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Calcined Clay Lightweight Ceramics Made with Wood Sawdust and Sodium Silicate

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This paper aims to study the influence of including wood sawdust and sodium silicate in the production process of calcined clay lightweight ceramics. In the production process first, a sample used by a company that produces ceramic products in Brazil was collected. The sample was analysed by techniques of liquidity (LL) and plasticity (LP) limits, particle size analysis, specific mass, X-ray diffraction (XRD) and X ray fluorescence spectrometry (XRF). From the clay, specimens of pure clay and mixtures with wood sawdust (10%, 20% and 30% by mass) and sodium silicate were produced and fired at a temperature of 900 °C. These specimens were submitted to tests of water absorption, porosity, specific mass and compressive strength. Results of this research indicate that the incorporation of wood sawdust and sodium silicate in the ceramic paste specimens can be useful to make calcined clay lightweight ceramics with special characteristics (low values of water absorption and specific mass and high values of compressive strength), which could be used to produce calcined clay lightweight aggregates to be used in structural concrete.

Keywords: *lightweight ceramics, lightweight concrete, sawdust, geo-polymers*

1. Introduction

Structural lightweight concrete is an important building material due to its load-reducing properties, and has been applied in many different areas. In Brazil, the main applications of structural lightweight concrete use expanded clay, which requires an energy intensive process to reach its final characteristics[1,2].

Preliminary analysis indicate the viability of utilizing calcined clay lightweight aggregate as building material, despite the high level of water absorption. Furthermore, the calcined clay lightweight aggregates show promising compatible characteristics relative to structural concrete[3-6].

However, to produce calcined clay lightweight aggregates with specific characteristics, such as low water absorption or high compressive strength, the production process requires adaption. Commonly used additives to produce character-specific lightweight ceramics are wood sawdust (to reduce the specific mass) and alkaline activators (to reduce the water absorption and increase the compressive strength).

A mixture of wood sawdust and ceramic paste to obtain low specific mass calcined clay lightweight ceramics has shown promising. The main downsides of the produced mixtures are a higher porosity of the material and increased water absorbency[7-9].

To reduce the level of water absorption of products rich in silicon and aluminum oxide, alkali activators can be used. Alkali activators cause the products, when kept in a low-temperature alkaline environment, to obtain cementitious properties due to an exothermic reaction[10]. The alkali activators have a positive effect on the hardening time of the products as well.

Geo-polymers are inorganic binders, as studied by Davidovits in the 1970s, originally referring to the investigations on the reaction of metakaolin in alkaline media forming aluminosilicate polymers[7].

Geo-polymers are composed of a three-dimensional network in which silicon atoms alternate with aluminum atoms in tetrahedral coordination, sharing all the oxygen atoms. Its structure consists of a polymeric network Si-O-Al, with SiO_4 and AlO_4 tetrahedrons linked alternately by the oxygen atoms. The presence of cations in the structure of geo-polymers is essential for balancing the negative charge of the AlO_4 [10,11].

This characteristic, combined with the SiO_2 and Al_2O_3 richness of the clay, can form geo-polymer chains by reacting with the alkali activator. The formed geo-polymer chains improve the absorbance and resistance characteristics of the fired samples, making them less absorbent and more resistant.

The pattern of geopolymer reactions comprises a set of reactions of dissolution, coagulation, crystallization and condensation. The first step consists of breaking the covalent Si-O-Si and Al-O-Si, that occurs when the pH of the alkaline solution increases. Then happens the accumulation of products of the breaking of bonds and simultaneous interaction between them, forming a coagulated structure, leading to a third phase which corresponds to condensation and formation of a crystallized structure[11,12].

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Preliminary studies on the addition of alkaline activators in red ceramic pastes to reduce the water absorption of calcined clay lightweight products indicate that the use of sodium silicate as an alkaline activator can be viable. Samples made with these activators presented lower values of water absorption relative to samples made with just clay and water[13].

