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Determination of expandability clay from San José de Cucuta metropolitan area, by varying calcination temperature and time

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Abstract. In this article, the volumetric change of the clay granules and sewage sludge (technological nutrient), San José de Cúcuta metropolitan area raw materials, when calcined at different temperatures and different burning times is shown. The main objective is to evaluate out the work right conditions (temperature and time), for the production of light aggregates from expanded clay. The methodology that was followed consists of three phases: preparation and pelletization of clay mixtures and technological nutrient; application of heat treatment in a set of muffle furnaces; and application of statistical treatment with the help of Software R. One of the most important results is that the expansion of the clay is indifferent to the time it takes to cook the pellets, and that this property depends solely on the temperature of burning. The temperature that generates the greatest expansion is 1,200 Celsius degrees, using a set of muffle furnaces.

1. Introduction

Lightweight aggregates are defined as natural or artificial materials, which are granular, porous and light [1]. The light aggregate from expanded clay is a product used in large quantities of buildings, due to its less weight properties than conventional aggregates, high strength to compression, thermal resistance, acoustic insulation, and durability [2].

The lightweight aggregates raw material must contain substances that develop gas when heated and, at the same time, the material must be transformed into a highly viscous plastic mass capable of expanding due to gas entrapment [3]. Technological nutrients are used to maximize the expansion capacity of the clays, which are industrial wastes, which are added in a relatively small amount. To carry out the study of this article, 20% of the technological nutrient was added to the clay samples [4].

In the literature there are many studies on the parameters that affect the expansion of clays to produce light aggregates of expanded clay, as well as the investigations carried out by: Decler J and Viaene W 1993 [5]; Bragdon 1996 [6]; Fragoulis et al 2004 [7]; Fakhfakh, *et al.* 2007 [8]; Ozguven, 2009 [9]; Bartolini et al 2010 [10] who present methodologies and conditions that clays must contain to be precursors of lightweight aggregates. However, the studies carried out to determine the expansive behavior of the clays by varying the temperature and cooking time of the clay granules are scarce, since only reference the research carried out by Ozguven 2009 [9].

For this reason, it is intended investigate, what will be the variation in the volume of clay pellets and technological nutrient, when being calcined at temperatures between 900 °C and 1200 °C, with



times of 5, 10, 15 and 20 minutes, by using a program statistical analysis of experimental design (R software) [11], with which it will look for the conditions of temperature and suitable time for the elaboration of light aggregates of expanded clay.

On the other hand, the clay of the San Jose de Cucuta metropolitan area has essential chemical properties to generate expansion, however, it does not show expansion since it does not contain organic matter that generates increase in the aggregates, so it was necessary to include the clay mixture 20% of technological nutrient, which in this case is sewage sludge.

2. Methods and materials

2.1. Methods

The realization of the experiments establishes three phases: first, preparation and pelletization of clay mixtures and technological nutrient; second, application of heat treatment in combination of muffle furnaces; and third, application of statistical treatment with the help of Software R. The consecutive processes of the experiments are shown in Figure 1.

