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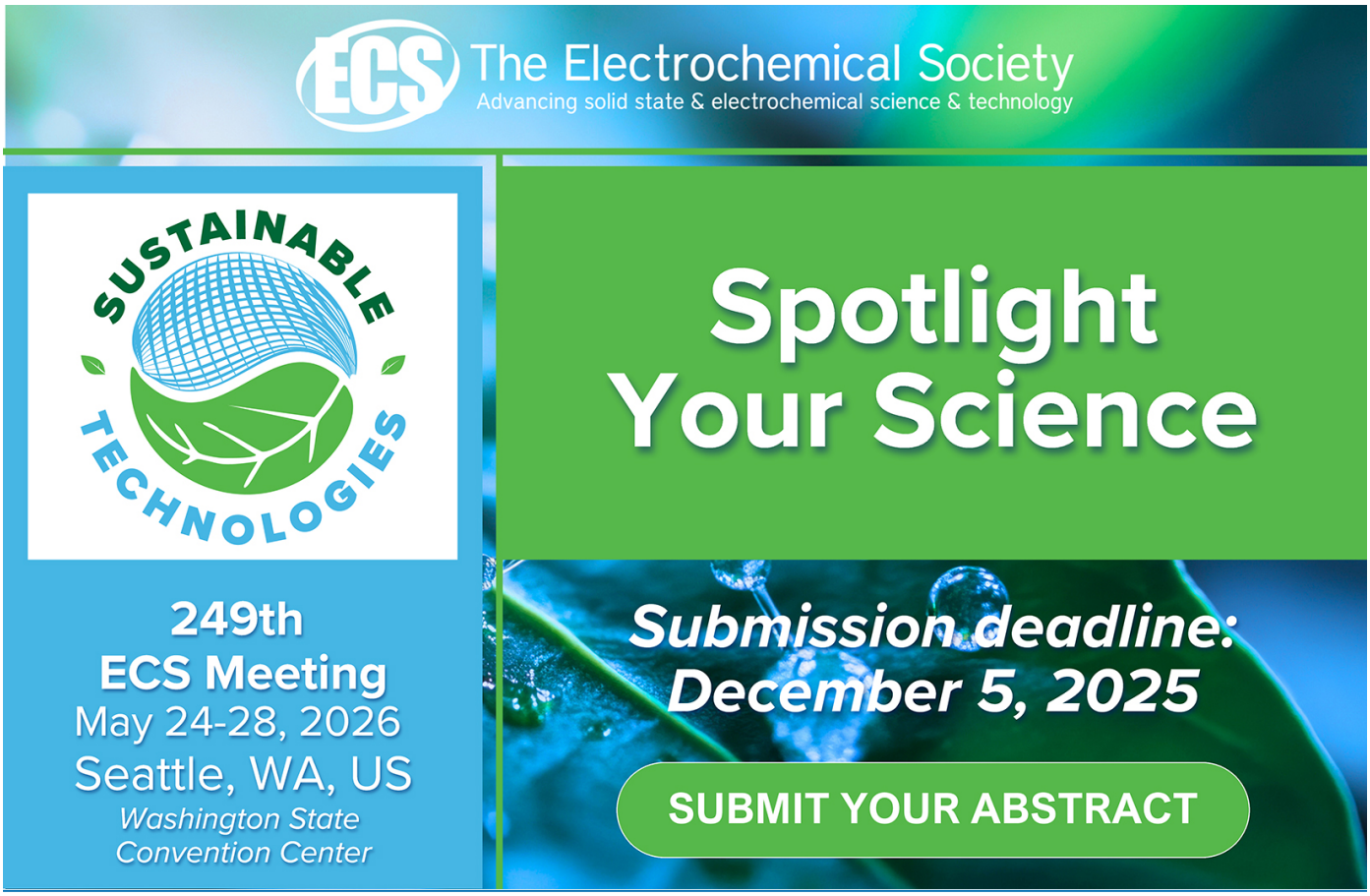
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Determination of physical properties and chemical composition of clay from intrusive and extrusive igneous formation

