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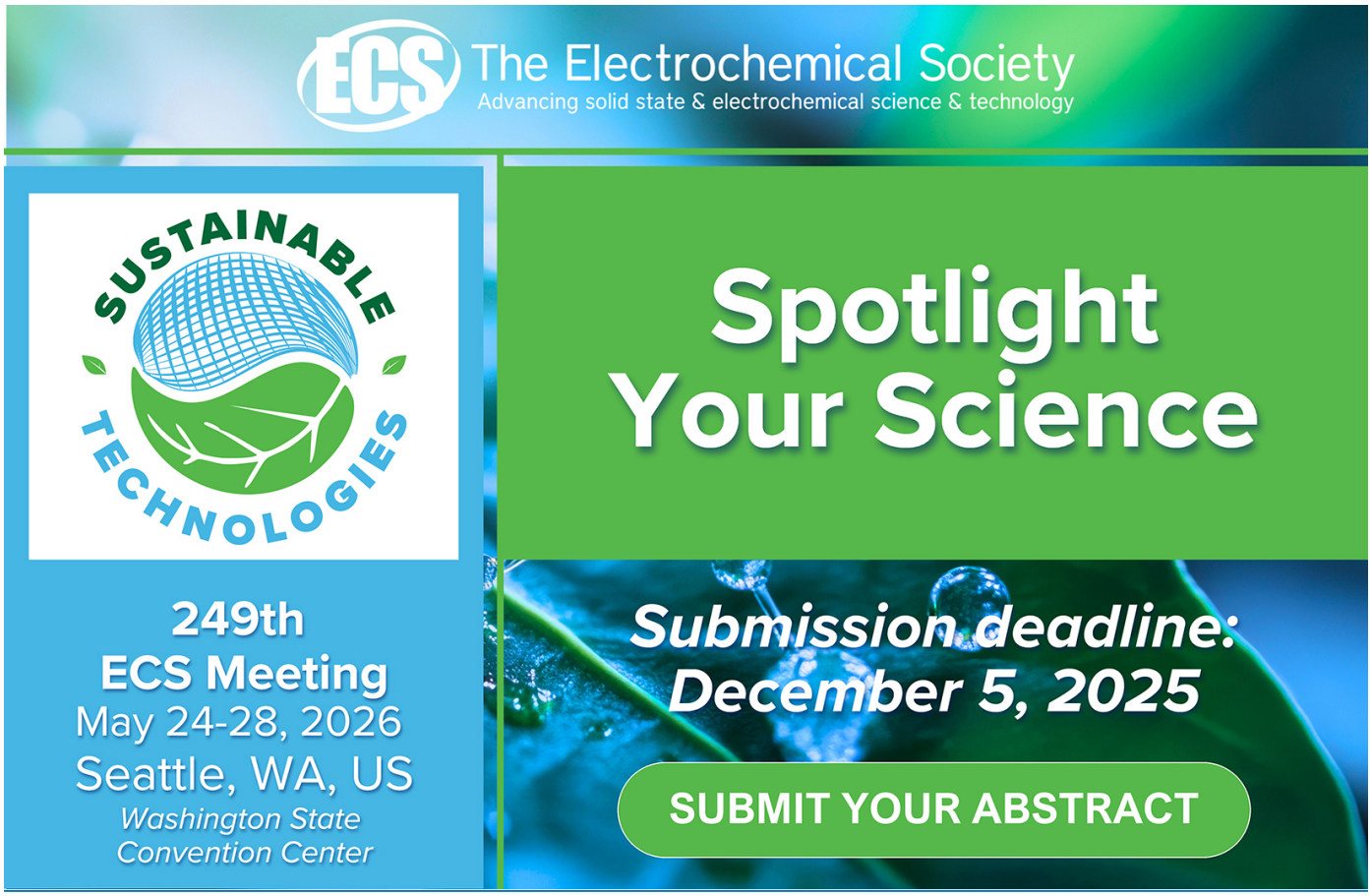
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To cite this article: G Guerrero-Gómez *et al* 2020 *J. Phys.: Conf. Ser.* **1587** 012035

