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Determination of expandability clay from San José de Cúcuta metropolitan area, by adding different percentages of sewage sludge

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Abstract. This article shows the research carried out on clay from the San José de Cúcuta metropolitan area, Colombia, to which was added several percentages of a wastewater sludge, which is called as a technological nutrient. The objective of the study is to find out with what percentage of technological nutrient the capacity of expansion of the granules is maximized. The methodology that was followed consists of four phases; preparation of raw materials; pelletizing of raw materials in granules of 5 to 10 millimeters; application of heat treatment in a rotary kiln at the pilot plant level; and application of descriptive statistics for analysis of results. It was determined that the mixture with a 20% of technological nutrient, produces the highest volumetric expansion ratio, with 1.56 times its initial size, while, when adding 30% of the nutrient the same expansion occurs when adding an 10% nutrient, which is 1.07 times the initial size of the particle.

1. Introduction

The raw materials used for the production of lightweight aggregates (LWA), are natural minerals such as clays, slate and shales, to which are added various by-products or industrial wastes such as fly ash, blast furnace slag, among others [1]. The aggregate of expanded clay is a product used in a large number of constructions, due to its properties of light weight, high strength, thermal and acoustic insulation, and durability [2]. To maximize the clays' expansion capacity, commercially technological nutrients are used, which are by-products or industrial wastes, which are added in a relatively small amount, predominantly in the mixtures, the clayey material.

Several studies have been carried out on the use of technological nutrients in the manufacture of lightweight aggregates, as well as: C.R. Cheeseman and G.S. Virdi (2005), who found that sewage sludge ash granules (SSA) sintered over a range of temperatures, have lower densities than light commercial aggregates (Lytag) and low water absorption when subjected to temperatures between 1050 °C and 1080 °C, approximately [3]. Similarly, Ing-Jia Chiou, Kuen-Sheng Wang, Ching-Ho Chen, Ya-Ting Lin (2006), used SSA, with characteristics similar to clay and sewage sludge (SS) as a mixture to sinter aggregates light, studying the influences of the composition of the raw material in the granulation and sintering, obtaining that both the SS and the SSA could be sintered to produce synthetic aggregates individually or mixed [4].

Another technological nutrient investigated is the bottom ash from the solid waste incinerator, studies that have been carried out by: C.R. Cheeseman, A. Makinde, S. Bethanis (2005) [5], Binyu Zhang, Chi Sun Poon (2015) [6], and Ciarán J. Lynn BE (2016) [7].

For the realization of the present article, an investigation was carried out about the maximization of the capacity of expansion of a clay of the San José de Cúcuta metropolitan area, Colombia, when adding several percentages of technological nutrient, which is a sewage sludge, extracted from settler of a drinking water treatment plant. In addition, to apply a statistical treatment, to validate all the data obtained in the experiments.

2. Methods and materials

2.1. Methods

For the development of the research, an experimental design was carried out consisting of four stages: first, preparation of raw materials; second, pelletization of raw materials in granules from 5 to 10 mm; third, application of thermal treatment in a rotary kiln at scale; and fourth, application of descriptive statistics for analysis of results (see Figure 1).

