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Factorial Design for 3^2 Experimental Planning of Clay Ceramic Incorporated with Ornamental Stone Waste

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Abstract. In recent decade there has been a worldwide trend towards encouraging the recycling of solid wastes. Both environmental and economical issues motivate recycling of industrial wastes from practically all productive sectors. In particular, the ornamental stone industry generates a significant amount of wastes in the form of small rocks and sludge. Several works have investigated the possibility of incorporation of these ornamental stone wastes (OSW) into red clay ceramics. Promising results were reported but the optimum condition the terms of composition and firing temperature are still to be defined. Therefore, the present work investigated the best values for the main technical properties through an experimental planning using 3^2 factorial design associated with the incorporation of OSW into clay ceramics. The best combination of strength and water absorption was found for 12 wt% incorporation and firing temperature of 920ºC.

Introduction

Industrial wastes are becoming of worldwide concern due to environmental problems associated with pollution, health issues and climate changes. In particular the solid wastes generated by industries related to civil construction correspond to one of the largest amount faced today by our civilization. In fact, not only during construction and later in demolition [1] but also in the preliminary productive chain of steel, stone, cement, wood, ceramics, plastic and composites supplied for the sector, a huge amount of wastes is produced. A particular case is that of the ornamental stone industry, which supplies granite, marble and other stone in the form of facing tiles and lining plates. A considerable amount of ornamental stone wastes (OSW) is generated from the initial explosions in the quarry to the final sawing and polishing operation [2]. It is estimated that a total of 30 wt% of the processed stone becomes waste. The characteristics of this OWS permit its recycling in other sectors of civil construction such as mortar fabrication and ceramic production. Research works have been dedicated to investigate the recycling of OWS mainly through the incorporation into clay ceramics [2-14]. In general, this incorporation improves the properties. Saboya et al [15] found that a marble powder waste enhanced the strength and decrease the open porosity of clay ceramics fired up to 950ºC.

Despite the numerous works on the incorporation of OSW into clay ceramics, an evaluation of the optimized factors to provide the best properties is still to be accomplished. Thus, the objective of the present work was to conduct an experimental planning using the 3^2 factorial designs, which permit to obtain surfaces of response for the optimum values.

Materials and Methods

The basic materials used in the present work were a natural clay and an ornamental stone waste (OSW). The alluvium type of kaolinite clay was collected at the Sardinha Ceramic industry located in the municipality of Campos dos Goytacazes, state of Rio de Janeiro, Brazil. The OSW was obtained from a firm in the municipality of Cachoeiro de Itapemirim, state of Espirito Santo, at 100 km north of Campos dos Goytacazes. All tests an experiment were carried out at the Laboratory of Civil Engineering, LECIV, in the Center of Science and Technology, CCT, of the state University of the Northern Rio de Janeiro, UENF, in Campos dos dos Goytacazes.

The as-received materials were initially sieved, to 0.045 mm and dried in a stove at 110ºC for one hour. The OSW was then sieved to 60 mesh (0.250 mm) before incorporation the clay was prepared according to the Brazilian norm [16]. Incorporations of 0, 5 and 10 wt% of OSW into the clay were done in a mixer until homogeneous condition was reached. Prismatic specimens with dimensions of 117x210x170 mm were molded under pressure of one MPa. The specimens, minimum of 5 for each testing condition, were dried at 110ºC for 24 hours in a stone and then fired at 750, 850 and 950ºC in an electric muffle type of furnace.

The fired specimens were subjected to water absorption tests [17] and three points bend test [18] according to both ASTM [17,18] and Brazilian norms [19]. The bend tests were performed with a distance of 90 mm between support points in a model 5582 Intron machine operating at room temperature and crosshead speed of 0.5 mm/min. For the interpretation of results, the statistical software [20] was used considering the 3^2 factorial designs. The chemical analysis of the material was performed by X-Ray dispersive energy in a model EDX-700 Shimadzu spectrometer.

Results and Discussion

Table 1 presents the chemical composition of the clay and the OSW. In this table it should be noticed that the SiO_2/ Al_2O_3 ratio of 1.38 is above that characteristic of a kaolinite clay [21], which is 1.18. This means that additional SiO_2 should exist, mainly in the form of quartz. Moreover, the total SiO_2 + Al_2O_3 composition of 84.3 wt% indicates a refractory behavior. The red color of the fired clay ceramic is due to the relatively high amount of Fe_2O_3. It is also important to notice that the amount of K_2O, 3.17 at% as a flux during firing and forms low melting temperature eutectic phase that contribute to close porosity and reduce the water absorption.

Table 1. Chemical composition (wt%) of the clay and ornamental stone waste.

Material	SiO_2	Al_2O_3	Fe_2O_3	K_2O	TiO_2	SO_3	CaO	MnO	V_2O_5	ZrO_2	CuO	ZnO	SrO
Clay	48.83	35.46	8.87	3.17	1.62	1.13	0.54	0.14	0.10	0.03	0.03	0.03	0.01
OSW	50.09	17.60	12.47	3.70	1.83	1.56	12.39	0.19	0.55	0.03	-	0.02	0.04

As for the OSW composition, Table 1, the relatively high amount of SiO_2 indicates a marked participation of quartz. The amounts of K_2O and CaO also contribute to a fluxing action that close porosity and consolidate the fired ceramic structure.

Figure 1 combines the water absorption variation with the amount of OSW (a) as well as the factorial design surface of response (b) and iso-water absorption level curves (c). The water absorption results in Figure 1(a) shows an increase above the limit of 22% specified by the Brazilian norm [19] for 5 wt% OSW incorporation. However, with 10 wt% incorporation the ceramics comply to the norm for any firing temperature. The participation of fluxing components presented in Table 1 are responsible for this behavior. If one considers the factorial design evaluation in Fig1(b) and (c), the lowest water absorption, below the standard [19], is obtained for incorporation between 8-12 wt% and firing temperatures around 750-960ºC.