This study aims to analyze the performance of calcined clay lightweight ceramics made with wood sawdust and sodium silicate as an alkaline activator, to produce less water absorbent and more resistant products with a low specific mass. Furthermore, it is assessed whether this production process can be used to produce calcined clay lightweight aggregates for structural concrete.

2. Experimental Set-up and Materials

To analyze the performance of calcined clay lightweight ceramics, samples were produced using clay collected from a company that produces ceramic roof tiles located at Leme/SP - Brazil. The collected clay is the same clay used in the production of the roof tiles. Before the sample production, the collected clay was analyzed by utilizing the following tests: liquidity (LL) and plasticity (LP) limits, particle size analysis, specific mass, X-ray diffraction (XRD) and X-ray fluorescence spectrometry (XRF)[14]. The tests allowed for the characterization of the clay and its potential alkaline features.

Several samples were produced with different proportions of wood sawdust and sodium silicate (alkali activator). The first samples were produced with only water and clay to allow for the performance comparison. The other samples included a proportion of sawdust and/or alkali activator (see Table 1). The wood sawdust was included as 10%, 20% or 30% by weight of the sample. If the alkali activator was included in the sample the sodium silicate was added with a 1:1 proportion relative to the water. Table 1 illustrates the amounts of materials used in each of the samples.

To produce the calcined clay aggregates the samples were extruded in a laboratory-sized extruder using a cylindrical mouthpiece, with 15 mm diameter. The specimens were cut in their final dimensions (100 mm length x 15 mm diameter) after extrusion and were kept at room temperature for 48 hours. Next, the produced aggregates were exposed to temperature of 60 °C in a ventilated oven for 72 h. Finally, they are fired in a Jung brand furnace (model 10013, 7 kW) at 900 ºC for 60 min of dwell time. The firing of the furnace was performed with 4 ºC/min of heating rate and cooling at 6 ºC/min.

Several tests allowed for the performance analysis of the produced aggregates. The calcined clay aggregates were subjected to water absorption tests (after a 24 hours immersion) and apparent porosity tests. Furthermore, the specific mass[15] and the compressive strength[16] of the aggregates were evaluated and compared.

3. Results

The presentation of the results is divided into two parts: the characterization of the raw material and the performance analysis of the calclined clay aggregates.

3.1. Characterization of the raw materials

The analyzed clay sample from the soil of Leme/SP – Brazil has values of liquid limit and plasticity limit of 58.0% and 25.9%, respectively. This corresponds to a plasticity index of 32.1%, characterizing the raw material as a material with plastic characteristics (plasticity index greater than 15%)[17].

The plasticity limit further represents the minimum amount of water required for the clay to obtain moldable characteristics. High values of plasticity limit require more water addition to transform the sample into a moldable form. The clay sample demonstrates a plasticity index (32.1%), between the appropriate limits for molding (10% to 35%)[17].

Table 2 displays the results of the particle size analysis of the clay. The sample is predominantly composed of clay (48.5%) and silt (37.5%), which confirms its highly plastic characteristics.

Figure 1 illustrates the X-ray diffraction pattern of the clay. The presence of clay minerals is clearly visible in the figure. The peak in the range of 2θ = 27° indicates the presence of free quartz the clay sample, indicating possibility to improve the compressive strength of the final product.

Table 1: Quantity of products used for molding the calcined clay samples.

Sample	Clay (g)	Wood sawdust (%)	Water* (g)	Activator (g)
100C-0WS	5210	0	1221	-
90C-10WS	5210	10	1221	-
80C-20WS	5210	20	921	-
70C-30WS	5210	30	657	-
100C-0WS-Si	5210	0	675	675
90C-10WS-Si	5210	10	675	675
80C-20WS-Si	5210	20	361.5	361.5
70C-30WS-Si	5210	30	435	435

* Water needed for both the clay and the wood sawdust absorption

Table 2: Particle size analysis of clay

Clay (%)	Silt (%)	Fine sand (%)	Medium sand (%)	Grit (%)	Boulder (%)
48.5	37.5	10.0	3.0	1.0	0.0