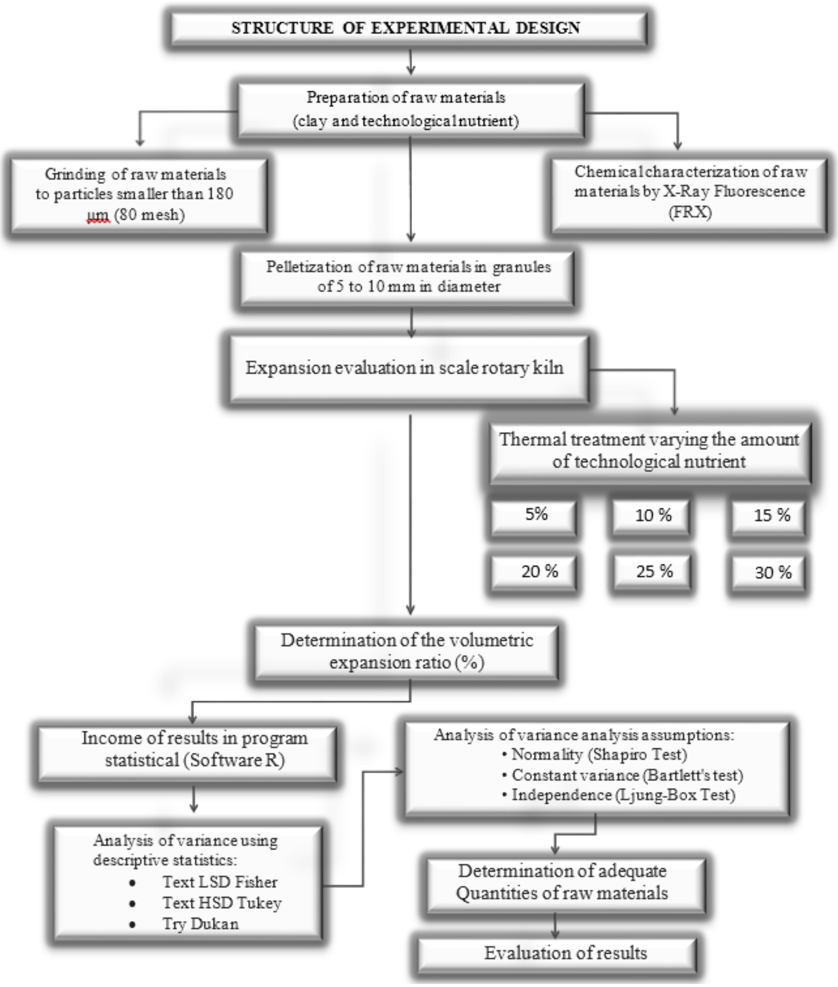

Figure 1. Flow chart with methodology followed in the investigation.

2.2. Procedure

Prepared raw materials were wetted and pelleted into spherical granules with sizes between 5 mm and 10 mm in diameter, using clay mixtures with different percentages of nutrient technological. The moisture was removed from the pellets in a dryer at 105 °C for 12 hours, then then a pre-treatment was carried out at a temperature of 600 °C in 5 minutes, according to Loutou, M. (2013) [8], Berg (1970) [9] and Decleer (1993) [10,12], and subsequently heated up immediately at a temperature of 1050 °C, in a rotary kiln at pilot plant scale. The volumetric expansion ratio was calculated with the equation (Vexp/Vi)*100 where, Vi is the volume before the burning process and Vexp is the volume after the burning of the aggregates.

Six different percentages of technological nutrient are added to a sample of clay found in the San José de Cúcuta metropolitan area, Colombia. The objective is to find out which of these additions gives the greatest expansion in the clays when they are calcined in a rotary kiln at scale with a temperature of 1050 °C. Six treatments are generated (six clay mixtures with different percentages of nutrient), whose factor is; types of clay mixture, for which 5 replications were made for each treatment (Table 1).

Table 1. Types of mixtures made.

Treatments	Clay percentage	Sewage sludge percentage
1	95%	5%
2	90%	10%
3	85%	15%
4	80%	20%
5	75%	25%
6	70%	30%

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 met: Normal assumption (Shapiro test); constant variance assumption (Bartlett test); Assumption of Independence (Ljung-Box test).

2.3. Materials

We used raw materials with a specific chemical composition (see Table 2), a sample preparation protocol was established, grinding the clay and the technological nutrient, until its particles were less than 180 μm, that is, passing the 80 mesh.

Table 2. X-ray fluorescence analysis of raw materials (FRX).