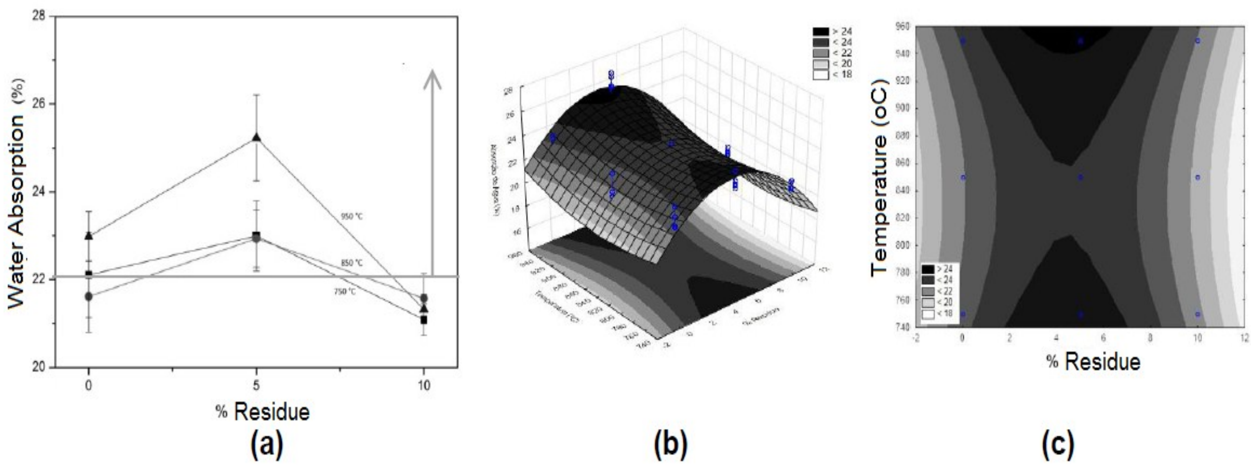

Fig. 1. Water absorption results: (a) variation amount of OSW and firing temperature; (b) surface of response; and (c) level curver.

Figure 2 combines the flexural strength variation with the amount of OSW (a) as well as corresponding surface of response (b), and iso-strength level curves (c).

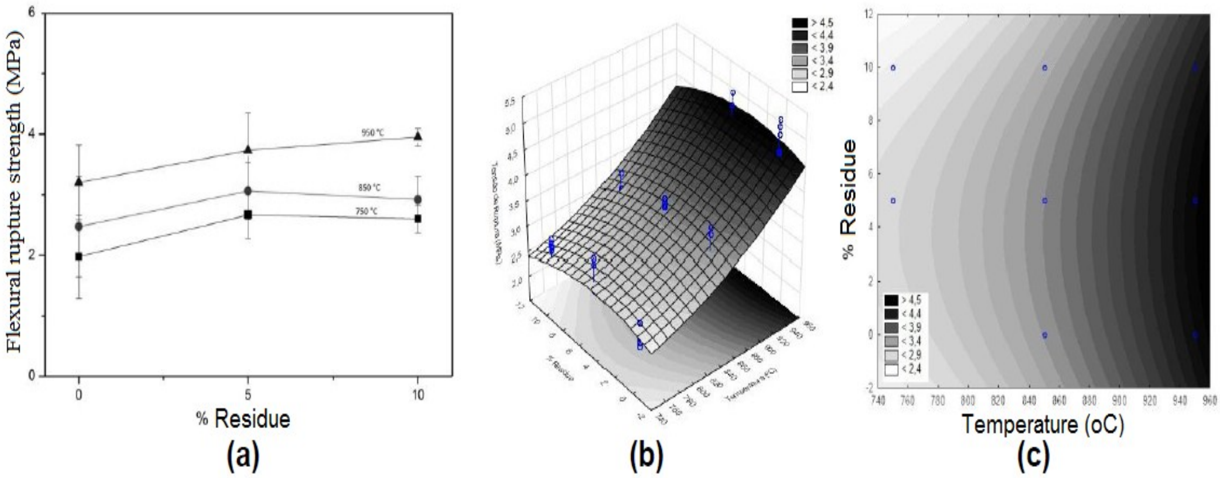

Fig. 2. Flexural rupture strength results: (a) variation with the amount of OSW and firing temperature, (b) surface of response; and (c) level curves.

As shown in Figure 2(a), there is a tendency to increase the strength both the amount of OSW and the firing temperature. This is certainly due to the effect of higher percentage of fluxing agents, Table 1, in the waste.

As indicated by the factorial design, through the surface of response, Figure 2(b), and level curves, Figure 2(c), the optimum results are associated with 8-12 wt% of OSW and firing temperatures in the range of 920-960ºC.

Conclusions

- Ornamental stone waste has a relatively higher content of fluxing compounds that contribute to enhance the properties of clay ceramic incorporated up to 10wt% and fired from 750 to 950ºC.
- The water absorption increases above the 22% maximum limit of the Brazilian standard for 5wt% waste incorporation. However for 10wt% incorporation the water absorption remained below the limit at all firing temperatures.
- The flexural strength tends to increase both with the amount of waste and the firing temperature. This is explained by the higher amount of fluxing compounds in the waste.
- The factorial design for 3^2 experimental planning indicates the optimum conditions to be a maximum of 12wt% of incorporated waste and ceramic firing at 960ºC.

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