

# Characterization of Different Clays for the Optimization of Mixtures for the Production of Ceramic Artifacts

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**Abstract** The *Baixada Campista* is a region located in the municipality of Campos dos Goytacazes and is known as an important pole producing ceramic artifacts in Brazil. The clay used in this production is usually extracted from different layers of the region itself undergoing a pretreatment process. The objective of this work was to characterize the different soils (A, B and C), from different layers of the reservoir, in order to characterize the different physical and chemical characteristics (granulometry, determination of Atterberg limits, moisture and chemical analysis). To optimize, using the simplex methodology, properties such as mechanical strength, porosity and linear retraction. The results pointed to an optimum proportion of the clays studied so that the properties meet the Brazilian technical norms, being the proportion of 30% of soil A, 30% of soil B and 40% of soil C well.

**Keywords** Clays · Ceramic artifacts · Simplex methodology

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## Introduction

The municipality of Campos dos Goytacazes, located in the northern region of the state of Rio de Janeiro, especially the region of the *Baixada Campista*, has one of the great highlights in its economy the red ceramic industry. Currently, according to data from the Campos Ceramists' Union (2015), the red ceramic industry that forms part of Campos dos Goytacazes industrial park consists of more than 100 unionized ceramics, which generate approximately four thousand direct jobs, with an estimated production of six million pieces per month, which makes the region the second largest Brazilian industrial park in red ceramics [1].

Study for characterization of clays in the Campos dos Goytacazes region showed different clays coming from fields located at the lower camped pole and performing chemical analysis, X-ray diffraction, differential thermal analysis, thermogravimetric analysis, particle size analysis and determination of physical-mechanical properties in test specimens molded with the studied ceramic masses [1]. The two authors concluded that clays found in the studied region are predominantly kaolinitic, and that can be used to manufacture massive bricks and ceramic blocks without problems. However, both authors concluded that for the manufacture of tiles and coatings, it is necessary to carry out a study in the formulation of the ceramic masses used, since for these types of materials the required minimum properties were not obtained or were very close to the limits established by norm [2].

Other studied showed of the ceramic masses of the Campos dos Goytacazes municipality, in order to verify the feasibility of the incorporation of residues for the production of ceramic artifacts clays used in this region [3]. One of the conclusions obtained by the authors is that it becomes evident the necessity of the planning of mixtures so that more satisfactory results are obtained in the properties of the studied ceramics [4].

More modern studies show the importance of experimental planning, since the outstanding studies did not succeed in their conclusions because of poor choice in the traits to be studied [5]. Proper planning would eliminate this problem. Researches shows faced the same problem in choosing the dosages to be studied in his work, since the author performed a little effective experimental planning, which caused some traces lost by the author [6]. In this context, the purpose of this work is to characterize three clay samples used in the Campos ceramics and mainly to use the simplex network blends planning to create a rational dosing methodology for the ceramic masses to be used in red ceramic artifacts [7].

The simplex method is a set of statistical planning and analysis techniques used in mathematical modeling of responses. It consists in varying the proportions of the components of a mixture, keeping its total quantity constant. Thus, the response value changes when changes are made to the relative proportions of the components [2]. The simplex space corresponds to the response surface for experimental data, or also to the response points of an experiment planning. Briefly, the method relates the parameters to the responses of the analyzed system.

In this work, three types of ceramic mass commonly used in the camper descent will be used, named by A, B and C. Therefore, a simplex network will be used in the triangular form, where each of the vertices represents one of the components that will be studied. To obtain the network response surface it is necessary to use one of the mathematical models available in the planning. For this work will be used the linear, quadratic and simple cubic models.

## Materials and Methods

Samples were collected and named as samples A, B and C. Samples were characterized by the following tests, which were the stages of sun drying, dewatering and homogenization:

- Granulometric Analysis;
- Specific grain mass, Liquidity and Plasticity limits;
- Identification of clay minerals with X-ray diffraction.

After the characterization phase of the materials, samples were prepared by varying the proportions of materials A, B and C in order to verify the most important technological properties for ceramic materials, namely: breaking stress and water absorption at temperature of 850 °C. Both tests are performed in accordance with Brazilian standards [8, 9]. The variation of the proportion of materials is defined according to the methodology proposed by simplex itself, where material A becomes associated with variable  $X_1$ , material B to  $X_2$  and the material C to  $X_3$ . It is noteworthy that other intermediate traits were proposed in order to statistically refine the model. The fractions studied and their respective nomenclatures are indicated in Table 1.