Compound	Clay (% in weight)	Technological nutrient (% in weight)
SiO_2	57.44	52.89
Al_2O_3	22.97	31.52
Fe_2O_3	10.339	8.82
SO_3	3.28	1.45
K_2O	1.47	2.72
TiO_2	1.36	-
MgO	1.32	0.67
CaO	1.08	1.25
Na_2O	0.54	0.15
P_2O_5	0.1	0.45
MnO	0.05	0.09

2.4. Equipment used

The equipment used in the calcination of the pellets, is a rotational furnace at the scale of a pilot plant, designed and built by the author. The oven has a length of 3 m, with an effective diameter of 20 cm and rotates at a rate of 5 rpm counter clockwise. The internal temperature is controlled with an infrared pistol (pyrometer), with an emissivity of 0.95. The burning curve is presented in Figure 2.

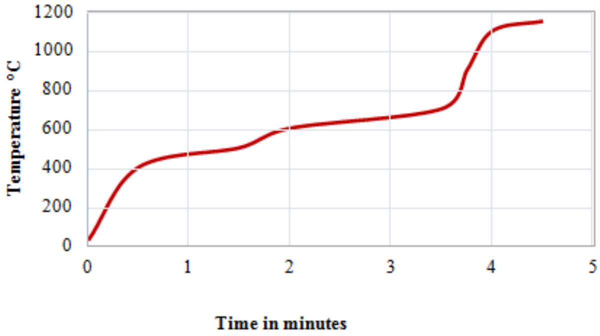

Figure 2. Calcination curve in rotary scale oven.

3. Results: Statistical analysis

The volumetric expansion ratio, determined for each one of the replicas of the treatments, varying the percentages of nutrient are shown in Table 3.

Table 3. Data of volumetric relation of expansion varying the percentage of technological nutrient.

Treatments					
1	2	3	4	5	6
1.0100	1.0638	1.2683	1.5767	1.2889	1.0630
1.0075	1.0701	1.2617	1.5606	1.2980	1.0682
1.0104	1.0594	1.2565	1.5756	1.2875	1.0764
1.0064	1.0698	1.2642	1.5589	1.2880	1.0634
1.0084	1.0678	1.2550	1.5542	1.2970	1.0831

3.1. Analysis of variance using software R

The variation of the percentage of technological nutrient, includes to the clay mixtures organic matter that maximizes the capacity of expansion, nevertheless, a quantity of organic material must be added that does not cause a backward movement in the volumetric expansion. Figure 3 shows that the mix of clay type 4, that is, when adding 20% of the nutrient technology, the highest expansion ratio occurs, with 1.56 times its initial size, on the contrary, when 30% of the nutrient is added, the same expansion as when 10% nutrient is added, which is 1.07 times the initial size of the particle, in volume.

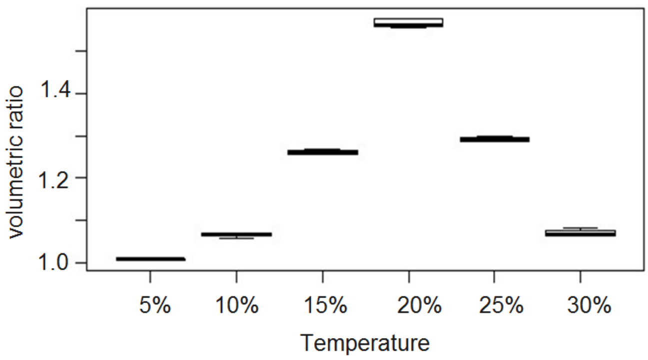

Figure 3. Box graph of volumetric expansion ratio for clay mixtures with different percentages of nutrient.

The model based on the regression coefficients it is statistically significant at the 95% confidence level ($\alpha = 0.05$). For Figure 3 the estimation of the developed model is * $P = 2.2e\text{-}16 < \alpha = 0.05$, which implies that the Null hypothesis is rejected, concluding that there are statistically significant differences in the volumetric expansion ratio according to the type of clay mixture with different percentages of nutrient.

The test LSD of Fisher, the test HSD of Tukey and the Dukan test present an average square error (mean square error) of 0.0000436, value that is approximated to zero, that is to say, that there are no errors in the experiments, statistically speaking, in addition, the maximum expansion is presented in treatment 4 (see Table 4). Fisher's test LSD shows that the smallest significant difference is 0.0086 and the Tukey Test HSD shows the least significant difference is 0.0129.

