Journal of Physics: Conference Series

Determination of the expansion capacity by calcination of the clays from San José de Cúcuta, Colombia

To cite this article: J F Márquez *et al* 2021 *J. Phys.: Conf. Ser.* **2046** 012052

Determination of the expansion capacity by calcination of the clays from San José de Cúcuta, Colombia

J F Márquez[1], **J R Cáceres**[1], **and J D Palacios**[1]
[1] Universidad Francisco de Paula Santander, San José de Cúcuta, Colombia

E-mail: jorgefernandomp@ufps.edu.co

Abstract. Viscoplasticity and pyroplasticity of clays are important characteristics to control in production processes of lightweight concrete aggregates. When clay is calcined under temperatures above 1,000 °C, its volume increases. For this reason, it is important to investigate how much volume can gain a given clay before using it in production of lightweight aggregates. This research work has evaluated the expansive capacity of some Colombian clays, applying to them static heat treatment in furnaces at temperatures above 1,000°C. The theoretical background of this work is based on the state of the art of production processes of lightweight clay-based aggregates, which were used to determine if the available clay is a precursor of lightweight aggregate. Also, chemical characteristics were determined using X-ray fluorescence tests. Raw material was chosen from several clays existing in San José de Cúcuta, Colombia. The clays were processed until particles' diameters resulted smaller than 75 μm. Using these particles, pellets with diameter varying from 10 mm to 15 mm were formed. To determine the expansion capacity and the reduction of volumetric density, volume and weight of pellets were measured before and after applying the heat treatments. When the clay presented a low expansion capacity, technological nutrients were added to the mixtures attempting to maximize the expansion capacity of the pellets. A swelling of up to 3.24 times the initial size of pellets was observed. technological nutrients based on organic matter were chosen and applied in a proportion of 1: 4, *i.e.*, one gram of technological nutrients was added to 4 g of clay. The density of manufactured aggregates resulted to be between $0.50\ \mathrm{g/cm^3}$ and $1.93\ \mathrm{g/cm^3}$. Clay mixtures added with technological nutrient showed density lower than 1 gram per cubic centimeter, this means that they could float on water.

1. Introduction

Lightweight aggregate (LWA) made of expanded clay is a product widely used in constructions due to its properties of lightness, high resistance, thermal and acoustic insulation, and durability [1]. Determination of chemical properties such as viscoplasticity and pyroplasticity is important to know if clays can be precursors of LWA when exposed to temperatures above 1,000 °C. When clay is precursor of LWA, it increases its size when subjected to high temperature augmenting the internal porosity of the constituent particles [2]. To maximize the expansion capacity of the clays, technological nutrients (NUT) are used. NUTs can be by-products or industrial waste which are added in small amount to the clay mixture. Sewage sludge, slags coming from incineration of municipal solid waste, and fine clays settled in ports and canals are the most commonly used by-products in the manufacture of LWA [3].

Chemical compounds existing in clays are mainly silica (SiO_2), alumina (Al_2O_3) and other fluxing constituents ($Fe_2O_3 + MgO + CaO + Na_2O + K_2O$). The compounds proportion required by a clay to be a precursor to LWA has been studied and reported in the scientific literature. Riley (1951) developed a ternary diagram useful to determine if a given clay has expansive capacity when subjected to calcination; the diagram delimitates a zone of expansibility that allows to identify if a clay has such expansive capacity [4]. Cougny (1990) modified the ternary diagram presented by Riley developing a quaternary diagram that predicts which clays have the appropriate properties to be a precursor to LWA [5]. Finally, Dondi, *et al.* (2016) integrated by-products and industrial waste into the clay mixture and created several two-dimensional diagrams which facilitate the verification of characteristics that raw materials should have for the manufacture of LWA [6].