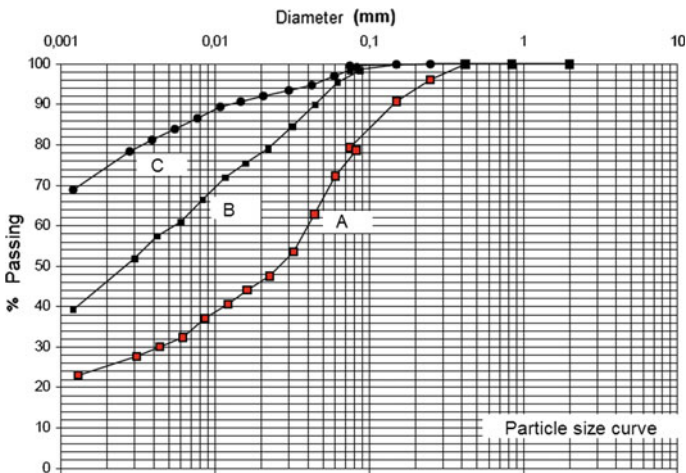
With the test specimens and the tests, it was possible to obtain the response surface through simplex network planning, which indicates the proportions of materials A, B and C should be used in order to obtain the best technological characteristics for the ceramic products.

## Results

The particle size distribution curve of samples A, B and C is shown in Fig. 1. Analyzing the distributions, it is easy to see that material A has a higher proportion of larger particles, whereas material C has the highest proportions of fine particles. This is of great importance for the planning of experimental mixtures, since, because they have different granulometry, the materials need to be compensated in order to obtain the mixture with the greatest possible packaging.

**Table 1** Nomenclature of the traits studied

Nomenclature	Proportion of material A (%)	Proportion of material B (%)	Proportion of material C (%)
$X_1$	100	0	0
$X_2$	0	100	0
$X_3$	0	0	100
$X_{12}$	50	50	0
$X_{13}$	50	0	50
$X_{23}$	0	50	50
$X_{123}$	33.33	33.33	33.33
$X_{1123}$	50	25	25
$X_{1223}$	25	50	25
$X_{1233}$	25	25	50
$X_{112}$	66.67	33.33	0
$X_{113}$	66.67	0	33.33
$X_{122}$	33.33	66.67	0
$X_{223}$	0	66.67	33.33
$X_{133}$	33.33	0	66.67
$X_{233}$	0	33.33	66.67



**Fig. 1** Granulometric distribution curve of samples A, B and C

The specific mass of the grains for the studied samples is presented in Table 2. It is extremely important to know these parameters, since it varies according to the mineralogical constituent of the soil particles. It is known that for most soils the value varies between 2.65 and 2.85 g/cm<sup>3</sup>, decreasing to soils containing high organic matter content, harmful to the manufacture of ceramic pieces, and increased

**Table 2** Parameters found for samples

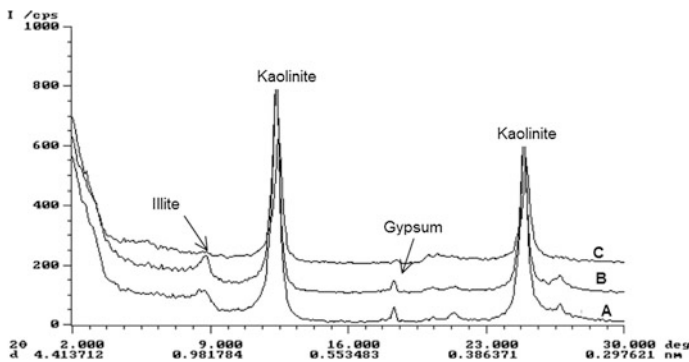
Sample	Specific mass (g/cm <sup>3</sup> )	LL (%)	LP (%)	IP (%)
A	2.68	44	27	17
B	2.65	64	28	36
C	2.62	76	37	39

to soils rich in oxides of iron. In the case of the samples studied, the specific mass value did not show significant variations, being in a range of 2.65 g/cm<sup>3</sup>.