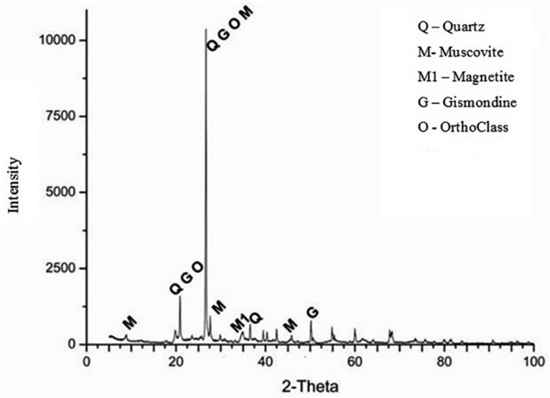

Figure 1: X-ray diffraction pattern.

Table 3 highlights the chemical composition of the clay sample. High levels of silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3) are clearly apparent, showing that the clay sample shows potential for alkali activation. Due to high levels of alkali oxides, the sample can be classified as clay, a class typically used to obtain materials with special properties[18].

The sawdust used in this work was collected from a sawmill located in São Carlos/SP. Table 4 displays the results of the particle size analysis of the sawdust, that's predominantly composed of fine grains.

Table 5 highlights the characteristics of the sodium silicate, which was produced by ISOTEC Metallurgy Products, a company from São Carlos/SP.

3.2. Characterization of the calcined clay lightweight samples

After the heat treatment, the samples may be considered calcined ceramics. Table 6 displays the values of apparent porosity and water absorption of the calcined samples.

A higher percentage of wood sawdust in the samples contributes to the increase of water absorption. The aggregate produced with just water and clay (100C-0WS) demonstrates a water absorption value of 18.21% while the 30% wood sawdust sample (70C-30WS) demonstrates a water absorption value of 47.27%.

Furthermore, the addition of sodium silicate in the ceramic pastes decreases the water absorption capabilities of the fired products, varying from 9.80% (100C-0WS-Si) to 43.87% (70C-30WS-Si).

The addition of wood sawdust has a stronger effect on water absorption relative to the addition of the sodium silicate. An increasing amount of sawdust reduces the amount of clay that can potentially react with the sodium silicate, which affects the efficiency of the alkali activator in the geo-polymerization process. Moreover, Table 6 demonstrates that a higher wood sawdust percentage increases the apparent porosity of the fired samples, because, when exposed to temperatures above 600 °C, the sawdust can turn into gas, producing voids in the final products, making them more porous and permeable. Higher apparent porosity makes the calcined ceramics less dense and more porous, as shown in Figure 2.

Table 3: Chemical composition of clay

SiO_2 (%)	TiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	MnO (%)	MgO (%)	CaO (%)	Na_2O (%)	K_2O (%)	P_2O_5 (%)
68.18	0.614	14.42	4.88	0.088	1.81	0.32	0.31	4.20	0.110

Table 4: Chemical composition of clay

opening (mm)	4.8	2.4	1.2	0.6	0.3	-
Retained mass (g)	1.29	9.47	21.60	106.11	517.30	296.02
%	0.14	0.99	2.27	11.15	54.35	31.10

Table 5: Characteristics of sodium silicate

Analysis	Unit	Specification	Result
Density (25° C)	g/cm³	1.560 to 1.580	1.567
Specific mass (25° C)	°Bé	52.00 to 53.00	52.500
SiO_2	cp	32.50 to 33.50	33.490
Na_2O	%	14.50 to 15.30	15.210
Relation (SIO_2/Na_2O)	%	2.12 to 2.31	2.200
Viscosity (25° C)	%	900.00 to 1200.00	1100.00

Table 6: Water absorption and apparent porosity of calcined ceramics

Sample	Water absorption (%)	Standard deviation	Apparent porosity (%)	Standard deviation
100C-0WS	18.21	0.25	30.40	0.22
90C-10WS	25.27	1.46	31.68	1.64
80C-20WS	35.83	1.33	37.09	1.09
70C-30WS	47.27	0.69	44.02	0.55
100C-0WS-Si	9.80	0.67	15.26	1.07
90C-10WS-Si	18.42	0.41	22.42	0.31
80C-20WS-Si	25.84	0.35	26.47	0.43
70C-30WS-Si	43.87	0.84	43.19	1.06

