Shadia Ikhmayies · Jian Li · Carlos Mauricio Fontes Vieira · Jean Igor Margem · Fabio de Oliveira Braga
Editors

Green Materials Engineering
An EPD Symposium in Honor of Sergio Monteiro
Preface

Green engineering is the design, use of processes, and development of products that conserve natural resources, reduce pollution, and exert the smallest possible impact on the environment. Green engineering often promotes sustainability and minimizes risk to human health without incurring economic feasibility and efficiency. So, green engineering is not actually an engineering discipline in itself, but a comprehensive framework for all engineering disciplines. The Green Materials Engineering symposium is a TMS Extraction and Processing Division (EPD) symposium, sponsored by the Characterization of Minerals, Metals, and Materials and the Biomaterials Committees. This symposium is held in honor of Professor Sérgio Monteiro from the Military Institute of Engineering, IME, Brazil. The symposium focuses on green materials including natural composites, bio-inspired armors, waste, clays added ceramics, lignocellulosic fibers, biodegradable polymers, and any type of natural material that could be related to engineering applications.

The Green Materials Engineering symposium held during the TMS 2019 Annual Meeting in San Antonio, Texas, USA, received 54 abstract submissions from different research groups, of which 34 were accepted as oral presentations and 20 accepted as posters. Of the presented papers, 33 are published in this book after being peer reviewed. These papers cover different fields including sustainable clays and ceramics, natural fiber composites, nano- and microgreen composites, properties and characterization of green materials, and biomass in armor composites. These materials are characterized using highly sophisticated techniques to examine their microstructure, mechanical, thermal, and functional properties.

This book will appeal to people from academia and industry who are interested in green engineering, sustainability, recycling, and environment, and it can help academia emphasize pollution prevention and incorporate risk into green engineering courses. It helps environmental and chemical engineers; postdoctoral, graduate and undergraduate students; people from industry; and environmental scientists to convert concepts of green engineering and sustainability to real designs, using the most valuable quantitative design tools and performance metrics.
The editors of this book express their genuine thanks and gratitude to TMS for giving the organizers and committee sponsors the opportunity to publish a stand-alone volume for this symposium. The editors also thank the publisher, Springer, who produced the book, and the authors, who are the basis of this scientific work.

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Study of Incorporation of Fuel and Fluxing Wastes in Red Ceramics

Gabriela Nunes Sales Barreto, Michelle Pereira Babisk,
Geovana Carla Giorindo Dalgua, Monica Custodi Borliai Gadioli
and Carlos Mauricio Fontes Vieira

Abstract Red ceramic industries are among the most important recyclers of industrial and urban wastes. The clays’ heterogeneity allows the incorporation of several types of wastes, which can be classified as fuel wastes, fluxing wastes, and property-affecting wastes. The fuel wastes usually have a high amount of carbon-containing 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 objective 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 into red ceramics, shaped by uniaxial pressing and fired at 850 and 1050 °C. The technological properties tested were apparent dry density, linear shrinkage, water absorption, and flexural rupture strength. Results indicate that incorporation of these wastes into red ceramics is viable, which can significantly improve the evaluated properties of the ceramics, but the quantity and the firing temperature must be controlled.

Keywords Red ceramics • Fuel wastes • Fluxing wastes • Eichhornia crassipes • Granite

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© The Minerals, Metals & Materials Society 2019
S. Iliamayes et al. (eds.), Green Materials Engineering, The Minerals,
Metals & Materials Series, https://doi.org/10.1007/978-3-030-10383-5_1
Introduction

Human consumption habits combined with current industrial activities have generated an increasing amount of waste types. Therefore, it is important to find practical and economic waste destination ways. The incorporation of industrial and urban wastes into red ceramic has been widely used nowadays, both in search for alternative raw materials and for an environmentally correct waste disposal.

The industry presents itself as an excellent alternative for different segments of waste management due to its large production, as well as traditional clay heterogeneity allows the incorporation of several types of wastes with a small sacrifice and, in some cases, improvement in the properties of the final product.

According to Vieira and Monteiro (2009), the different types of solid wastes that can be incorporated into red ceramics, according to their nature and to the ceramic products properties, can be classified in three categories:

- 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 categories [1].

Several works study the effects of these different wastes incorporated into red ceramics, but always separately: oil, paper, stone, glass, red mud, iron ore, biomass, different ashes and sludge [2–16]. However, few works mix two different wastetypes to analyze the effects of blend incorporation.

The objective of this work was to study the effect of incorporation of fuel (dry Eichhornia Crassipes biomass) and fluxing (granite) wastes in the properties of clay ceramics fired at different temperatures, as well as incorporated with blends of the two waste types with different proportions.

