRODUCCIÓN

In 2014 the Brazilian ceramic tile industry produced 903.3 million square meters, for an installed capacity of 1,084 million m^2. Total sales reached 922.4 10^6 m^2, 853.2 million square meters sold domestically and 69.2 million square meters were exported [1]. Brazil ranks second in production and consumption, only topped by China, which in 2012 produced 7.4 billion m^2 [1].Companies in the Brazilian market for ceramic tiles are divided into two distinct processes: the dry process and the wet one, where the first represents 73% of national production and the second 27% [1].The wet process is more common for the production of porcelain tiles. According to Anfacer, "in 2014, the production of porcelain tiles in Brazil was 93 million square meters, representing 10.7% of national production" [1].

For larger tiles (e.g., 1.00×3.00m^2 or 1.20×3.60m^2), an alternative pressing technology was proposed by Gozzietal. [3,4], which applies the screw press principle. In this process, thin ceramic sheets (3 mm thick) can be produced with high dry bulk density values(1,880–2,030 kg/m^3) that correspond to alow total green porosity(23–29%). Da Silva etal. [5] introduced tape casting as an alternative process for shaping porcelain tiles with thickness around 2 mm. For tape cast tiles, sintering reactions occur at lower temperatures, mechanical streng this higher and water absorption is lower when compared to pressed tiles with the same thickness

Therefore, this work was undertaken to optimize the ceramic formulation methodology using mixture design(DoE) to investigate how the technological properties of low-thickness porcelain tiles (response variables) are influenced by specific raw materials (input variables).To limit the scope of the work, only three raw materials were used: a plastic clay and two feldspars, in order to enable the use of simplex mixture design with only three factors, the raw materials. In this way, the effect of each raw material on the thickness reduction of porcelain tiles could be determined without any doubts.

2. METHODS

The selected raw materials were: Plastic clay (kaolinite), potassium feldspar and sodium feldspar. The selection of only three raw materials aimed to apply a simplex mixture design. A commercial plasticizer was used. Chemical (XRF), mineralogical (XRD, CuK_α (1.5405 Å) at 30 kV and 15 mA, 5 to 80° 2θ range and 0.017°/min step) and particle size (PSD, low angle laser diffraction, 0.2 to 500 µm) characteristics of the raw materials were determined. The raw materials were mixed according the mixture design (Table 1) and were dry ground for 30 min in laboratory ball mills (500 mL volume, high alumina grinders and jars). After milling, the mixtures were mixed with water (10 wt.%) and plasticizer (1 wt.%) and passed through a 500 µm sieve in order to granulate them. The powders were pressed in a laboratory hydraulic press at 50 MPa in cylindrical (Φ= 50 mm and h= 3 mm) and rectangular (100×30 mm^2) shapes. After shaping, the samples were dried at 110 °C for 24 h.

The thermal behaviour of the samples was determined by DSC/TG (20 to 1200 °C, 30 °C/min) and optical dilatometry (20 to 1200 °C, 30 °C/min). The samples were fired in a laboratory electric roller kiln at 1200 °C for 1 h cycle. After firing, the samples were characterized by water absorption, tensile strength by diametrical compression and pyroplastic deformation. Water absorption (%) was determined according ISO 10545/31997. The tensile strength (MPa) by diametrical compression was determined according ASTM D3967 2008, with modifications. Finally, the pyroplastic index (cm^{-1}) was determined

Mix	K feldspar	Na feldspar	Plastic clay	WA (%)	σ_{comp} (MPa)	PI (cm^{-1})
1	1	0	0	0.50	19.3	0.4239
2	0	1	0	0.04	14.3	0.6931
3	0	0	1	9.11	9.3	0
4	0.5	0.5	0	0.05	14.5	0.7129
5	0.5	0	0.5	3.64	13.1	0.2796
6	0	0.5	0.5	2.30	12.6	0.2053
7	0.66	0.17	0.17	0.02	11.0	0.4273
8	0.17	0.66	0.17	0.04	16.7	0.6123
9	0.17	0.17	0.66	5.19	12.4	0.1715
10	0.33	0.33	0.33	0.41	13.9	0.3919

Table 1. *Simplex mixture design and main results for water absorption (WA, %), tensile strength by diametrical compression (σ_{comp}, MPa) and pyroplastic index (PI, cm^{-1})*

3. RESULTS AND DISCUSSION

The plastic clay presents silica (66 wt.%), alumina (22 wt and iron and titanium oxides (2.5 wt.%). The loss on ignition (9 wt.%) is assigned to hydroxyls of the clay structure and to organic matter. The plastic clay is formed by kaolinite, quartz and muscovite mica. The potassium feldspar presents silica (70 wt.%), alumina (17 wt.%) and K_2O (9 wt.%). In turn, the sodium feldspar is a typical one, presenting silica, alumina and sodium oxide as main components. For the sodium feldspar, albite and quartz were identified and for the potassium feldspar, microcline, albite and quartz were identified, with a good match with the chemical composition. Before milling, the K feldspar showed 12 µm as average particle size, very similar to the sodium feldspar, with 11 µm as average particle size. The plastic clay presented an average particle size of 13 µm, higher than the feldspar, probably due the presence of quartz as contamination. The thermal behaviour of the raw materials and the central point of the mixture design were used to determine the firing conditions of the mixtures.