LWA is used in back wall fillings, agronomic applications, manufacture of thermo-acoustic insulators made of gypsum, asphalt and lightweight concrete, manufacture of precast structural, concrete, geotechnical applications, gardening, and hydroponics [7-10]. Selection of LWA for each application depends on needed physical and chemical properties (loose bulk density, water absorption or compressive strength) [11]. In particular, the density of particles decreases while their diameter of increases when they are calcined. Furthermore, aggregates with different densities can be produced by changing the production process [12]. Expanded clay LWA is produced by calcination clay granules at temperatures between $1{,}000\ ^{\circ}\mathrm{C}$ and $1{,}200\ ^{\circ}\mathrm{C}$. Gases generated within the mass expand clay mixture producing particles which have density values ranging from $400\ \mathrm{Kg/m^3}$ to $800\ \mathrm{Kg/m^3}$ [13-15].

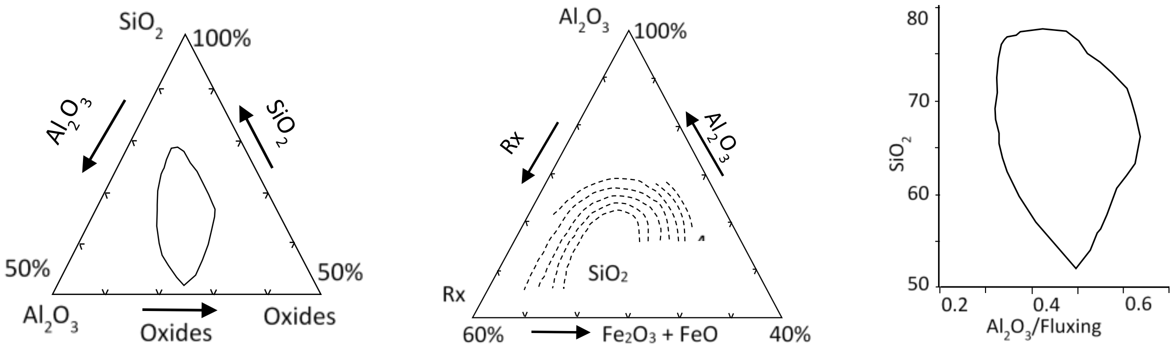
This work presents the analysis of the chemical properties of clays existing in San José de Cúcuta, Colombia. X-ray fluorescence tests were done and used to compare the chemical composition of clays with the references stated by the diagrams presented by Riley [4], Cougny [5] and Dondi, *et al.* [6]. Using the results of the comparison it was possible to predict which clays are precursors of LWA. In addition, an $1{,}100\ ^{\circ}\mathrm{C}$ furnace calcination process was applied to determine the expansion capacity of clays. As a complement, a NUT, based on sewage sludge, obtained as a by-product of the water purification process was used.

2. Methodology

A total of 11 types of clays coming from different quarries of San José de Cúcuta, Colombia, were selected. Subsequently, a preparation of the clays was carried out by means of a grinding process in a ball mill, with which the clay was pulverized until obtaining particles smaller than $75\ \mu\mathrm{m}$. To identify the chemical compounds of selected clays, an X-ray fluorescence test was carried out. To prepare samples for the test, the clay was dried at $105\ ^{\circ}\mathrm{C}$ for 12 hours and mixed with spectrometric wax in a $10{:}1$ ratio (homogenized by shaking). Using a hydraulic press, the prepared mix was subjected to $120\ \mathrm{KN}$ for one minute to conform a pressed tablet of 37 mm-diameter.

A semi-quantitative analysis was carried out taking 11 scans to detect the chemical compounds existing in the sample. Mentioned analysis did not include hydrogen (H), carbon (C), lithium (Li), beryllium (Be), boron (B), nitrogen (N), oxygen (O), and the transuranic elements. The compounds identified in this analysis were inserted within the diagrams of Riley [4], Cougny [5] and Dondi *et al.* [6].