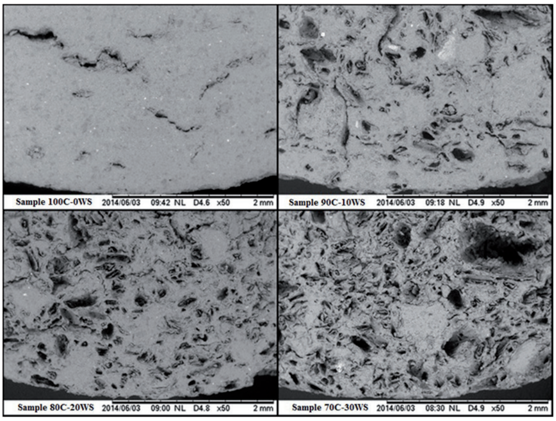

Figure 2: Electron microscopy images of the calclined aggregates made with different amount of wood sawdust.

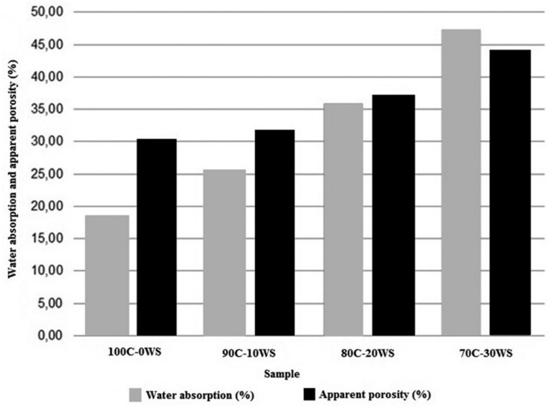

Figure 3: Water absorption and apparent porosity of calcined aggregates made with clay and wood sawdust, fired out at 900ºC

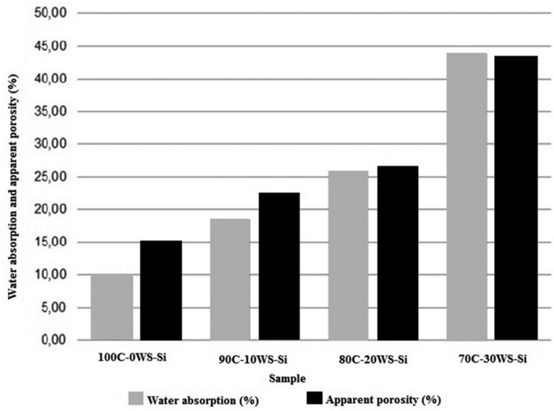

Figure 4: Water absorption and apparent porosity of calcined aggregates made with clay and wood sawdust and sodium silicate, fired out at 900ºC

Water absorption values of expanded clay aggregates are 6% (aggregates with dimension varying from 0 mm to 4.8 mm), 7% (aggregates with dimension varying from 6.3 mm to 12.5 mm) and 10% (aggregates with dimension varying from 12.5 mm to 19 mm). Relative to the samples presented in Table 6, it can be seen that specimens made just with clay, water and sodium silicate (100C-0WS-Si) presented water absorption values (9.80%) at the same level that those observed for the expanded clay aggregates[2].

In Figures 3 and 4, the relative increase in water absorption and apparent porosity are compared for the calcined clay samples. A share of at least 20% wood sawdust increases both water absorption and apparent porosity significantly. As demonstrated before, the addition of sodium silicate reduces the water absorption and apparent porosity of the calcined samples, until a maximum of 20% wood sawdust. Higher percentages of wood sawdust lead to interference with the geo-polymerization, due to the lower amount of clay available for reaction. The interference with the geo-polymerization is caused by the firing of sawdust. When exposed to temperatures above 600 ºC, the sawdust can turn into gas. The elimination of the gases in the final products can produce voids, making them more porous and permeable. This effect is strengthened due to the absence of a waterproof external layer. As a result the calcined samples with high levels of sawdust are more absorbent.