Regarding the mixture design analysis, Table 1 also shows the results for water absorption, tensile strength by diametrical compression, and pyroplastic index. For water absorption, using both feldspars the WA is lowered, yielding dense products with low water absorption and high shrinkage. The plastic clay acts in the opposite way during sintering, yielding porous products with higher water absorption and lower shrinkage, (99.95% reliability). For tensile strength determined by diametrical compression, the response surface shows that using any raw material – both feldspars and the plastic clay – the tensile strength is raised. This effect is higher when using the potassium feldspar (99.95% reliability). Finally, for pyroplasticity, the response surface shows that both feldspars raise the pyroplastic index and the plastic clay decreases it. This effect is higher when using the sodium feldspar. Sodium feldspar is a more intense flux than the potassium one and, therefore, the glassy phase is formed at lower temperatures for sodium feldspar and a larger amount of liquid is formed at a given temperature. This glassy phase has a low viscosity at high temperatures which causes the pyroplastic deformation of the samples. The reliability is very high, 99.98%.

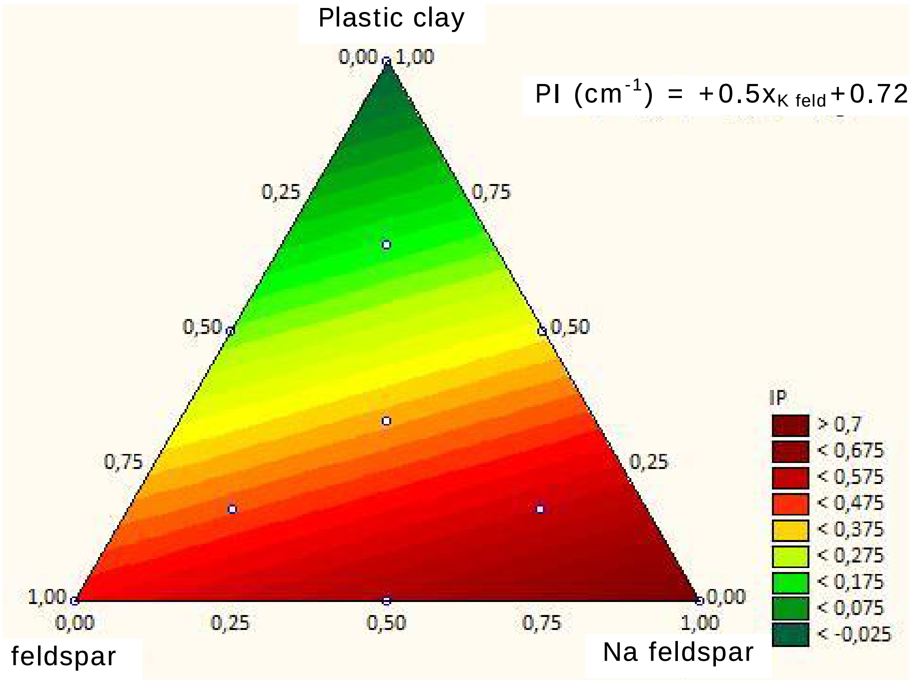

Figure 4. *Response surface for the pyroplastic index (cm^{-1})*

4. CONCLUSION

The large consumption of raw materials for the production of porcelain tiles and the need to produce tiles with the same characteristics using a smaller amount of raw material gave rise to this study. Using mixture design for the optimization of the formulation methodology it was possible to obtain low-thickness porcelain tiles while maintaining the desirable characteristics of the product. The response surface analysis for low-thickness porcelain tiles can help the understanding of the effects of each raw material on specific properties of the final product.

The water absorption (WA) results showed lower values for compositions (mixtures) with higher amounts of both feldspars. The same feature could be seen for the tensile strength due to the same reason: both feldspars act during firing as fluxes, improving the amount of liquid (glass) phase, therefore decreasing the porosity and raising the mechanical (tensile) strength. The results for pyroplasticity showed a greater tendency to pyroplastic deformation for mixtures with a higher amount of both feldspars, mainly for the sodium feldspar.

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