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Evaluation of the influence of calcium carbonate on the firing process and sintering time

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Abstract. This paper presents the use of calcium carbonate as an additive in the manufacturing process of ceramic bricks, in order to reduce the temperature and sintering time of the clays and to improve the quality of the products. In the study, samples of solid bricks with two different proportions were made. One was composed of 100% clay and the other of 70% clay and 30% calcium carbonate, which were fired in an intermittent kiln. The temperatures of the firing process were recorded using a virtual instrument. The samples manufactured were tested for water absorption and strength. The addition of calcium carbonate brought advantages such as an 8.66% decrease in fuel consumption, a 23.88% decrease in firing time, a 12.11% and 26.67% decrease in the firing temperature and gases released into the environment, respectively, as well as improvements in the quality of the product. The use of calcium carbonate as an additive decreased the time and temperature of ceramic firing and fuel consumption and increased energy efficiency, productivity and product quality, as well as reduced gas emissions into the environment.

1. Introduction

The energy crisis is a problem generated by the indiscriminate use of fossil fuels over many years that has caused shortages and made their extraction from nature more difficult, requiring more advanced technology and increasing their prices. The construction industry in "Ocaña, Colombia", especially the ceramic sector, is one of the sectors with the highest energy consumption. The products obtained are the result of firing in traditional kilns. This is carried out by small and medium-sized producers, which due to the low level of technology do not have temperature, air, and fuel controls that result in incomplete combustion in the firing process [1]. As a result, it leads to increased consumption of coal and wood [2], low quality of its products [3], high production costs, environmental and health problems due to polluting emissions, as well as deforestation [4]. Therefore, it is increasingly necessary to save fuel in order to achieve a better use of energy.

The ceramic sector in "Ocaña, Colombia", generates a significant number of direct jobs and produces 1.027.600 products per month [5], making it one of the main development alternatives for the region. For this reason, it is of great importance to study and improve the production processes in order to achieve competitiveness and remain in the current market. Bricks are widely accepted products used in civil works due to their low cost and thermal insulation [6]. During sintering, they acquire mechanical resistance [7], but if their quality is not adequate, seismic vulnerability increases [8]. On the other hand,

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their use is limited by the high energy consumption required for their production [9]. During the firing process, kaolinite dehydrolysis and carbonate decarbonation take place, and amorphous material is created, leading to the formation of new phases: Anorthite, Ghelenite [10].

In the sector, studies have been carried out on the drying of bricks [11], mechanical properties of bricks [12], and studies on gas emissions [13]. This sector should be the national and bi-national leader in the field of high-quality ceramic products by 2020 [14]. To this end, the processes must be modified. The addition of fluxes such as calcium carbonate in the required proportions changes the mineralogical properties of the clay. This reduces the temperature at which sintering occurs during the thermal treatment of the clays, the firing time, as well as fuel consumption without affecting their physical properties, which are important factors in energy savings.

The aim of this research is to demonstrate the influence of calcium carbonate on thermal processes in the production of ceramic materials, such as sintering time and temperature in ceramic materials, as well as the mechanical properties of the products.

2. Methodology

The study presents the use of calcium carbonate as a flux in the production of ceramic bricks in "Ocaña, Colombia". Samples of solid bricks with two different proportions were made, one composed of 100% clay and the other with 70% clay and 30% calcium carbonate. The clay used is from the intrusive and extrusive igneous rock formation of Algodonal site in "Ocaña, Colombia".