Figure 1(a) shows the ternary diagram proposed by Riley [4]. When studied clays fall within the demarked central zone, it is said they have a good expansive capacity. Chemical compounds SiO_2–Al_2O_3–Oxides ($CaO+MgO+FeO+Fe_2O_3+K_2O+Na_2O$) are located to configure the vertices of the diagram; Figure 1(b) shows the quaternary diagram presented by Cougny [5], which is composed of three vertices with the following chemical compounds: Al_2O_3–(Fe_2O_3+FeO)–Rx($CaO+MgO+K_2O+Na_2O$). Composition curves plotted in the central zone of the diagram represent the compound SiO_2 which is the fourth factor of the diagram. Clays with a good expansion capacity will be located within those composition lines. Figure 1(c) shows the two-dimensional diagram proposed by Dondi, *et al.* [6] with modified Riley´s criteria. The axes of the diagram show the proportions of SiO_2 and Al_2O_3/Fluxes. Fluxes are made up of the following chemical compounds: $Fe_2O_3+MgO+CaO+Na_2O+K_2O+P_2O_5+TiO_2$.



(a) (b) (c)

Figure 1. Theoretical background based on the selection of clay for producing lightweight aggregates. (a) Riley's ternary diagram; (b) Cougny's quaternary diagram; (c) Dondi's two-dimensional diagram.

Clays identified as good for expansion capacity according to their chemical composition were subjected to a calcination process. Two mixtures were made for each one of the clays analyzed. One mixture was made using only clay (100% clay), and the other was made mixing 80% of clay with 20% of a NUT (sewage sludge). A total of 22 samples were subjected to a calcination process. Calcination process was applied as shown in Figure 2, in which two stages can be observed. The first stage occurs when the temperature of the particle reaches 600 °C. In this stage decomposition of water and crystallization of clay mineral occur. The second stage occurs when the particles reach a temperature of 1,100 °C. In this stage, Fe_2O_3 reacts chemically releasing oxygen and making the pellets to swell [3].

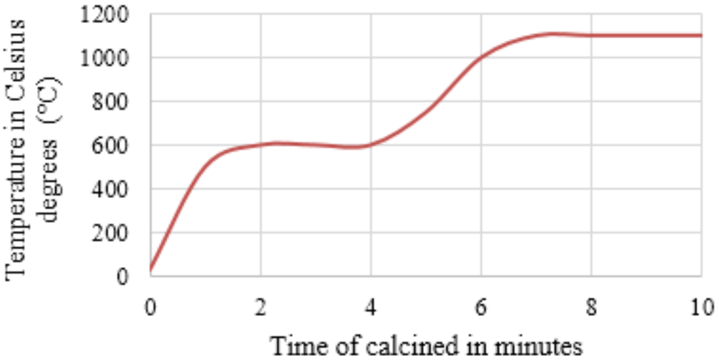


Figure 2. Calcination curve related to application of heat treatment.

Volumetric expansion and density of calcinated particles were computed using laboratory measurements. To determine the volumetric expansion capacity of the calcined pellets Equation (1) was used. The resultant number from application of Equation (1) expresses the number of times that the pellet increased its initial volume due to the calcination process. Equation (2) allows to compute the density of the calcined particles. In this research, those aggregates having a density lower than the density of water ($1{,}000\ \mathrm{Kg/m^3}$) will be considered as lightweight.

$$\text{Expansion} = \frac{\text{Final volume (after calcination)}}{\text{Initial volume (before calcination)}}, \qquad (1)$$

$$\text{Density} = \frac{\text{Weight after calcination}}{\text{Final volume (after calcination)}}. \qquad (2)$$