Materials and Methods

Materials

The raw materials used in this work were ceramic clay, dry macrophyte biomass waste, and granite waste. The ceramic clay was collected at ArCerâmica Sardinha, located in the city Campos dos Goytacazes, in 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 the 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, from the filter press waste pile.

**Preparation of Materials**

The ceramic clay was dried at 110 °C for 24 h in a stove of the laboratory of Advanced Materials, LAMAV, of the State University of Northern Rio de Janeiro, Brazil. The dried ceramic clay was then separated in a jaw crusher, disintegrated in a porcelain mortar and in 42 mesh powders.

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 laboratory stove at 110 °C, then sieved at 42 mesh.

**Methods**

**Waste Characterization**

Waste characterization was made to evaluate their behaviors as fuel and fluxing wastes. Differential scanning calorimetric (DSC) and thermogravimetric (TG) analysis were made in *Eichhornia crassipes* biomass to evaluate its thermal behavior and the reactions' nature. X-ray fluorescence (XRF) analysis was made in the granite waste to evaluate and quantify the presence of alkaline and earth alkaline compounds.

**Preparation of Compositions**

Table 1 presents the nine compositions formulated for the clay ceramic bodies incorporated with different percentages of wastes. The compositions were prepared with clay ceramic with additions of up to 5% biomass waste and up to 30% granite waste, as well as blend of both wastes.

**Confection of Ceramic Bodies**

The nine prepared composition masses were wetted with 5% water and conformed by uniaxial pressing in a hydraulic press with 15 TNF compression pressure, in a rectangular steel die (114 × 25 mm) to prepare the ceramic bodies. The samples were dried at room temperature and in a laboratory stove at 110 °C for 24 h, then fired at 850 and 1050 °C in a laboratory oven. The heating rate was 3 °C/min with
Table 1: Nomenclature and compositions (weight %)

<table>
<thead>
<tr>
<th>Composition</th>
<th>Ceramic clay (%)</th>
<th>Dry biomass (%)</th>
<th>Granite waste (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>100</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>B</td>
<td>80</td>
<td>0</td>
<td>20</td>
</tr>
<tr>
<td>C</td>
<td>70</td>
<td>0</td>
<td>30</td>
</tr>
<tr>
<td>D</td>
<td>97.5</td>
<td>2.5</td>
<td>0</td>
</tr>
<tr>
<td>E</td>
<td>95</td>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>F</td>
<td>77.5</td>
<td>2.5</td>
<td>20</td>
</tr>
<tr>
<td>G</td>
<td>67.5</td>
<td>2.5</td>
<td>30</td>
</tr>
<tr>
<td>H</td>
<td>75</td>
<td>5</td>
<td>20</td>
</tr>
<tr>
<td>I</td>
<td>65</td>
<td>5</td>
<td>30</td>
</tr>
</tbody>
</table>

an hour at the maximum temperature, with natural convection cooling after shutting off the oven.

Determination of Physical and Mechanical Properties

In order to evaluate the use of biomass as a raw material for the ceramic industry, the dry apparent density, linear shrinkage, and water absorption tests were done according to ASTM C373-72 [17] and the three-point flexural rupture strength, in accordance with ASTM C674-77 [18].

Results and Discussion

Figure 1 shows the DSC and TG biomass analysis, which performed to investigate its thermal behavior. In this figure, one should notice that around 100 °C there is a little endothermic peak that characterizes biomass loss of adsorbed water, associated with an average mass loss. Two well-defined exothermic peaks between 200 e 600 °C, associated with a ±55% mass loss, show that biomass releases heat in this temperature range.

This released heat will aid the 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 that of clays used to make red ceramics. It is possible to observe the presence of SiO₂ (69.9%) followed by Al₂O₃ (17%), the significant amount of alkaline oxides, K₂O and Na₂O, in a total of 8.9% which act as fluxes and contribute to the formation of liquid phase and sintering of the ceramic part during firing [19].
Another granite waste advantage is its low content of 1.3% Fe₂O₃, when compared to the clays used in Rio de Janeiro/RJ (Brazil) [20, 21]. 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% Fe₂O₃ are indicated for the manufacture of light-colored products.

One final comment on the results of Table 2 is that the granite waste Low loss on ignition (LOI), 0.55%, indicates thermal stability. Excessive LOI can cause greater retraction and porosity after firing.

Figure 2 shows the dry apparent density of the nine studied compositions. The denser the ceramic body, the better "packaged" it is, because the closer its grains are, 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 body's permeability, which can impair the drying and the organic matter elimination during firing cycle. Statistically, one can observe that the granite and biomass waste incorporation did not change the dry apparent density when comparing to the pure ceramic clay.

Figure 3 shows the linear shrinkage of all compositions fired at 850 and 1050 °C. In this figure, one can observe that the linear shrinkage increases according to the increase of firing temperature. This is due to more efficient sintering at higher temper-
Fig. 2. Dry apparent density.