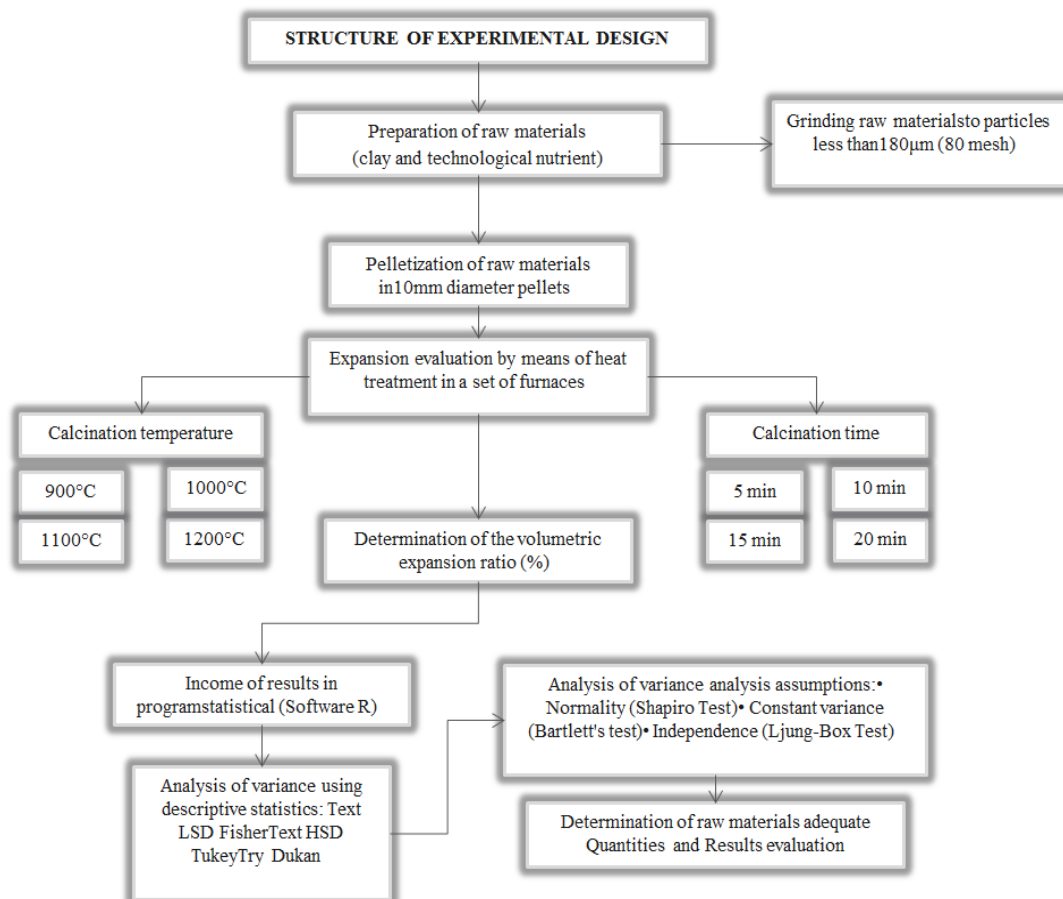


Figure 1. Flowchart of the design of experiments.

2.2. Procedure

The raw materials were pelleted into spherical granules with sizes of approximately 10 mm in diameter, using mixtures of clay with 20% technological nutrient. The pellets were dried at 105 °C for 12 hours, and then, according to Loutou M. (2013) [12], Berg (1970) [13] and Decler (1993) [5], who recommend heat treatment preheat at 600°C for 5min and then enter at working temperature (900 °C, 1000 °C, 1100 °C and 1200 °C) for the stipulated time of 5, 10, 15 and 20 minutes. Five replications

were made with five granules, a process carried out in a set of Muffle furnaces. The volumetric expansion ratio was calculated with the equation $(V_{exp}/V_i) \cdot 100$, where V_i is the volume before the burning process and V_{exp} is the volume after the burning of the granules.

The objective is to find out which of the treatments (varying temperature and time) gives more expansion in the clays when being calcined. 16 treatments are generated, whose factors are; Temperature and Time. The analysis of the results is done with the help of Software R (experimental design analysis program) [11]. The study carried out with the computer program is an analysis of variance, and when this presents a difference between the treatments applied, a comparison is made between them, using the Test LSD of Fisher, Test HSD of Tukey and the Dukan Test. In addition, checks are made to ensure that the experiment and procedure do not have significant errors that could cancel the study, for them it is verified that the assumptions of the analysis of variance are fulfilled: Normal assumption (Shapiro test); constant variance assumption (Bartlett test); Assumption of Independence (Ljung-Box Test).

2.3. Materials

The raw materials used are; a clayey material from San José de Cúcuta metropolitan area, and sewage sludge (technological nutrient). The materials were crushed until their particles were less than 180 μm in size. The technological nutrient works as an additive that maximizes the capacity of expansion of clays, this is added with an amount of 20% of the total sample, by weight. The chemical characteristics of the raw materials were identified by means of an X-ray fluorescence analysis and are presented in Table 1.

