

Evaluation of the Effect of the Incorporation of Blends of Fuel and Fluxing Wastes in Red Clay Ceramics

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The clay samples endure the incorporation of industrial and urban wastes, which do not cause large variations in their properties if added in controlled quantities. Wastes can be classified as fuel, fluxing and property affecting wastes. The fuel wastes usually have a high amount of organic matter, that when heated cause exothermic reactions, releasing heat to the process. The fluxing wastes cause a reduction in the ceramic melting points. The main aim of this work was to study the behavior of ceramics incorporated with fuel wastes (*Eichhornia crassipes* dry biomass) and fluxing wastes (granite) as well as blends of both wastes. Different compositions were prepared with incorporation of different percentages of these wastes in the clay samples, conformed by uniaxial pressing and fired at 550, 650, 750, 850, 950 e 1050°C. The technological properties tested were: dry apparent density, linear shrinkage, water absorption and flexural rupture strength. Results indicate that the high calorific value of the biomass waste represents a great possibility of saving energy for the firing process. Incorporations can be indicated as a possibility of correct destination for the investigated wastes.

Keywords: Red ceramics, fuel wastes, fluxing wastes, *Eichhornia crassipes*, granite.

1. Introduction

An increasing amount of all waste types is resulted by the human consumption habits combined with current industrial activities. The wastes are produced and when their final disposal is performed improperly, it can generate series of disorders like landscape, fauna and flora destruction, air and soil contamination bringing health risks to the population. One of the ways to minimize that impact is the waste reuse¹.

Therefore, it is important to find practical and economic waste destinations. The incorporation of industrial and urban wastes in red ceramic matrices has been widely used nowadays, both as alternative raw materials and as an environmentally correct waste disposal. The masses used in ceramic industry are heterogenic by nature, resulting in the presence of residual materials from different types and sources, even in high percentages². Waste incorporation can be beneficial to the final product, since the waste may have chemical or mineralogical compositions of interest to the ceramic industrial process or they can provide heat of combustion which reduces the energy consumption during the firing stage³.

According to Vieira and Monteiro⁴, the different types of solid wastes that can be incorporated into red ceramics can be classified in three categories according to their nature and to the ceramic products properties.

- Fuel wastes – wastes that present high calorific value due to its high concentration of organic matter that contributes energetically to the ceramic sintering process through the energy release from the exothermic firing reactions.
- Fluxing wastes – wastes containing alkaline and earth alkaline compounds that in reaction with silica and alumina form liquid phases, increasing densification, reducing the melting point, helping to reduce the sintering temperatures.
- Property affecting wastes – wastes containing substances that modify ceramic behavior such as those that interfere in mechanical resistance, water absorption and linear shrinkage, but cannot be included in the first two categories⁴.

Several works study the effects of these different wastes such as stone, glass, red mud, biomass, different ashes and sludge incorporated into red ceramics separately⁵⁻¹³. However, few works mix two different waste types to analyze the effects of their incorporation blended. As fuel wastes tend to increase ceramic porosity after firing, its use combined with fluxing wastes may minimize this deleterious effect.

The objective of this work was to evaluate the effect of the incorporation of fuel (dry *Eichhornia crassipes* biomass) and fluxing (granite) wastes in the properties of clay ceramics

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fired at different temperatures, as well as incorporated with blends of the two waste types with different proportions.

2. Materials and Methods

Clay ceramic, dry macrophyte biomass and granite waste were the raw materials used in this work. The clay ceramic was collected at ArtCerâmica Sardinha, located in the city of Campos dos Goytacazes, Rio de Janeiro, Brazil, and it is constituted by 1:1 portions of “weak” and “strong” yellow clays. The *Eichhornia crassipes* biomass was collected at Lagoa do Vigário also located in Campos dos Goytacazes. The granite waste was collected in a sawmill in the city of Cachoeiro de Itapemirim, Espírito Santo, Brazil, from the filter press waste pile.

The clay ceramic is typical clay mass normally used by local ceramic industries in the production of bricks and tiles. Dry biomass waste is from *Eichhornia crassipes* (an aquatic macrophyte popularly known as *aguapé*), a common plant in local rivers and lagoons, most often introduced on purpose by man to purify water. However, the excess of this plant in water surface caused by its high reproduction rate presents damage to the ecosystem. Granite waste comes from granite industry, due to sawing and polishing process of granite rocks with diamond wire technology.