G Guerrero Gómez[1], N Afanador García[2], and C Nolasco[3]
[1] Grupo de Investigación en Tecnología y Desarrollo en Ingeniería, Universidad Francisco de Paula Santander, Seccional Ocaña, Colombia
[2] Civil Engineering Research, Universidad Francisco de Paula Santander, Seccional Ocaña, Colombia
3 Grupo de Investigación de la Facultad de Educación Artes y Humanidades, Universidad Francisco de Paula Santander, Seccional Ocaña, Colombia

E-mail: gguerrerog@ufpso.edu.co, nafanadorg@ufpso.edu.co

Abstract. In the mixtures used to produce ceramic materials in Ocaña, Colombia, clays from the intrusive and extrusive igneous formation are used, to which grain size, chemical composition, and thermal conductivity are determined. Scanning electron microscopy tests at 700x magnification were applied to the prepared clay sample to find grain size and chemical composition. The results of the chemical composition of the clay indicate that the major component present is silicon with 27.96%, followed by 13.81% of aluminum, 3.29% of potassium and 58.95% of oxygen. Also, the grain size is in the range of 82701 nm to 96980 nm, density of 1.0705 g/cm^3 and average thermal conductivity of 1.78 W/m×ºC. The morphological and physical-ceramic properties of the clays are within the admissible ranges to be used in technological applications in the wastewater treatment plants, in designs of clay filters for water purification; they can also be used as refractory material for kiln walls in the ceramics sector.

1. Introduction

Minerals such as clay, which is composed of aluminosilicates, quartz [1] and organic matter [2], among others, are found in nature and are used to produce ceramic materials [3] due to their physical, mechanical, and morphological properties [4]. The ceramic industry in Ocaña, Norte de Santander, Colombia, has developed in an empirical and traditional manner [5], generating a number of direct jobs that have allowed the region to develop [6]. Producers of ceramic materials use kilns with no controls on combustion processes [7], which generate large heat losses [8], and high levels of particulate matter in the environment [9].

The national planning department establishes in its productivity and competitiveness agenda for Norte de Santander that the ceramic sector should be the national leader in the field of high-quality ceramic materials production [10]. For this reason, the ceramic sector in the region should be technician and classification studies of raw materials should be carried out, in order to offer better quality products that will allow achieving competitiveness and sustainability in the current market, complying with government regulations. Therefore, studies should be carried out to establish whether clay from the extrusive and intrusive igneous formation can be used in technological applications such as clean water and wastewater treatment and to offer new products to improve their thermal insulation.

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This research allows knowing the chemical composition, grain size, and thermal conductivity of the clays of the intrusive and extrusive igneous formation, which can be used in the treatment of residual waters of the plant located in the village Filipote of Ocaña, decontaminating the Hatillo stream and protecting the health of more than 11000 inhabitants in the sector, as well as the design of clay filters for the potabilization of water in alliance with the local association of clay and related products trader. They can also be used for the manufacture of refractory material for replacing kiln walls to produce ceramic materials.

2. Methodology

The selected clay sample comes from the extrusive and intrusive igneous formation, which is used for the manufacture of ceramic products in the brick kilns located in Ocaña, Colombia. For the preparation of the clay mixture of plastic texture [11], the drying process is carried out first. This is followed by the crushing and grinding to reduce the grain size, in which, the sample is introduced in a compacting mold to be crushed by means of a compaction hammer, until obtaining materials with low grain size and in a granular state [12]. Finally, the pulverized clay is sieved to a particle size of less than 2 mm.

Scanning electron microscopy (SEM) was performed to determine the grain size and chemical components of the clay. This was carried out using Denton Vacuun Desk V and Sem Jeol JCM 6000 Plus equipment under high vacuum conditions using secondary electrons and 15 kilovolts (kv), in which energy dispersive spectroscopy (EDS) was performed. Finally, micrographs were taken at 700× magnification to observe the grain size of each of the granular materials.