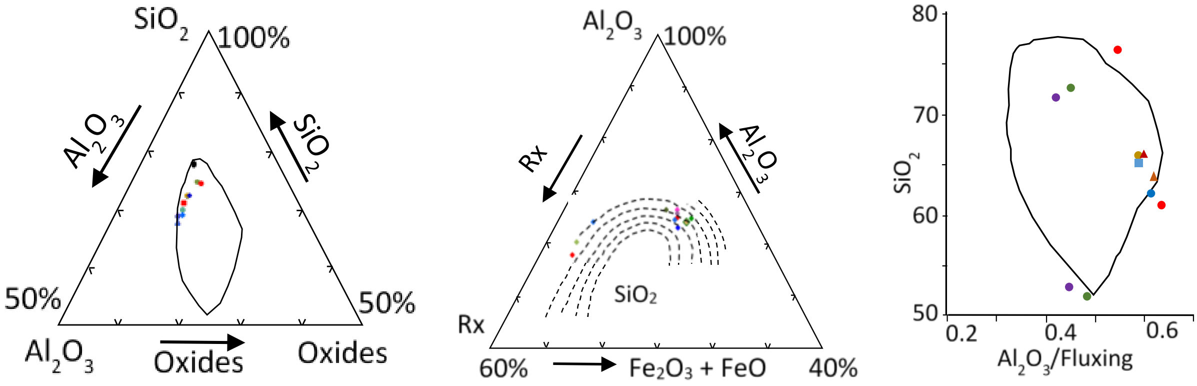
3. Results and discussion

From Table 1, it can be seen, silicon dioxide or oxide of silicon (IV) with chemical formula SiO_2, also called silica, is the most abundant compound within all samples. This compound, commonly found in the nature in form of quartz, is also the major constituent of sand. In the same way, dialuminium trioxide or oxide of aluminum (III) with chemical formula Al_2O_3, also known as alumina, is the second most abundant component in clay. Silica and alumina are the main components that provide strength and a crystalline surface when the clay is calcined at temperatures above 1,000 °C. Also, Table 1 shows that other components are present in not negligible amount within the clay mixtures. For example, the presence Fe_2O_3, K_2O and Na_2O indicates that the investigated clays contain iron, potassium and sodium which contribute to improve the necessary chemical reaction required to generate volumetric changes when clay is calcined at high temperatures.

Table 1. Chemical composition by X-ray fluorescence test.

Code of clay	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	Fe_2O_3	BaO	P_2O_5	ZrO_2
ACR-01	61.95	20.07	0.13	0.78	0.18	1.55	0.89	6.76	0.05	0.14	0.03
ACR-02	63.60	19.21	0.27	0.73	0.21	2.02	0.87	5.66	0.06	0.14	0.04
ACR-03	72.47	15.87	0.35	0.24	3.29	5.38	0.18	0.86	0.05	-	-
ACR-04	76.35	14.27	0.08	0.15	1.72	4.63	0.19	0.94	0.02	-	0.03
ACR-05	59.83	21.19	0.50	0.84	0.20	1.69	0.95	5.85	0.04	0.15	0.03
ACR-06	58.58	21.81	0.27	0.86	0.28	1.71	0.92	6.22	0.03	0.60	0.03
ACR-07	60.28	21.55	0.47	0.67	0.30	2.07	0.88	5.26	0.06	0.62	0.05
ACR-08	60.75	20.68	0.32	0.77	0.33	1.84	0.98	6.09	0.04	0.61	0.05
ACR-09	71.61	15.20	0.45	0.26	3.08	5.58	0.18	1.02	-	0.54	-
ACR-10	52.36	20.94	0.98	1.20	0.49	1.34	1.24	9.47	-	0.09	-
ACR-11	65.80	18.00	0.30	0.70	0.10	1.70	0.90	6.00	-	0.10	-

Using the criteria of Riley [4], Cougny [5], and Dondi, *et al.* [6], clays that have expansive capacity were predicted. According to the results plotted into Figure 3(a) and Figure 3(b), all the analyzed clays are within the expansion zone, therefore all of them have expansion capacity. However, Figure 3(c) shows that four clays fell outside the expansion zone, so these clays required the inclusion of a technological nutrient to help maximize their expansion capacity. Sewage sludge was used as technological nutrient in the mixture of those four clays. This choice was based on the literature review in which it was found that sewage sludge has the better effectiveness as a nutrient.



(a) (b) (c)

Figure 3. Clay swelling capacity prediction: (a) Riley's criteria; (b) Cougny's criteria; (c) Dondi's criteria.

Pellets of approximately 10mm-diameter were made. As showed in Figure 2, a heat treatment was applied, going from preheating at 600 °C to calcination at 1,100 °C. The heat treatment applied to each sample lasted 10 minutes. Using Equation (1) and Equation (2), expansion capacity and density of each sample (100% clay and 80% clay plus 20% sewage sludge) were computed. 25 replicas of each sample were statistically analyzed. Figure 4 shows the ratio between the expansion capacity and the density of the pellets. It is evident that while the expansion of the pellets increases, the density decreases. This trend confirms what was explained previously related to porous systems of the pellets. Also, as exposed above, the LWA are those which developed an expansion larger than 1.75, that is, they increase their volumetric size by 75%.