The clay ceramic was dried at 110°C for 24 hours, and then separated in a jaw crusher, disintegrated in a porcelain mortar and sieved to 42 mesh powder.

The biomass was washed in running water, dried in an air circulation stove and then in a laboratory stove at 60°C. After dried, the material was ground in a knife mill and sieved through a 24 mesh sieve. The granite waste was dried at room temperature for excessive moisture loss and dried in a stove at 110°C, then sieved at 42 meshes.

Waste characterization was made to evaluate their behaviors as fuel or fluxing wastes. Differential scanning calorimetric (DSC) and thermogravimetric (TG) analysis were performed to evaluate the thermal behavior and the nature of the reactions of the *Eichhornia crassipes*. The DSC/TG analyses were done with a heating rate of 10°C/min in a SDT Q600 V20.9 Build 20. To evaluate its behavior as a fuel, heat combustion analysis was performed according to ASTM D5865-13¹⁴.

X-ray fluorescence (XRF) analysis was performed in the granite waste in AXIOS Paranalytical spectrometer to evaluate and quantify the presence of alkaline and earth alkaline compounds.

Particle size distribution of clay ceramic and granite waste were done by sifting and sedimentation process while particle size distribution of biomass was investigated by laser diffraction. Optical microscopy (OM) was performed on an OLYMPUS Microscope model LEXT OLS4000 3D.

Table 1 presents the five compositions formulated of clay ceramic samples incorporated with different percentages

of wastes. The compositions were prepared with blend additions of different biomass and granite waste weight percentages in clay ceramic. Those specific percentages were chosen based on the literature review which showed that red ceramics incorporated with up to 5%w of biomass^{9,15} and up to 30%w of granite⁸ do not present depreciation in mechanical properties.

Table 1. Nomenclature and compositions (%weight).

Composition	Clay Ceramic (%)	DryBiomass (%)	Granite Waste (%)
0	100	0	0
2.5B20G	77.5	2.5	20
2.5B30G	67.5	2.5	30
5B20G	75	5	20
5B30G	65	5	30

The samples of the five compositions prepared were wetted with 8% water and conformed by uniaxial pressing in a hydraulic press in 15 MPa, set in a rectangular steel mold (114 X 25 mm). The samples were dried at room temperature and in a laboratory stove at 110°C for 24 hours, then fired at 550, 650, 750, 850, 950 and 1050°C in a laboratory oven. The heating rate used was 3°C/min maintaining the maximum temperature for an hour in each firing temperature mentioned before and with a natural convection cooling after shutting off the oven.

In order to verify the mobility of pollutants leaching and solubilizations tests, according to NBR 10005¹⁶ e NBR 10006¹⁷, were performed in granite and biomass. The elements, as well as other substances required by the NBR 10004¹⁸ standard were determined in the solubilization and leaching extracts.

In order to evaluate the mechanical properties, the dry apparent density, linear shrinkage and water absorption tests were done according to ASTM C373-72¹⁹ and the three-point flexural rupture strength based on the ASTM C674-77²⁰.

3. Results and Discussion

Figure 1 shows the DSC and TG biomass analysis, performed to investigate its thermal behavior.

In Figure 1 it is noticed that around 100°C there is an endothermic peak that characterizes biomass loss of adsorbed water, associated with an average mass loss. Two well-defined exothermic peaks between ±280 e 525°C, associated with a ±55% mass loss which shows that the biomass releases heat in this temperature range assisting sintering process of clay ceramics through heat releasing as well as energy savings during the firing process.

Table 2 shows the chemical composition of the granite waste, which is similar to one of the clays used to prepared the red ceramics.

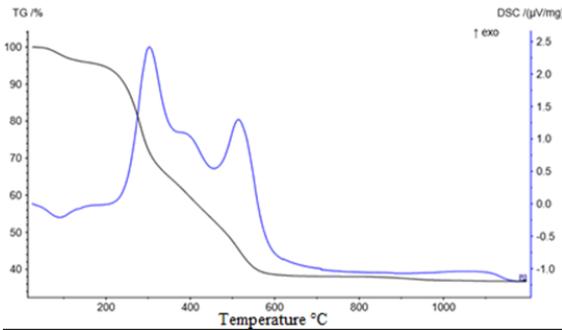


Figure 1. DSC/TG *Eichhornia crassipes* dry biomass curves.