To evaluate the thermal conductivity of the clay sample, a thermal conductivity test bench was used, which applies the infinite cylinder experimental technique in which heat conduction is considered at steady state temperature in the radial direction [13]. To perform the thermal conductivity test, 101 cm^3 of the granular material sample is required, which was placed inside the sample. To achieve a better uniformity in the distribution of the material in the specimen, a vibration regulator available in the equipment is used, which eliminates the presence of air in the sample, avoiding heat transfer by convection, which guarantees a better prediction of the thermal conductivity of the material.

Once the sample has been placed inside the test tube, K-type thermocouples are connected to measure the temperatures of the granular material sample that is in contact with the resistor and the tubular sample casing. These are located as follows: one is in the central shaft whose material is Inconel 600 wire, 6.7 millimeters in diameter, and 215 millimeters long; and three thermocouples are located on the surface of the sample, two at the ends of the sample and one in the center, see Figure 1.

Subsequently, the equipment is connected by passing an electrical voltage to the heating resistance of the shaft and then testing is performed, programming the temperature data acquisition time, and executing the program, thus achieving the analog-digital processing of the information received from the thermocouples. Then, they are stored through the Lab view software in the data acquisition report of temperatures of the selected positions, average temperature of the casing surface, average temperature of the shaft and casing, voltage, current, and thermal conductivity of the sample, as well as the schematic of the thermal conductivity of the material (see Figure 1). To determine the value of the thermal conductivity of the sample material at the temperature at which the sample is located, the Fourier's law of thermal conduction was used, the following Equation (1) given by [7], was used for the evaluation.

$$Q = \frac{T_i - T_e}{\frac{\mathrm{Ln}\left(\frac{r_e}{r_i}\right)}{2\pi k L}}, \tag{1}$$

where Q is the conduction heat transfer through the casing given in W; T_i is the haft temperature given in °C; T_e is the average casing surface temperature given in °C; r_e is the outer casing radius in m; r_i is the inner casing radius in m; k is the thermal conductivity of the sample material given in W/m×°C; L is the casing length in m. To determine the heat flow by conduction, the relationship between voltage, current and heat flow is used, the following Equation (2) given by [7], was used for the evaluation.

$$Q = VI, \quad (2)$$

where V is the voltage given in volts, and I is the current given in amperes.

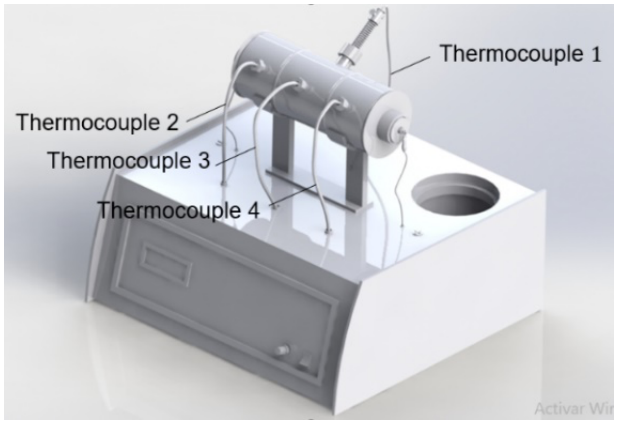


Figure 1. schematic of the thermal conductivity of the material.

3. Results and discussions

Table 1 shows the results of the composition in weight and atomic percentage of the clay analyzed by EDS. The results of the EDS analysis derived from the scanning electron microscopy test at 700x magnification indicate that the chemical composition of the sample is composed of a high percentage of silicon with 27.96% and 13.81% of aluminum and in lower percentages of potassium with 3.29% of potassium and the remainder is oxygen with 58.95% from the surroundings.

Table 1. chemical composition in weight and atomic percentages of the clay.