Pellets of approximately 10 mm-diameter were made. As showed in Figure 2, a heat treatment was applied, going from preheating at 600 °C to calcination at 1,100 °C. The heat treatment applied to each sample lasted 10 minutes. Using Equation (1) and Equation (2), expansion capacity and density of each sample (100% clay and 80% clay plus 20% sewage sludge) were computed. 25 replicas of each sample were statistically analyzed.

Figure 4 shows the ratio between the expansion capacity and the density of the pellets. It is evident that while the expansion of the pellets increases, the density decreases. This trend confirms what was explained previously related to porous systems of the pellets. Also, as exposed above, the LWA are those which developed an expansion larger than 1.75, that is, they increase their volumetric size by 75%.

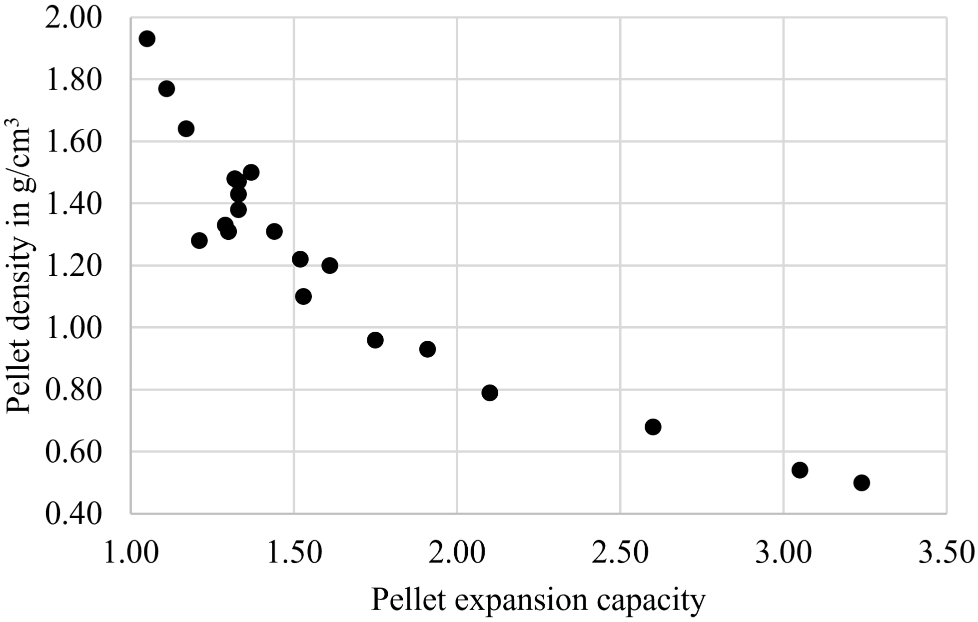


Figure 4. Ratio between expansion capacity and density of manufactured pellets.

Six LWAs with greater expandability were studied more deeply. Figure 5 shows microscope images of typical LWA clay produced during the development of this work. In all cases, it is evident that the typical granule has ideal microstructural characteristics and pore structure. Figure 5(a) shows a pellet made with 80% ACR-03 and 20% NUT; in this case, sample was expanded 2.05 times its initial size and developed a density of 0.54 g/cm^3. Figure 5(b) shows a pellet made with 80% ACR-04 and 20% NUT; this sample reached an expansion of 1.10 times the initial size and a density of 0.79 g/cm^3. Figure 5(c) shows a pellet made with 80% ACR-05 and 20% NUT; this sample reached an expansion of 2.24 times the initial size and a density of 0.50 g/cm^3. Figure 5(d) shows a pellet made with 80% ACR-06 and 20% NUT; this sample reached an expansion of 0.91 times the initial size and a density of 0.93 g/cm^3. Figure 5(e) shows a pellet made with 80% ACR-08 and 20% NUT; this sample reached an expansion of 1.60 times the initial size and a density of 0.68 g/cm^3. Figure 5(f) shows a pellet made with 80% ACR-05 and 20% NUT; this sample reached an expansion of 0.75 times the initial size and a density of 0.96 g/cm^3.