Complementary to the water absorption and apparent porosity tests, the specific mass and compressive strength of the calcined aggregates were analyzed (Table 7).

The specific mass of the calcined aggregates decreases with the addition of wood sawdust, as expected. The values vary between 1.76 g/cm³ (100C-0WS) and 1.15 g/cm³ (70C-30WS). A higher wood sawdust percentage increases the porosity of the calcined ceramics, which leads to a reduction of the specific mass of the final products.

Table 7: Specific mass and compressive strength of calcined aggregates

Sample	Specific mass (g/mm^3)	Standard deviation	Compressive strength (MPa)	Standard deviation
100C-0WS	1.76	0.02	21.11	4.29
90C-10WS	1.51	0.04	17.92	3.94
80C-20WS	1.32	0.03	13.12	1.04
70C-30WS	1.15	0.02	8.30	1.18
100C-0WS-Si	1.92	0.02	25.81	3.95
90C-10WS-Si	1.58	0.01	16.81	3.05
80C-20WS-Si	1.44	0.01	14.16	2.57
70C-30WS-Si	1.16	0.01	7.72	1.12

A similar effect is apparent in the sodium silicate aggregates, with specific mass varying between 1.92 g/cm^3 (100C-0WS-Si) and 1.16 g/cm^3 (70C-30WS-Si). However, the addition of the sodium silicate tends to increase the specific mass of the samples, due to the geo-polymerization process during the heat treatment. The geo-polymerization process produces more dense and less porous aggregates.

The aggregate made just with clay, water and sodium silicate (100C-0WS-Si) has the highest value of specific mass (1.92 g/cm^3), the lowest value of water absorption (9.80%) and the lowest value of apparent porosity (15.26%) among all analyzed aggregates, due to the stronger geo-polymerization process (high clay availability and no sawdust).

Expanded clay lightweight aggregates have specific mass values varying from 1.51 g/cm^3 (aggregates with diameter until 4.8 mm) to 0.64 g/cm^3 (aggregates with diameter from 12.5 mm to 19 mm). Comparing the values presented on Table 7 with the values presented by expanded clay aggregates and basalt aggregates (specific mass equal to 2.95 g/cm^3), the calcined clay samples of this research present intermediary values of specific mass[3].

The compressive strength test utilized to characterize the calcined aggregates is based on the compressive strength test for cylindrical specimens of concrete[15]. The compressive strength of the aggregates decreases with the increasing amount of wood sawdust (see Table 7). A higher percentage of wood sawdust makes the clay samples more porous, less resistant aggregate.

Furthermore, the aggregates with the added sodium silicate demonstrate an increase of compressive strength, due to the geo-polymerization process. This process results in more compact, less porous aggregates with a higher compressive strength resistance.

Expanded clay lightweight aggregates present compressive strength resistance of 8.4 MPa, for aggregates with diameter varying from 12.5 mm to 19 mm. Basalt aggregates present compressive strength resistance of 120 MPa for samples with similar diameter. Consequently, the calcined clay lightweight samples of this research present intermediary values of compressive strength relative to expanded clay lightweight aggregates and basalt aggregates[2].

4. Conclusion

The use of wood sawdust in calcined clay aggregates provides a significant reduction in specific mass relative to clay-only aggregates. However, the aggregates do become more porous and water absorbent. The addition of an alkali activator to the calcined clay aggregates can counteract these negative effects partially due to a geo-polymerization process, which results in more dense, resistant and less water absorbent aggregates.

The use of a mixture of wood sawdust and sodium silicate in the aggregate products allows for the production of materials with special characteristics (more resistant and less absorbent, with lower values of specific mass). These mixtures demonstrate to be a viable alternative to produce lightweight aggregates in structural concrete, due to the possibility to produce lightweight aggregates with same characteristics of expanded clay aggregates but with an energetic gain.

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