Table 1. X-ray fluorescence analysis of raw materials (FRX).

Compound	Clay (% in weight)	Technological nutrient (% in weight)
SiO ₂	57.44	52.89
Al ₂ O ₃	22.97	31.52
Fe ₂ O ₃	10.339	8.82
SO ₃	3.28	1.45
K ₂ O	1.47	2.72
TiO ₂	1.36	-
MgO	1.32	0.67
CaO	1.08	1.25
Na ₂ O	0.54	0.15
P ₂ O ₅	0.1	0.45
MnO	0.05	0.09

2.4. Equipment used

The equipment that was used to calcine clay mixtures and technological nutrient was a set of muffle furnaces from the Materials Science Laboratory of the Universidad Nacional Experimental del Táchira (UNET), Táchira, Venezuela.

3. Results: Statistical analysis

The volumetric expansion ratio, for each replica of the treatments, varying temperature and calcination time.

3.1. Analysis of variance using Software R

The model based on the regression coefficients is statistically significant at the level of confidence 95% ($\alpha = 0.05$). For the model developed, the factor Time is* $P = 0.09447 > \alpha = 0.05$, that implies that the null hypothesis must be accepted, which means that there are no statistically significant differences in the ratio of volumetric expansion of the mixture of clay and technological nutrient with different calcination times. On the other hand, for the developed model, the factor Temperature is* $P = 2e-16 <$

$\alpha = 0.05$, that implies that the null hypothesis must be rejected, which means that there are statistically significant differences in the relationship of volumetric expansion of the mixture of clay and technological nutrient with different temperatures of burning.

With the data of the previous paragraph it is deduced that, the change of volume of the pellets with mixtures of clay and technological nutrient, takes place when happening the granules of a temperature of preheating of 600 °C for 5 minutes, at the stipulated working temperatures (900 °C, 1000 °C, 1100 °C and 1200 °C), producing expansion at temperatures of 1100 °C and 1200 °C, while, at temperatures of 900 °C and 1000 °C, there is a contraction or reduction in the volume of the pellet (see Figure 2 (a)). On the other hand, the change in size of the pellets with varying sintering times, does not present a significant difference between the different treatments (see Figure 2 (b)), demonstrating that the increase or decrease in the size of the pellets depends of a thermal shock, and that the calcination time affects other characteristics, which were not the focus of study in the research carried out.

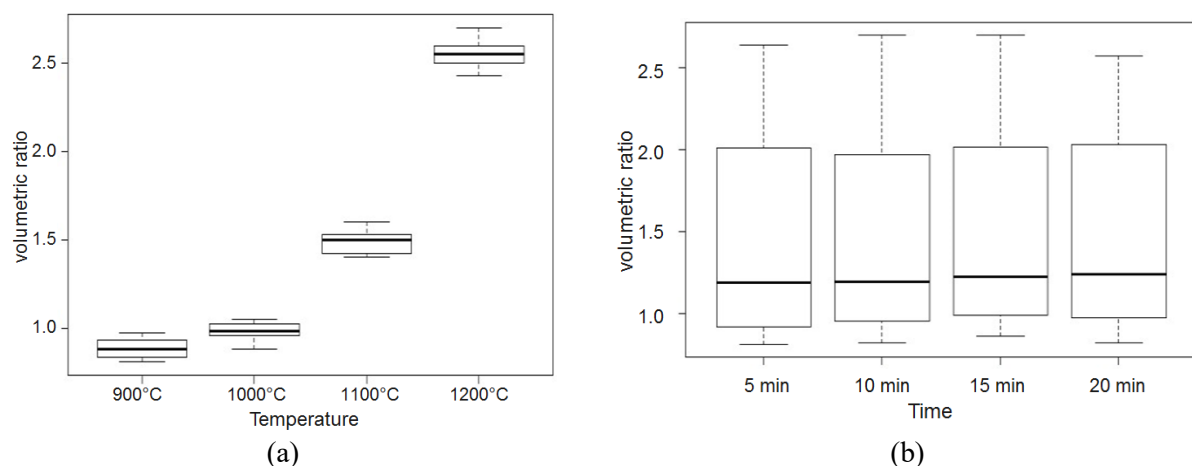


Figure 2. (a) Volume chart of volumetric expansion ratio with different calcination temperatures (b) Volumetric expansion volume case graph with different calcination times.