Element	Electron volts (keV)	Mass (%)	Atomic (%)
Si	1.739	23.78	16.83
Al	1.486	11.00	8.11
K	3.312	1.92	0.97
O	0.525	58.13	72.24

Figure 2 shows SEM at different magnifications, where a laminar morphology, in the form of superimposed plates of silicon aluminates [14]. Associated with the presence of kaolin, can be observed. Around the central particle, a homogeneous particle size, whiteness and brightness, characteristic of kaolinite, is evident. To determine the bulk density of the clay, the sample was deposited in a 40 cm^3 container, which was proceeded to weigh, and its average mass was 42.8186 g obtaining a bulk density of the clay sample of 1.0705 g/cm^3. Also, the grain size of the sample ranges from 64.13 μm to 196 μm approximately.

Due to the presence of kaolin or hydrated aluminosilicate in the sample, product of the decomposition of feldspathic rocks, whose main component is kaolinite, it can be used as raw material in the elaboration and development of new products such as ceramics, paper, paints, pharmaceuticals, cosmetics. Also, it can be used in other research fields such as granulometry, oil exploration, soil engineering, water and oil microfiltration systems, water, and soil bioremediation, and as a support for catalysts.

The particle size distribution of the sample with sizes between 64.13 μm and 196 μm, are in line with those reported for porous ceramics with sizes between 75 μm to 425 μm [15]. In addition, the grain size and uniform pore distribution contribute to small values in thermal conductivity [16]. These values of grain size and composition of the clay are within the admissible ranges to be used in wastewater treatment, turbidity removal, as well as in water microfiltration systems using clay filters in partnership with the local association of clay and related products trader.

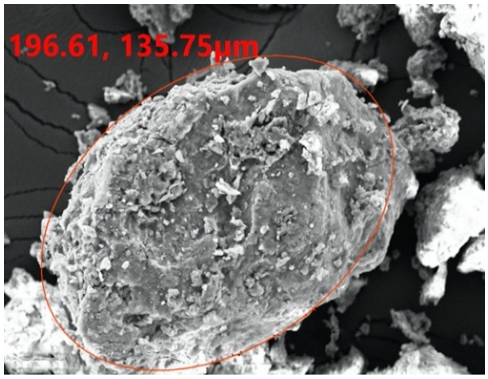


Figure 2. SEM micrograph 700x: clay before sintering.

The thermal conductivity of the sample material was determined at medium temperature between the casing surface and the shaft [17], using the thermal conductivity test bench for granular materials, by initially supplying a voltage to the electrical heating resistor, which generates heat radially. The data acquisition software of the thermal conductivity equipment was programmed to record temperatures, current flowing through the circuit and working voltage applied to the resistor at the time interval of every minute. The monitoring lasted for sixty minutes and 240 temperature data, 60 current data, and 60 operating voltage data were recorded.

Figure 3 reports the results of the axial measurements of temperatures located at the different positions on the shaft, average temperature of the casing surface, average temperature of the shaft and casing and the thermal conductivity of the sample as a function of the average temperature between the shaft and the casing surface. It can be observed that it took about ten minutes from the beginning of the test to reach thermal stability, registering a higher heating in the center of the shaft, where a temperature of 160 °C is reached, while in the superficial positions of the casing temperatures of 110 ºC is reached, this is because in these positions there is no resistive wire.

At the beginning of the test, it is observed that the highest temperatures occur in the shaft because the working power is applied directly to it and the temperatures in the casing begin to increase due to the contact between the shaft, the sample, and the casing, which heat up due to the effect of heat transfer by conduction based on Fourier's law. After the temperature stabilization in the thermal conductivity equipment, the thermal conductivity results of the sample are in the range of 1.13 W/m×K to 1.94 W/m×K. The average temperature of the heat source ranges from 111.6 °C to 130 °C, the thermal resistance ranges from 2.57 ºC/W to 4.48 ºC/W, and the heat flux ranges from 136.5 W to 151.9 W.