In the firing process of the samples, an intermittent kiln was used from "Ladrillera el Estanco", located in the municipality of "Ocaña, Colombia", built with solid bricks produced in the same factory. The processes are carried out in a traditional and empirical way, from the kneading of the clay to the loading of the products, as well as the coal used as fuel for the firing. The drying of the bricks is done by natural process and the drying time of the products depends on weather conditions. The samples fired without calcium carbonate used 1500 kg of coal, produced 4300 bricks, and lasted two days and nine hours. On the other hand, 1370 kg of coal was used in the firing of the samples with calcium carbonate, producing 4300 bricks, and the firing process lasted two days and three hours. A temperature data acquisition system was designed, and temperature acquisition was performed for each firing process.

2.1. Temperature acquisition system design

For the acquisition of temperatures in the firing process with and without additives in the intermittent kiln, a virtual instrument was designed, using a NI 9213 I/O data acquisition card, a NI cDAQ-9184 Chassis, and the lab view software, which allowed the analog-digital processing of the information received through the installed thermocouples. In order to acquire temperatures, two positions in the kiln were recorded. One was placed in a brick located in the central part of the kiln, and the other allowed the recording of the temperature of the gas emitted to the environment, for which thermocouples made of chrome-aluminum alloy type K with ceramic insulation were used. They were installed simultaneously with the brick loading process in the kiln.

2.2. Sample selection

For the two firing processes, 20 samples were selected, and 5 samples were used in each test according to the methodology, selection processes, and sample preparation stipulated in the "Norma Técnica Colombiana, NTC 4017 [15]".

2.3. Tests

For the evaluation of the mechanical properties of the bricks, non-destructive tests were carried out such as initial rate of absorption and 24-hour water immersion, which measure the amount of water absorbed by the sample in 1 minute and for 24 hours respectively. All 40 samples were dried for 24 hours at a temperature of 110 °C in a muffle furnace. Subsequently, they were taken to a cooling chamber at a temperature of 24 °C ± 8 °C and humidity between 30% and 70% for 4 hours. At this point, for the non-destructive tests, ten samples were selected from each artisanal and industrial firing process to which

the dry mass was determined. Of these samples, five from each firing process were selected for the initial rate of absorption test. Then, they were partially immersed in clean water for 1 minute as established in the standard, as a show the Figure 1, to which the final mass was determined. The other ten samples, to which the dry mass was determined, were used for the 24-hour water immersion test. They were completely immersed in a tank with clean water at a temperature between 15 °C and 30 °C. Finally, the final mass of the samples was determined after the test.

Also, destructive quality control tests such as compressive strength and modulus of rupture were performed, which allowed the evaluation of the maximum compressive stress [16] and flexural modulus. The twenty dry samples used for these tests were given a 3 mm thick layer of plaster on the opposite sides. For the compression test, five samples of each firing process were selected and taken to a universal machine with a capacity of 1000 kN, to which load was gradually applied by means of a neoprene device, which ensured uniformity in the application of the load. The sample was taken to failure [17], allowing the recording of the ultimate tensile strength. The other ten samples, to which the dry mass was determined, were used for the bending test, which were taken to a universal machine and placed between two supports, subjecting the samples to a point load in the center of the upper face using a steel plate. The samples were taken to failure, recording the flexural strength.

3. Results

The advantages of using calcium carbonate as a fluxing additive in the brick manufacturing process are detailed below.

3.1. Virtual instrument validation

After placing the temperature acquisition equipment, the virtual instrument was programmed to record every 5 minutes the temperatures in the thermocouples installed in the kiln in the two firing processes, with and without additives. Monitoring in the firing process without the additive took two days and nine hours. Also, 804 data per position were recorded in the firing process. On the other hand, monitoring in the firing process with the additive took two days and three hours, and 612 data per position were recorded in the firing process. Figure 1 shows the temperature profile for the brick in the central part of the kiln in the firing process with and without additive.