For the model developed, the interaction of the factors Time: Temperature (Figure 3) the value of significance is $* P = 0.1822 > \alpha = 0.05$ implying that the null hypothesis must be accepted, inferring that, there is no statistically significant interaction between the time and the calcination temperature, in the ratio of volumetric expansion of the mixture of clay and technological nutrient, that is to say, the increase or decrease of the size in volume of the granules, only depends on the temperature at which it is sintered, no matter how long it stays at a certain temperature.

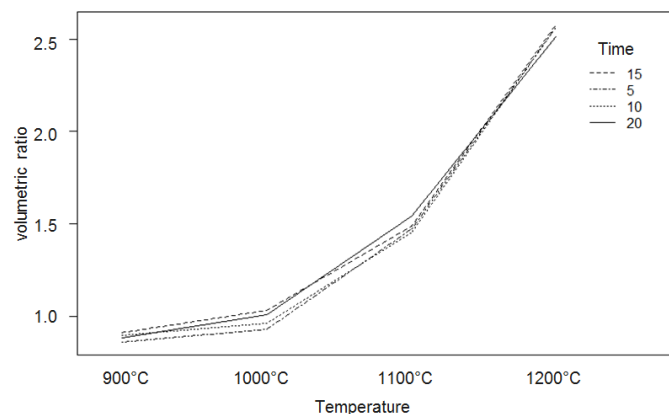


Figure 3. Interaction of different temperatures versus volumetric expansion ratio at different calcination times.

When the null hypothesis for the factor Time is accepted, a statistical analysis is performed with the Test LSD of Fisher, Test HSD of Tukey and the Dukan Test, for a single factor: Temperature, in order to identify which of the temperatures (900 °C, 1000 °C, 1100 °C and 1200 °C) there is greater expansion.

The Test LSD of Fisher, the Test HSD of Tukey and the Dukan Test present an average square error (mean square error) of 0.0040117, a value that is close to zero, that is, there are no errors in the experiments, statistically speaking, in addition, the maximum expansion is presented in treatment 4 (see Table 2). Fisher's Test LSD shows that the smallest significant difference is 0.03989 and Tukey's Test HSD shows the least significant difference is 0.05261.

Table 2. Statistical analysis results by Fisher's Test LSD, Tukey's Test HSD and the Dukan Test, in the volumetric expansion ratio.

Treatment	Expansion	std	r	LCL	UCL	Min	Max
1	0.8865	0.05603	20	0.85829	0.914708	0.81	0.97
2	0.9825	0.04598	20	0.95429	1.010708	0.88	1.05
3	1.4890	0.06624	20	1.46079	1.517108	1.40	1.60
4	2.5540	0.08003	20	2.52579	2.582208	2.43	2.70

3.2. Assumptions of the analysis of variance

The Shapiro test gives $* p = 0.2223 > \alpha = 0.05$ indicating that the residual errors, of the volumetric expansion ratio at different temperatures, have the behavior of a normal distribution, statistically speaking. Bartlett's test results $* P 0.1069 > \alpha = 0.05$ indicating that the residuals of the volumetric expansion ratio at different temperatures, they have constant variance, statistically speaking (see Figure 4).

The Ljung-Box test results $* P 0.4107 > \alpha = 0.05$ indicating that the residuals of the volumetric expansion ratio at different calcination temperatures are independent, statistically speaking (Figure 5).

It is important to highlight that the statistics applied for the verification of the tests carried out for the design of experiments, is intended to rule out the errors that may be generated, and thus present a reliability in the results obtained.