Table 4. 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	1.0085	0.00167	5	1.0024	1.0146	1.0064	1.0104
2	1.0662	0.00454	5	1.0601	1.0723	1.0594	1.0701
3	1.2611	0.00548	5	1.2550	1.2672	1.2550	1.2683
4	1.5652	0.01027	5	1.5591	1.5713	1.5542	1.5767
5	1.2919	0.00517	5	1.2858	1.2980	1.2875	1.2680
6	1.0708	0.00873	5	1.0647	1.0769	1.0630	1.0831

3.2. Assumptions of the analysis of variance

The Shapiro test results * p = 0.5992> α = 0.05 indicating that the residual errors of the volumetric expansion ratio of the clay mixtures have the behavior of a normal distribution, statistically speaking. Bartlett's test results * P 0.0593> α = 0.05 indicating that the residuals of the volumetric expansion ratio of clay mixtures have constant variance, statistically speaking (see Figure 4).

The Ljung-Box test gives as a result * P 0.3669> α = 0.05 indicating that the residuals of the volumetric expansion ratio of the clay mixtures have independence, statistically speaking (Figure 5).

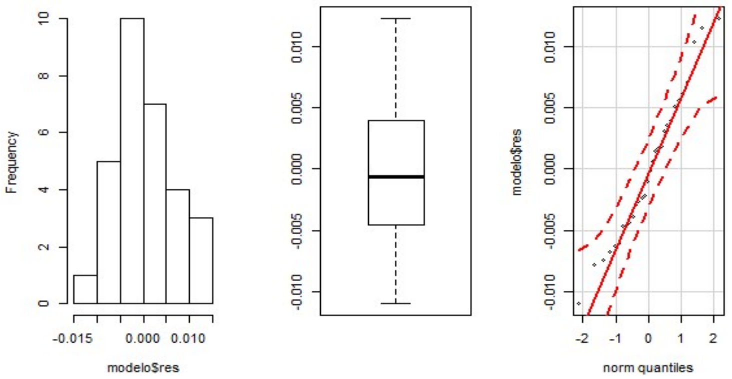

Figure 4. Residual model of the expansion volumetric relationship for the different types of clay mixtures.

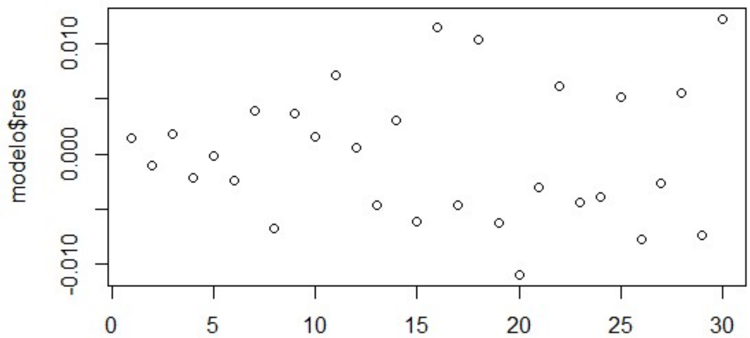

Figure 5. Residuals against the order of the volumetric expansion ratio for the different types of clay mixtures.

4. Conclusions

The addition of a 20% technological nutrient to clay mixtures produces the highest expansion ratio, with 1.56 times the initial size in volume.

The addition of 30% of the nutrient produces the same expansion as when 10% of the nutrient, which is 1.07 times the initial size of the particle, in volume, so when comparing the two treatments, there are no statistically significant differences.

The model based on the regression coefficients, estimates that the developed model is * $P = 0 < \alpha = 0.05$, which implies that the Null hypothesis is rejected, that is, there are statistically significant differences in the volumetric expansion ratio according to the type of clay mixture with different percentages of technological nutrient, when the six treatments are compared together.

Fisher's test LSD, Tukey's test HSD, and the Dukan test present an average quadratic error of 0.00000436, indicating that there are no errors, statistically speaking, in the experiments.

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 Bartlett´s test 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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