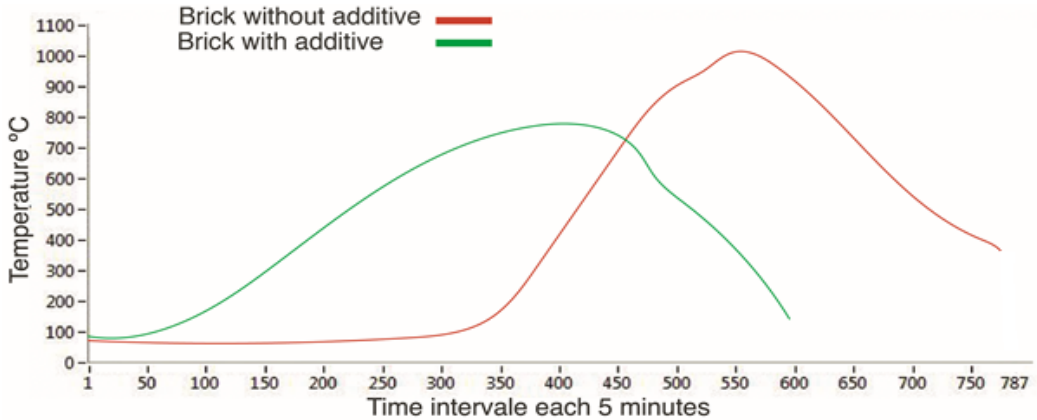


Figure 1. Temperature profile on the brick located in the central part of the kiln.

The preheating process of the products took 10 to 12 hours, until it reached an approximate temperature of 350 ºC to 400 ºC. Then, the temperature was raised until it reached the firing temperature. The firing curve for the sample with additive shows a faster drying than the curve that is composed of 100% clay. Therefore, the more plastic the clays are, the more difficult it is to dry them since the water is adsorbed in the inter-layered spaces, and the more clays there are, the more difficult it is to extract the water. Figure 2 shows the temperature profile for the gases emitted into the environment in the firing process with and without additives.

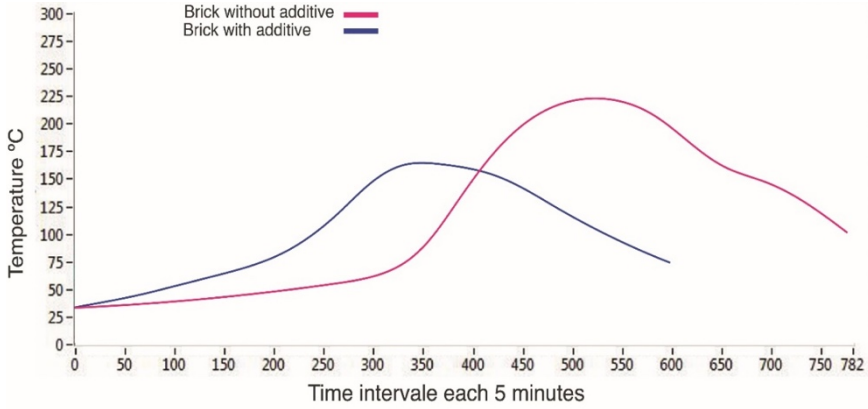


Figure 2. Temperature profile of gases emitted to the environment.

The temperature of the gases emitted into the environment in the firing process without the flux, during the first 18 hours of firing, was kept constant with a value of 50 °C, reaching the maximum temperature of 225 °C to 35 hours after the start of the firing due to incomplete combustion of the fuel. On the other hand, in the firing process with the additive, the maximum temperature of the gases released into the environment was reduced to 165 °C. The Table 1 show the comparison of aspects of the firing process with and without additives.

Table 1. Comparison of aspects of the firing process with and without additives.