It is possible to observe the presence of SiO_2 (69.9%) followed by Al_2O_3 (17%) and a significant amount of alkaline oxides (K_2O and Na_2O), in a total of 8.9%, in contrast with the local clays collected in Campos dos Goytacazes/RJ (Brazil), that naturally have a low flux content⁸ as a consequence of being kaolinitic clays²¹. The alkaline oxides may act as fluxes, improving the sintering process by reacting with silica and alumina to form liquid phase by eutectic reaction⁸.

Another advantage of the granite waste is its low content of 1.3% Fe_2O_3 , when compared to the clays collected in Rio de Janeiro/RJ (Brazil)²². The absence of iron compounds in the clay masses not only avoids damage to the wear of processing equipment, but also ceramic clays with a content below 3% Fe_2O_3 are indicated for the manufacture of light-colored products.

One final comment on the results of Table 2 is that the granite waste low fire loss (LoI) of 0.55% indicates thermal stability. Excessive LoI can cause more retraction and porosity after firing.

Figure 2 shows the particle size distribution curves of raw materials. In this figure, the different particle size range is associated with the clay, silt and sand classification²³. The clay fraction is associated to particle sizes smaller than 0,002 mm, the silt fraction between 0,002 e 0,02 mm and the sand fraction particles bigger than 0,02 mm. The clay fraction provides the clay samples greater plasticity since in its range of particle size, there is a higher percentage of clay minerals. The sand fraction represents coarser particles being associated with quartz and responsible for the adjustment of workability, reduction of plasticity and linear shrinkage²³.

Table 3 shows the particle size distribution of raw materials. Granite waste presents granulometry distributed in 85.6% of silt, 11.9% of clay and only 2.5% of sand. Biomass presents granulometry distributed in 83.3% of sand, 15.6% of silt and only 0.6% of clay. Both presented coarser granulometry than the clay ceramic (43% of clay, 42% of silt and 15% of sand).

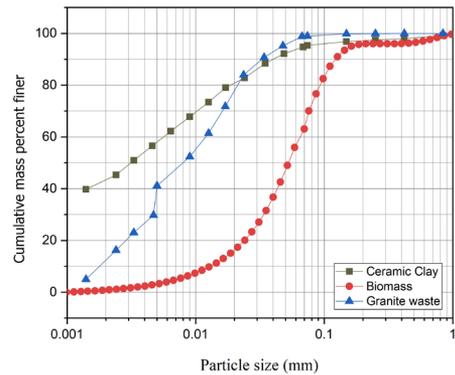


Figure 2. Particle size distribution of raw materials.

Despite of the wastes presenting a coarser granulometry than the clay, they have enough fine granulometry to be used in the formulation of the clay body.

Figure 3 shows the images of raw materials obtained by optical microscopy.

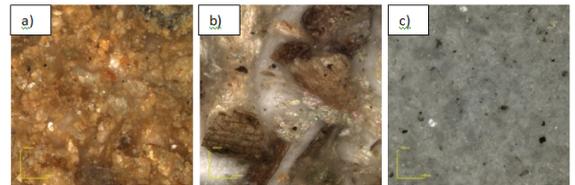


Figure 3. Images by Optical microscopy a) ceramic clay b) biomass c) granite waste (430x).

In Figure 3a it can be noticed different size particles and also it's possible to observe quartz particles, as well as dark and red particles associated with iron compounds. In Figure 3b diversified size particles can be observed, mostly large, with irregular shape. In Figure 3c it is possible to analyze a large number of agglomerates and thin particles and it is possible to observe dark and red particles associated with minerals founded in the granite waste, such as biotite and ferromagnetic minerals²⁴.

When comparing the leaching results for metals are presented in Table 4 with the maximum values allowed by NBR 10004 – Annex F¹⁸, it is verified that the obtained parameters did not exceed allowed limits, thus, granite waste and *Eichhornia crassipes* dry biomass do not present toxicity. Therefore, in relation to the analyzed metals both are classified as being Class II, non-hazardous wastes.

The results of the solubilization test of the wastes are presented in Table 5 and comparing those results with the maximum values allowed by NBR 10004 – Annex G¹⁸ it

Table 2. Chemical composition of granite waste (wt.%).

Components	SiO_2	CaO	K_2O	Na_2O	Al_2O_3	Fe_2O_3	MgO	P_2O_5	TiO_2	LoI
	69.9	1.5	3.4	5.5	17	1.3	0.46	0.14	0.24	0.55

Table 3. Particle size distribution of raw materials (%).