The limits of Atterberg, which correspond to the limit of liquidity and plasticity limit, indicate parameters of soil workability. The liquidity limit (LL) can be defined as the moisture content for which the soil passes from the plastic state to the liquid state while the plasticity limit (LP) indicates the moisture where the soil begins to fracture when mold it. It is extremely important to know these values of moisture so that in the molding of the ceramic pieces, no excess water or scarcity is used [3]. The limits found for the studied samples are shown in Table 2, where it is possible to see that soil A presents lower plasticity parameters, while soil C can be considered the most plastic among the studies. It is important to note that the use of a tool that performs the planning of the mixtures of the three samples studied is essential to find the trait that will provide the best technological properties for the ceramic pieces to be manufactured.

Analyzing Fig. 2, which demonstrates the X-ray diffractograms for the three samples studied, it is apparent that the predominant clay in the samples studied is kaolinite. This fact bought what the bibliographical already predicted, according to works of Alexandre (2001) and Vieira (2000). It is of great interest to prove the predominance of kaolinite clay, because as widely published by the bibliographical, since this clay is heat treated around 550 °C, metacaulinite is formed, a compound of great interest for presenting excellent technological parameters for ceramics [10].

After the mechanical strength and water absorption tests were carried out for the specimens burned at 850 °C, the data and results obtained were treated in a simplex



**Fig. 2** X-ray diffraction of samples A, B and C

**Table 3** Mechanical resistance of the models studied at 850 °C

Nomenclature	Observed value (kgf/cm <sup>2</sup> )	Linear model (kgf/cm <sup>2</sup> )	Quadratic model (kgf/cm <sup>2</sup> )	Cubic model (kgf/cm <sup>2</sup> )
X <sub>1</sub>	17.19	17.19	17.19	17.19
X <sub>2</sub>	44.77	44.77	44.77	44.77
X <sub>3</sub>	60.27	60.27	60.27	60.27
X <sub>12</sub>	31.30	30.98	31.30	31.15
X <sub>13</sub>	27.77	27.80	27.77	27.77
X <sub>23</sub>	52.50	52.50	52.50	52.50
X <sub>123</sub>	34.10	40.74	35.88	34.10
X <sub>1123</sub>	23.30	28.96	24.17	23.29
X <sub>1223</sub>	40.60	42.75	41.56	40.63
X <sub>1233</sub>	42.20	50.50	45.62	44.66
X <sub>112</sub>	28.60	26.38	26.50	26.60
X <sub>113</sub>	23.50	31.54	21.84	21.88
X <sub>122</sub>	28.60	35.57	35.63	35.69
X <sub>223</sub>	49.30	49.93	49.93	49.93
X <sub>133</sub>	36.70	45.91	36.14	36.12
X <sub>233</sub>	50.40	55.10	55.10	55.10
Average waste		4.84	2.24	1.91

methodology. Tables 3 and 4 present the results for mechanical resistance and water absorption at the burning temperature of 850 °C respectively. Figures 3 and 4 present the response surface that best suited the parameters mechanical strength and water absorption, respectively. For the mechanical resistance parameter, the model that best fit was the cubic, as can be observed by the mean of the residues. For the parameter water absorption and specific mass, the quadratic model provided a very low average of residues, and therefore the cubic model was not calculated for this parameter. It is also worth noting that the parameter water absorption was better adjusted with the linear model, as seen by the average of the residues [11].

The calculations in Table 3 were performed applying the expressions described below that came from applying the simplex methodology.

$$y_{linear} = 17.19 * X_1 + 44.77 * X_2 + 60.27 * X_3$$

$$y_{quadratic} = 17.19 * X_1 + 44.77 * X_2 + 60.27 * X_3 - 44.22 * X_1 * X_3$$

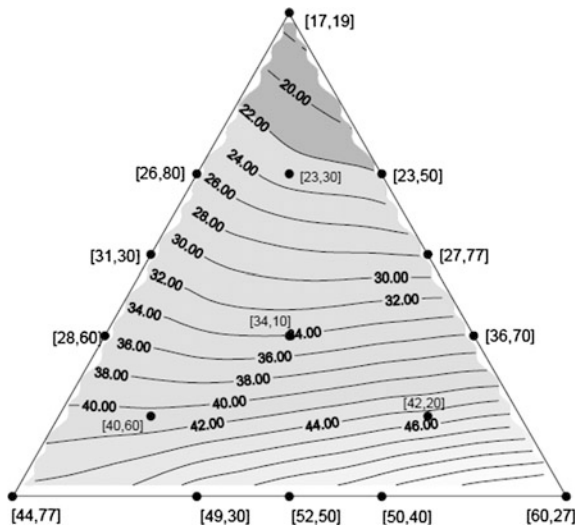
$$y_{cubic} = 17.19 * X_1 + 44.7 * X_2 + 60.27 * X_3 - 44.22 * X_1 * X_3 - 52.17 * X_1 * X_2 * X_3$$

The calculations in Table 4 were performed applying the expressions described below that came from applying the simplex methodology. For this parameter, the cubic model was not calculated, since the linear model presented greater adjustment than the quadratic model.