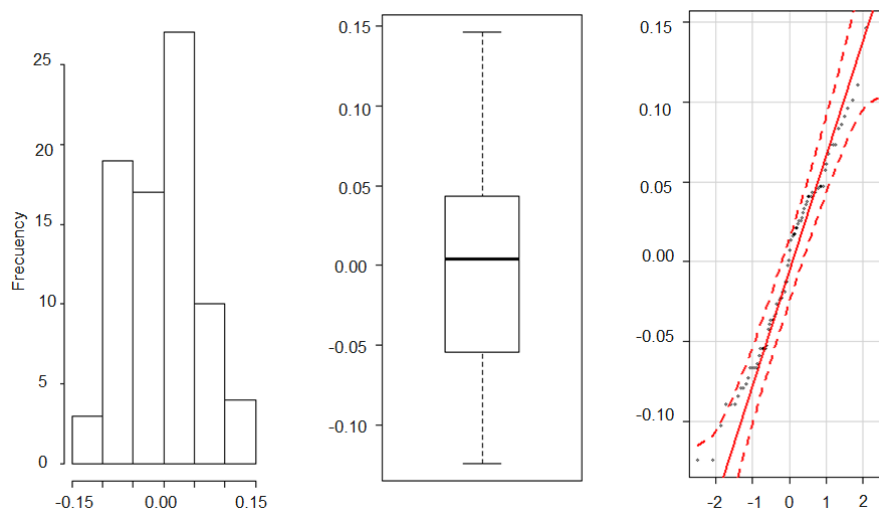


Figure 4. Residual models of the volumetric expansion ratio for the different temperatures.

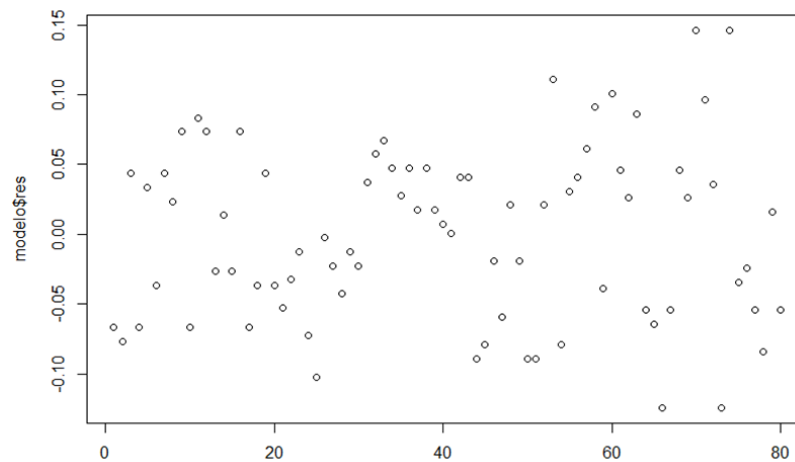


Figure 5. Residuals against the order of the volumetric expansion ratio for the different calcination temperatures.

4. Conclusions

Treatment 4, that is, when calcining a mixture of clay and waste water mud with a temperature of 1200 °C, generates the highest volumetric expansion ratio, with an expansion of 2.55 times the initial size of the granules.

Calcining the samples at temperatures of 1100 °C and 1200 °C, produce expansion, while, at temperatures of 900 °C and 1000 °C, there is a contraction or reduction in the volume of the pellet. In addition, the change in the size of the pellets with varying sintering times does not show a significant difference between the different treatments, demonstrating that the increase or decrease in the size of the granules they depend only on a thermal shock.

The evaluation of the interaction of the factors time: Temperature, implies that the null hypothesis must be accepted, deducting that, there is no statistically significant interaction between the time and the calcination temperature, in the volumetric expansion ratio of the clay mixture and technological nutrient.

Fisher's Test LSD, Tukey's Test HSD, and the Dukan Test present an average square error of 0.0040117, indicating that there are no errors in the experiments, statistically speaking, for different temperatures of work.

The Shapiro test results in the residual errors of the volumetric expansion ratio of the clay mixtures having the behavior of a normal distribution; The proof of Bartlett results in that the residuals of the volumetric expansion ratio of the clay mixtures have constant variance; The Ljung-Box test results in that the residuals of the volumetric expansion ratio of the clay mixtures have independence, statistically talking.

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