	Particle Size Distribution of Raw Materials (%)		
	Clay Fraction	Silt Fraction	Sand Fraction
Clay ceramic	43	42	15
Biomass	0.6	15.6	83.3
Granite waste	11.9	85.6	2.5

Table 4. Analytical results of the leaching extract.

Parameters	NBR 10004:2004 (mg/L)	Granite waste (mg/L)	Results	Biomass (mg/L)	Results
Arsenic	1	0.02	Inside the limit	0.03	Inside the limit
Barium	70	0.73	Inside the limit	0.48	Inside the limit
Cadmium	0.5	<0.0004	Inside the limit	< 0.0002	Inside the limit
Lead	1	0.03	Inside the limit	<0,008	Inside the limit
Total chrome	5	<0.001	Inside the limit	0.01	Inside the limit
Mercury	0.1	<0.009	Inside the limit	0.05	Inside the limit
Silver	5.0	<0.005	Inside the limit	< 0.005	Inside the limit
Selenium	1	<0.008	Inside the limit	0.05	Inside the limit

is verified that for granite waste, the parameter aluminum above the maximum limit allowed. Regarding the biomass, the parameters iron, manganese, mercury and sodium are above the maximum limits allowed. Thus, the both wastes are classified as Class II A - non-inert in relation to the metals analyzed. These metals come from the *Eichhornia crassipes* phytoremediation assimilation, which is a characteristic of this plant, to absorb chemical elements from the environment in which they developed²⁵. The Vigario Lagoon, where the plants were collected to prepare dry biomass for this research is polluted due to the discharges of domestic and industrial effluents and garbage²⁶.

Figure 4 shows the clay samples' location with different percentages of blend in an extrusion prognostic diagram based on the Atteberg limits.

As shown in Figure 4, the ceramic clay is outside the region of acceptable plasticity for extrusion. The addition of blend compositions to the clay samples provides an acceptable extrusion workability.

Figure 5 shows the dry apparent density of the 5 studied compositions.

The denser the ceramic samples, the better "packaged" it is, because its grains are closer and there are less empty spaces between them. It is beneficial in the ceramic industry because the increase of particles contact area helps sintering. However, the increase in the densification causes a decrease in the ceramic sample's permeability, which can impair the drying and the organic matter elimination during firing cycle. Statistically, it can be observed that the granite and biomass waste incorporation did not change the dry apparent density when comparing to the pure ceramic clay.

Figure 6 shows the linear shrinkage of all fired compositions.

In Figure 6 it can be observed that the linear shrinkage increases according to the increase of firing temperature. This is due to more efficient sintering at higher temperatures, which results in a larger pore closure and reduction of the bodies' dimensions²².

From Figure 6 it is also noticed that the linear shrinkage tends to have a small decrease with blend incorporation, and this decrease is more pronounced in those compositions that contain the highest amount of granite waste. At 1050°C there was an increase in linear shrinkage for all compositions due to its higher liquid phase formation, because of the granite fluxing action, as discussed above.

Figure 7 shows the percentage water absorption of the compositions.

In Figure 7 it is possible to notice that at 1050°C, water absorption decreases which is due to more effective sintering at higher temperatures, resulting in a greater closure of open pores, thus reducing water absorption. The lower water infiltration in the ceramic body determines, for example, the greater durability and resistance to the environment to which the material is exposed.

It should be noted that the compositions with the lowest amounts of biomass cause a decrease in the water absorption and the ones with the highest amounts of biomass caused an increase. At 1050°C all blend compositions caused a decrease in water absorption. The smaller the amount of biomass waste and the greater the amount of granite waste, the greater the water absorption reduction was, due to the liquid phase formation by the present fluxing oxides.

These results are explained by the combustion of the biomass organic matter during the firing stage, associated with a mass loss that causes ceramic porosity, a natural behavior

Table 5. Analytical results of the solubilization extract.