The mixtures in Figure 5(a) and Figure 5(c), which have the highest total porosity in the core, belong to some samples related to the lower density LWAs. Figure 5(b) and Figure 5(e), which show a moderate total porosity, belong to granules that had a medium expansion capacity. Figure 5(d) and Figure 5(f), which are related to granules that show low total porosity, are related to low expansion capacity.

When comparing the results of this work with previous research related to LWA made of expanded clay, it is evident that clays existing in San José de Cúcuta, Colombia, have a good expansion capacity. This fact represents promising possibilities of developing new products which help to optimize the construction of civil works in Colombia.

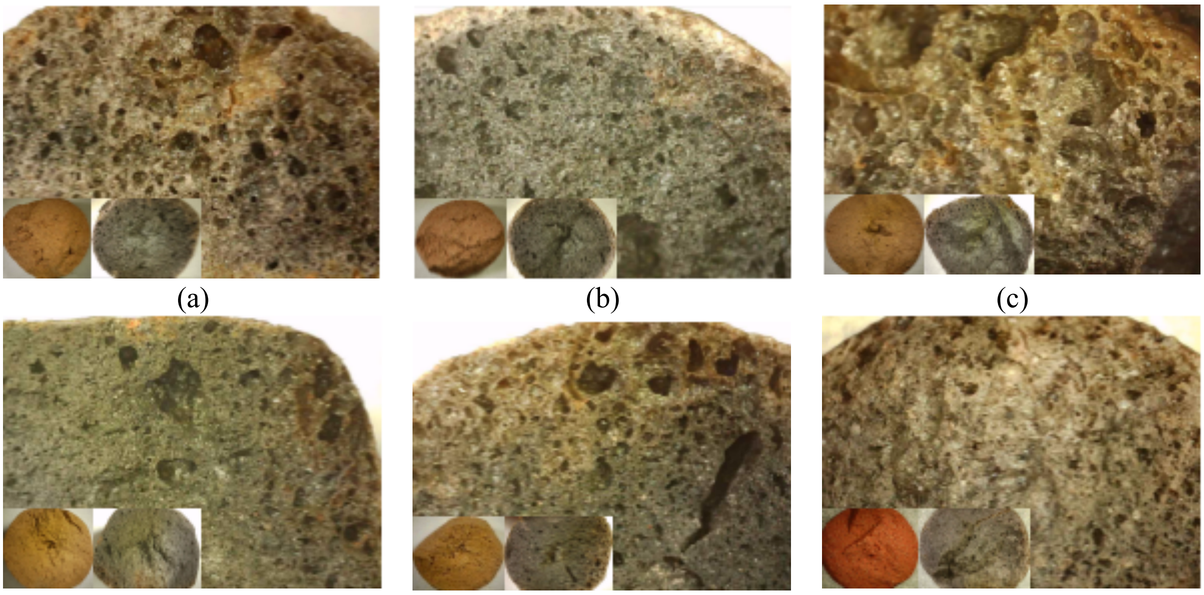

(a) (b) (c)
(d) (e) (f)

Figure 5. LWA manufactured of clay and NUT (sewage sludge) from San José de Cúcuta, Colombia. (a) 80% ACR-03+20%NUT; (b) 80% ACR-04+20% NUT; (c) 80% ACR-05+20% NUT; (d) 80% ACR-06+20% NUT; (e) 80% ACR-08+20% NUT; (f) 80% ACR-10+20% NUT.

4. Conclusions

Eleven types of clay existing in San José de Cúcuta, Colombia, were found to have a good expansion capacity according to the criteria of Riley (1951), Cougny (1990), and Dondi, *et al.* (2016). Silica (SiO_2), alumina (Al_2O_3) and Fe_2O_3 were the most common chemical compounds in studied clays with proportions of $52\% - 76\%$, $14\% - 22\%$, and $1\% - 7\%$ respectively.

A thermal treatment was applied during 10 minutes in two successive stages. The first stage took the pellets from room temperature to 600 °C. The second stage completed the temperature increment reaching rapidly 1,100 °C, generating thermal shock, which caused swelling in the calcined pellets.