Fig. 3. Linear shrinkage of compositions: a A, B, C, D, E and b A, F, G, H, I.

structures, which results in a larger pore closure and reduction of the bodies’ dimensions [22].

One can also notice that, for both temperatures, the *Eichhornia crassipes* biomass incorporation (compositions D and E) increases the linear shrinkage of ceramic clay. Granite waste incorporation (compositions B and C), however, presents two different effects depending on the firing temperature. At 850 °C, granite waste causes a decrease in linear shrinkage, and at 1050 °C it causes an increase, because at this temperature the fluxes present in granite waste have already formed liquid phase, decreasing pore volume, and as a consequence, the structure contracts.

In blend compositions (F, G, H, and I), one can also notice two different effects depending on the temperature. At 850 °C, there was a decrease in linear shrinkage for all compositions, and this decrease is more pronounced in those with the highest amount of granite waste (G and I). 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 4 shows the percentage water absorption of the compositions. In this figure, one can notice that at higher temperatures, water absorption decreases. This is because sintering is more effective at higher temperatures, resulting in a greater closure of open pores, thus reducing water absorption [22]. 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.

For the ceramics fired at both temperatures, the results indicated that the 20 and 30% granite waste incorporation (A and B) caused a decrease in water absorption comparing to the pure ceramic clay (A).

The water absorption increases with the *Eichhornia crassipes* biomass incorporation at 850 °C, including for blends, except for the composition D (2.5%), which maintains the same value of pure ceramic mass. 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 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.

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.

For 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.

For structural clay tiles, according to NBR 15270 (2005), the water absorption index must be at least less than 8% or no more than 22% [23]. NBR 15310 (2009) indicates that the maximum permissible limit of water absorption for ceramic roof tiles is 20% [24]. The results obtained for all studied compositions, at 1050 °C, meet the normative limits for both manufactured pieces above. At 850 °C, the compositions
are suitable for roof tiles manufacture, except all that incorporate 5% biomass (E, H, and I).

Figure 5 shows flexural rupture strength of the compositions. The flexural strength, or stress at which the material breaks, is the most important property for structural materials. One can observe that the flexural strength increases with the temperature increase, mainly up 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 this graph, one can observe that in comparison with the pure ceramic (A), granite waste incorporation (compositions B and C) at 850 °C decreases the flexural rupture strength and *Eichhornia crassipes* biomass incorporation (compositions D and E) increases. The organic matter combustion of biomass releases heat that contributes to the ceramic sintering process, reaction that occurs between 200 and 600 °C as presented in thermal analysis, while the granite waste was inert at this temperature and therefore the presence of quartz and other minerals may have acted as stress concentrators. At 1050 °C, there was liquid phase formation, and the granite waste incorporation was effective.

All blend compositions fired at 850 °C, when compared to the pure ceramic (A), cause a decrease in the flexural rupture strength. At 1050 °C, all blend compositions with smaller biomass amounts (F and G) caused an increase.

According to Santos (1989), the minimum resistance required for the masonry bricks manufacture is 2 MPa, for hollow bricks is 5.5 MPa, and for roof tiles is 6.5 MPa [25]. The results obtained at 1050 °C for all compositions are above the minimum strength for manufacturing of these three products.

All studied compositions, at both temperatures, are above the minimum strength for masonry bricks manufacture, except for the composition with 30% granite waste fired at 850 °C. It is noteworthy that at this temperature, all other compositions with granite waste incorporations (B, F, G, H, and I) only meet the requirements for masonry bricks manufacture and that the biomass incorporation (compositions D and E) fits the ceramic for hollow bricks manufacture.
Conclusions

Regarding to the objective of this work, one can notice that the *Eichhornia crassipes* biomass waste and granite waste own suitable characteristics for the use as raw materials in the ceramic industry. The characterization results, as well as its influences in ceramic properties, proved that they behaved as fuel and fluxing wastes, respectively, as proposed.

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

- Both the granite and biomass wastes as well as their blends did not case significant changes in the ceramic dry apparent density.
- Linear shrinkage increases for all ceramics incorporated with biomass waste. There were increasing and decreasing effects for ceramics incorporated with granite wastes and blends, with a decrease at 850 °C and an increase at 1050 °C.
- Water absorption increases with incorporation of 5% biomass waste, while granite waste incorporation caused a decrease. Blend compositions, except the ones with 5% biomass, caused a decrease in water absorption, for both temperatures.
- Flexural rupture strength, at the temperature of 850 °C, increased with biomass waste incorporation, while decreased with granite waste incorporation. At the temperature of 1050 °C, both wastes increased flexural rupture strength.

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.

Acknowledgments The authors thank the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior, CAPES, program PNPD2013/1134—310330160058 UENF/Materials Engineering and Science for the support of the research project.

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