Parameters	NBR 10004:2004 (mg/L)	Granite waste (mg/L)	Results	Biomass (mg/L)	Results
Aluminium	0.2	3.85	Out of limit	<0.007	Inside the limit
Arsenic	0.01	<0.008	Inside the limit	<0.008	Inside the limit
Barium	0.7	0.26	Inside the limit	0.29	Inside the limit
Cadmium	0.005	<0.0004	Inside the limit	< 0.0002	Inside the limit
Lead	0.01	<0.008	Inside the limit	<0.008	Inside the limit
Copper	2	<0.01	Inside the limit	< 0,0003	Inside the limit
Total chromium	0.05	<0.001	Inside the limit	< 0.001	Inside the limit
Iron	0.3	0.19	Inside the limit	0.35	Out of limit
Manganese	0.1	0.06	Inside the limit	14.7	Out of limit
Mercury	0.001	<0.009	Inside the limit	0.065	Out of limit
Silver	0.05	<0.005	Inside the limit	<0.005	Inside the limit
Selenium	0.01	<0.008	Inside the limit	0.055	Out of limit
Sodium	200	19	Inside the limit	226	Out of limit
Zinc	5	0.065	Inside the limit	0.77	Inside the limit

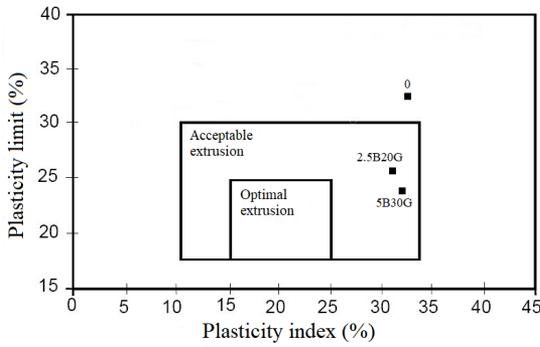


Figure 4. Extrusion prognostics.

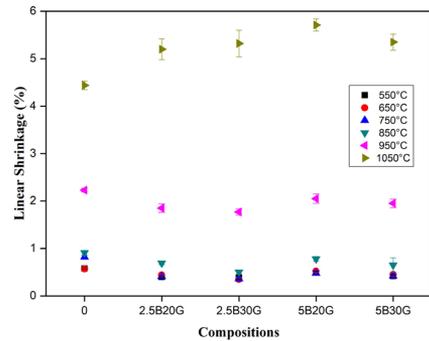


Figure 6. Linear shrinkage of compositions.

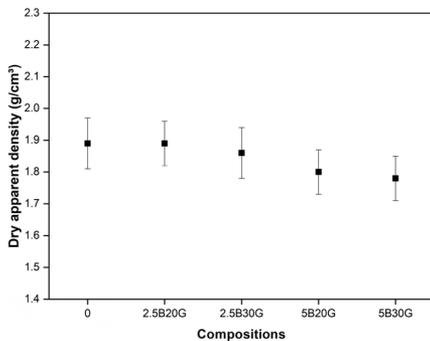


Figure 5. Dry apparent density of compositions.

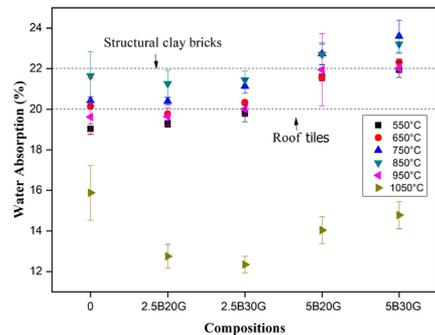


Figure 7. Water absorption of compositions.

of this type of residue, as evidenced by thermal analysis. Meanwhile, at 1050°C the formation of liquid phase helps to fill these pores and the water absorption values decrease.

For structural clay bricks, according to NBR ISO 15270:1 (2017), the water absorption index must not be less than 8%

or more than 25%²⁷. The results obtained for all studied compositions meet the normative limits for structural clay bricks. NBR ISO 15310 (2009) indicates that the maximum permissible limit of water absorption for ceramic roof tiles is 20%²⁸. All compositions burned at 1050°C and also

compositions with 2.5% of biomass burned at 550, 950 and 1050°C are suitable for roof tiles manufacture.

Figure 8 shows flexural rupture strength of the compositions.

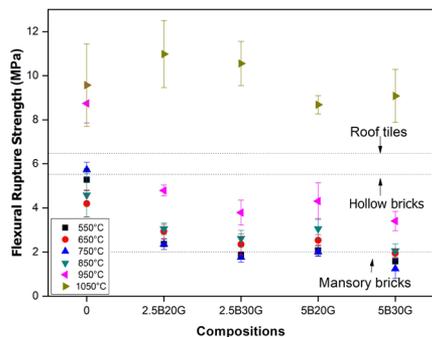


Figure 8. Flexural rupture strength of compositions.