Aspects of the firing process	Brick without additive	Brick with additive	Reduction %
Firing time (h)	67	51	23.88
Firing temperature (°C)	950	835	12.11
Maximum temperature the gases released into the environment (°C)	225	165	26.67
Fuel consumption per brick kg per brick (kJ/kg)	11441	10450	8.66

The firing time without the use of calcium carbonate was 67 hours. On the other hand, the firing time was reduced by 16 hours when calcium carbonate was used. The maximum temperature of the gases expelled to the environment in the firing process without the use of calcium carbonate was 225 °C. However, when the firing was done with calcium carbonate, it dropped to 165 °C. In the firing process without the use of additives, 1500 kg of coal were consumed, producing 49.200.000 kJ and a fuel consumption per brick of 11441 kJ/kg brick. On the other hand, in the firing process using additives, 1370 kg of coal were consumed, producing 44936000 kJ of energy, and a fuel consumption per brick of 10450 kJ/kg brick. For the analysis of the research results, an experimental study was carried out at the laboratory level on the samples with and without additive. Table 2 shows the results of the initial rate of absorption, 24-hour water immersion, compression strength, and flexural strength tests carried out on the samples in accordance with the standard.

Table 2. Results of water absorption and strength tests on the samples.

Parameter	Brick without additive	Brick with additive	Standard
Initial rate of absorption (g/cm^2/min)	0.40	0.21	0.25
Water absorption 24 hour (%)	21.00	16.94	17.50
Compressive strength (MPa)	4.26	6.87	14.00
Flexural strength (MPa)	1.98	2.53	0.29 a 0.88

In the initial absorption rate test, the samples without additive have a high absorption of 0.40 g/cm^2/min compared to 0.21 g/cm^2/min of those with additive. In other words, a 47.50% decrease in

water absorption per minute is achieved when using the additive. The samples without additive in the 24-hour water absorption test have 21% compared to 16.94% of those with additive, which means that a 19.33% decrease in water absorption per day is achieved when using the additive. The samples made with the additive acquired a compressive strength of 6.87 MPa compared to 4.26 MPa of those without the additive. In other words, a 40% increase in strength is achieved when using the additive. Samples made with an additive have a modulus of rupture of 2.53 MPa compared to 1.98 MPa of those without additive, which means that an increase of 21.74% in the modulus of rupture is achieved when using the additive.

4. Conclusions

The addition of calcium carbonate as a fluxing additive in the production of ceramic material improved the quality parameters of the produced bricks such as water absorption and strength, as well as reduced fuel consumption in the kilns during the brick firing process. On the other hand, the temperature, the firing time, the brick production cost, and the polluting emissions into the environment are also reduced. This provides a work alternative for small ceramic producers who do not have the economic resources to switch to a Hoffman kiln, artificial dryers, and technology in their processes.

The firing time using the additive was reduced by approximately 23.88% compared to the firing time without the use of the flux, which resulted in an increase in the production of the company. The temperature of the gases released into the environment using calcium carbonate was reduced by 26.67% compared to the firing process without the use of the additive. Exhaust gas temperatures in the kiln are around 200 °C, which presents great opportunities for waste heat recovery for parts drying, as well as drying and preheating of combustion air. These temperatures are below 250 ºC as established for discontinuous kilns according to Article 30 of Resolution 909 and Resolution 802 of 2014 issued by "Ministerio de Ambiente, Vivienda y Desarrollo Territorial Colombiano".

Fuel consumption in the firing process with the use of the additive was reduced by 130 kg of coal, as well as a reduction in fuel consumption of 991 kJ/kg, which corresponds to 8.66% of the consumption in the firing process without the use of the additive. The reduction of the firing time in the samples with the addition of calcium carbonate is attributed to an increase in capillary conductivity, and therefore, higher drying rates. In the initial rate of absorption and 24-hour absorption test, the samples with the additive meet the requirements of the NTC 4205 standard, which establishes a value of 0.25 g/cm^2/min and 17.5% respectively. In the compressive strength test, neither sample meets the requirements of NTC 4205 which sets a value of 14 MPa. In the modulus of rupture test, both samples comply with NTC 4205 standard, which establishes a range of values between 0.29 Map and 0.88 MPa.

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