The expansion capacity of studied clays resulted to be between 1.06 and 3.24. Upper value shows that some pellets triplicated their initial volume.

A relationship between the expansion capacity and the density of the clay pellets was observed. The greater the expansion the lower the pellet density. Six mixtures of clay prepared adding sewage sludge reached density values lower than the density of water. In such case, the pellets are considered as lightweight aggregate. Pellet density varied between $0.50\ \mathrm{g/cm^3}$ and $1.931\ \mathrm{g/cm^3}$.

When comparing the results of this work with previous research related to lightweight aggregate made of expanded clay, it is evident that clays existing in San José de Cúcuta city have a good expansion capacity. This fact represents promising possibilities of developing new products which help to optimize the construction of civil works in Colombia.

Acknowledgment

Special recognition to the Universidad Francisco de Paula Santander, San José de Cúcuta, Colombia, for the financial support given for the development of this research.

References

[1] Holm T A, Ries J P 2007 *Reference Manual for the Properties and Applications of Expanded Shale, Clay and Slate Lightweight Aggregate* (Chicago: Expanded Shale, Clay and State Institute)

[2] Cáceres J R, Rojas J P, Sanchez J 2019 Determination of expandability clay from San José de Cúcuta metropolitan area, by adding different percentages of sewage sludge *Journal of Physics: Conference Series* **1388** 012013

[3] Cáceres J R, Rojas J P,Sanchez J 2019 A review about the use of industrial by-products in the lightweight aggregates production of expanded clay *Journal of Physics: Conference Series* **1388** 012011

[4] Riley C M 1951 Relation of chemical properties to the bloating of clays *Journal of the American Ceramic Society* **34(4)** 121

[5] Cougny G 1990 Spécifications sur les matières premières argileuses pour la fabrication de granulats légers expansés *Bulletin of the International Association of Engineering* **41(1)** 47

[6] Dondi M, Cappelletti P, D'Amore M, de Gennaro R, Graziano S F, Langella A, Raimondo M, Zanelli C 2016 Lightweight aggregates from waste materials: Reappraisal of expansion behavior and prediction schemes for bloating *Construction and Building Materials* **127** 394

[7] Ozguven A 2014 Development of expandability charts for Ankara Kalecik Clay *Applied Clay Science* **101** 533

[8] Priyadharshini P, Mohan Ganesh G, Santhi A S 2012 A review on artificial aggregates *International Journal of Earth Sciences and Engineering* **5(3)** 540

[9] Ayati B, Ferrándiz-Mas V, Newport D, Cheeseman C 2018 Use of clay in the manufacture of lightweight aggregate *Construction and Building Materials* **162** 124

[10] González-Corrochano B, Alonso-Azcárate J, Rodas M, Barrenechea J F, Luque F J 2011 Microstructure and mineralogy of lightweight aggregates manufactured from mining and industrial wastes *Construction and Building Materials* **25(8)** 3591

[11] González-Corrochano B, Alonso-Azcárate J, Rodas M 2014 Effect of prefiring and firing dwell times on the properties of artificial lightweight aggregates *Construction and Building Materials* **53** 91

[12] Ardakani A, Yazdani M 2014 The relation between particle density and static elastic moduli of lightweight expanded clay aggregates *Applied Clay Science* **93-94** 28

[13] Korat L, Ducman V, Legat A, Mirtič B 2013 Characterisation of the pore-forming process in lightweight aggregate based on silica sludge by means of X-ray micro-tomography (micro-CT) and mercury intrusion porosimetry (MIP) *Ceramics Internatioanl* **39(6)** 6997

[14] Shafigh P, Ghafari H, Mahmud H B, Jumaat M Z 2014 A comparison study of the mechanical properties and drying shrinkage of oil palm shell and expanded clay lightweight aggregate concretes *Materials and Design* **60** 320

[15] Sanchez J, Cáceres J R, Rojas J P 2019 Determination of expandability clay from San José de Cucuta metropolitan area, by varying calcination temperature and time *Journal of Physics: Conference Series* **1388** 012012