To determine the thermal conductivity value, the temperature gradient was taken as the average value of the values of the casing surface and the shaft from the stabilization time until the end of the test. At the beginning of the test, it is observed that the highest temperatures occur in the shaft because the working power is applied directly to it and the temperatures in the casing begin to increase due to the contact between the shaft, the sample, and the casing, which heat up due to the effect of heat transfer by conduction based on Fourier's law. After the temperature stabilization in the thermal conductivity equipment, the thermal conductivity results of the sample are in the range of 1.13 W/m×K to 1.94 W/m×K. The average temperature of the heat source ranges from 111.6 °C to 130 °C, the thermal resistance ranges from 2.57 ºC/W to 4.48 ºC/W, and the heat flux ranges from 136.5 W to 151.9 W.

On the other hand, comparing the effective thermal conductivity value of the clays of the extrusive igneous intrusive complex, measured experimentally at an average temperature of 150 °C of 1.78 W/m×K with those reported for a silty clay [18], which are in the range of 1.2 W/m×K to 1.4 W/m×K, indicates that the thermal conductivity of the soil decreases as the temperature of the clay decreases. In addition, a comparative experimental analysis of physical and mechanical properties and

heat flow in houses with traditional masonry and masonry manufactured with new materials of the igneous rock system can be carried out.

While comparing the bulk density of the clay sample of 1.0705 g/cm^3 and a thermal conductivity of 1.78 W/m×K with a silt clay sample] with density of 1.75 g/cm^3 and a thermal conductivity of 1.4 W/m×K, it indicates that the thermal conductivity of the soil increases with increasing bulk density [15]; likewise, the bulk density increases the heat transfer rate.

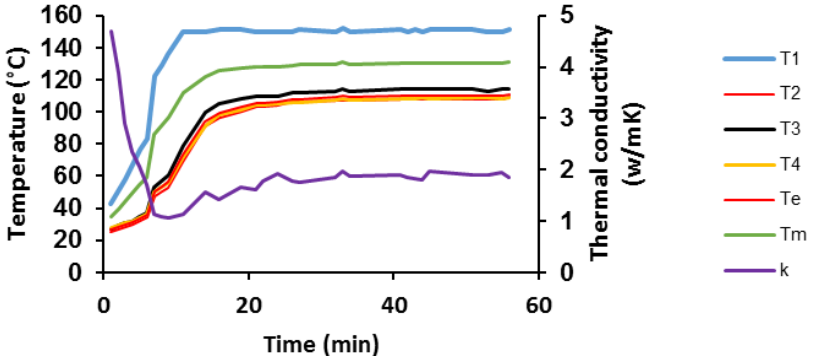


Figure 3. Heating profile measured and calculated by conduction during the measurements made to determine the thermal conductivity of the clay and the thermal conductivity of the sample.

4. Conclusions

The clays of the kaolin-based extrusive and intrusive igneous formation report morphological and physical-ceramic properties within the admissible ranges to be used in technological applications, such as in wastewater treatment, eliminating turbidity, organic matter and suspended solids, as well as in the use of vertical crops by adding nutrients such as sodium, nitrogen, potassium to the clay, providing greater support to the soil, allowing the anchoring of the roots inside the micro and macropores, and in the use of microfiltration systems.

The low thermal conductivity value of the sample and its high porosity make the heat transfer through them inefficient and slow and can be used in thermal and acoustic insulation systems, applicable as refractory material in muffle and metallurgical furnaces. The value found for the thermal conductivity of the sample will be essential to determine the overall heat transfer coefficient, which represents the amount of energy that passes through a surface per unit of time when there is a temperature gradient, which is used for the calculation of insulation and energy losses in the design of heating in buildings that use such materials.

The chemical composition of the sample is important because it can be used to design the optimum clay mixture to improve the properties of traditional products and the manufacture of new ceramic products, in order to perform a comparative experimental analysis of physical and mechanical properties and heat flow through a wall with traditional masonry and a wall made with new materials of the igneous rock system.

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