The flexural strength, or stress at which the material breaks, is the most important property for structural material and this property increases with the temperature increase, mainly to 1050°C. As previously discussed, this is due to the sintering mechanisms that allow greater formation of liquid phase, thus reducing the porosity of the material and promoting a better consolidation of the particles.

In Figure 8 it is noticed that the blend incorporation causes a decrease in the flexural rupture strength, except for the ones burned at 1050°C, in which the compositions with the smaller biomass amount (2.5%) cause an increase.

The minimum resistance required for the masonry bricks manufacture is 2MPa, for hollow bricks is 5.5MPa and for roof tiles is 6.5MPa²³. The results obtained at 1050°C for all compositions are above the minimum strength for manufacturing of these three products. Composition with highest amounts of granite, 2.5B30G and 5B30G, are above the minimum strength for masonry bricks when fired at 550 and 750°C.

The heat of combustion of a fuel material is divided into upper and lower heat of combustion and it is expressed by the energy content that is released from the materials when it is fired with air atmosphere. The higher heat of combustion is the parameter of interest for this research, and it refers to the amount of calories released by the material in its combustion. The higher this the high heat of combustion is, the bigger the energy contained in the fuel²⁹. The heat of combustion obtained for the dry biomass of *Eichhornia crassipes* was 2.389 kcal/kg.

For economic and energy analysis, data on the energy savings provided by the incorporation of 2.5%w biomass in the ceramic mass will be provided. The energy saving survey was carried out for the firewood burning vaulted oven considering 900°C threshold temperature.

Data:

- ✓ Firewood heat of combustion: 2.600 kcal/kg.
- ✓ *Eichhornia crassipes* biomass heat of combustion: 2.389 kcal/kg.
- ✓ Oven consumption of vaulted oven: 780.000 kcal/T.

To produce 1 ton of the final product using firewood as fuel, the vault oven spends, on average: (oven consumption (kcal/ton)/ firewood heat of combustion (kcal/kg)) = 780.000/2.600 = 300 kg of firewood.

With the incorporation of 2.5% biomass waste, there is a reduction in the oven energy consumption reduction of: (amount of waste (kg)x(biomass heat of combustion (kcal/kg)) = 25x2.389 = 59.725 kcal.

With this data it is possible to calculate the percentage of energy saved during the firing, which will be: (calories provided by biomass)/(oven consumption) = 59.725/780.000 = 7.7%.

However, the theoretical heat required for firing 1kg of ceramic clay is 256 kcal. This shows that the efficiency of the vault oven is about 30%.

Considering that all the heat generated by the waste would be used, the firewood economy would be: firewood calories needed to provide the same energy from biomass = 100x59.725/30 = 199.083 kcal

This is the energy saved with the incorporation of 2.5% biomass in ceramic clay and corresponds to a real reduction of 25% of the energy from the firewood (59.725/780.000).

4. Conclusions

Regarding the objectives of this work, it can be noticed that the *Eichhornia crassipes* biomass waste and granite waste have suitable characteristics to be used as raw materials in the ceramic industry. The characterization results, as well as their influences in ceramic properties showed that they behaved as fuel and fluxing wastes, respectively, as proposed.

Both wastes were classified as non-hazard – Class II A (non-inert) according to NBR 10004.

Thus, it is evident the direct influence of both wastes and their blends in the technological ceramic properties evaluated:

- Blend compositions did not cause significant changes in the ceramic dry apparent density.
- With the blend incorporation, linear shrinkage presented a decrease for ceramic samples burned at all tested temperatures, except 1050°C, which presented an increase.
- Compositions with lower amount of biomass presented a decrease in the water absorption while the ones with the higher amounts of biomass cause an increase of that.
- Flexural rupture strength decreases with blend incorporations, except for the ones fired at 1050°C,

in which the compositions with the smaller biomass amount (2.5%) caused an increase of the property.

The heat of combustion of *Eichhornia crassipes* biomass is high, releasing heat during the firing process generating energy saving and a shorter firing time. The incorporation of 2.5%w biomass in mass reduces around 25% of energy from the firewood (commonly used fuel).

In addition to the improvements of some ceramic mechanical properties, the use of these wastes and their blends allows the recycling of a material that could be unduly disposed, causing environmental damage.

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