**Table 4** Water absorption of the models studied at 850 °C

Nomenclature	Observed value (%)	Linear model (%)	Quadratic model (%)
X <sub>1</sub>	19.86	19.86	19.86
X <sub>2</sub>	23.10	23.10	23.10
X <sub>3</sub>	25.20	25.20	25.20
X <sub>12</sub>	21.90	21.49	21.49
X <sub>13</sub>	23.40	22.54	23.39
X <sub>23</sub>	24.90	24.18	24.89
X <sub>123</sub>	22.96	22.73	23.44
X <sub>1123</sub>	20.80	21.30	21.76
X <sub>1223</sub>	23.00	22.93	23.35
X <sub>1233</sub>	24.70	23.98	24.67
X <sub>112</sub>	20.80	20.94	20.94
X <sub>113</sub>	20.90	21.64	22.41
X <sub>122</sub>	20.60	22.04	22.04
X <sub>223</sub>	21.80	23.82	24.47
X <sub>133</sub>	22.60	23.44	24.19
X <sub>233</sub>	23.40	24.53	25.16
Average waste		0.70	1.09

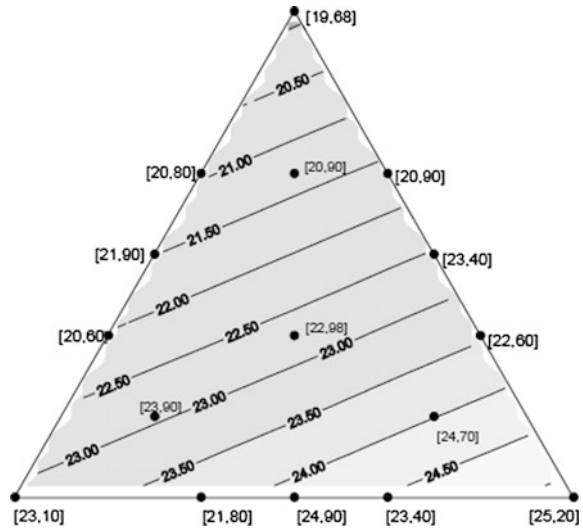
**Fig. 3** Surface of response of the simple cubic model for the parameter mechanical resistance



$$y_{linear} = 19.86 * X_1 + 23.13 * X_2 + 25.23 * X_3$$

$$y_{quadratic} = 19.86 * X_1 + 23.13 * X_2 + 25.23 * X_3 + 3.77 * X_1 * X_3 + 2.60 * X_2 * X_3$$

**Fig. 4** Response surface of the linear model for the parameter water absorption



Based on the results obtained by the tables and the response surfaces of the simplex, the effectiveness of the simplex network planning for the analysis and prediction of the technological parameters of the ceramic pieces is proved. By analyzing the optimal regions obtained, it is easy to see that material C contributes most to mechanical strength, while material A helps in reducing water absorption, while material B exhibits intermediate behavior between the two. A ceramic material with good technological properties will be obtained if the correct proportion between these materials is realized. Several traces can be used, being obtained through the equations resulting from the simplex methodology. A possible trace would be 30% of A, 30% of B and 40% of C, which would result in values of resistance and water absorption compatible with the recommended values [2, 11].

## Conclusion

After the study of the methodology of clay samples in simplex network can be concluded:

- The characterization of samples A, B and C of clayey soils typically used in camellary ceramics demonstrates what has been studied by researchers such as Alexandre (2000) and Vieira (2001), that the clays of the region are typically kaolinitic and have a great granulometric variation and the Atteberg boundaries.
- Because of this large size range and liquidity and plasticity limits, it is necessary to use some kind of mix planning in order to obtain better technological parameters for the ceramic materials produced.



- The use of planning in simplex networks allows a better interpretation and prediction of these parameters using mathematical and statistical models, as observed when analyzing the mixture of samples A, B and C and to analyze the mechanical resistance and water absorption properties.
- The effectiveness of using simplex modeling to predict the technological parameters of ceramics is effectively proven in this way.

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