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Characterization of ceramic bricks incorporated with textile laundry sludge

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Abstract

Solidification/stabilization (S/S) treatment of wastes is a suitable alternative to immobilize contaminants within a chosen material. This work proposes the mixing of textile laundry wastewater sludge with clay in order to produce bricks for civil construction. The applied sludge and clay were previously characterized in terms of their composition, microstructure and thermal behavior. Ceramic bricks were fabricated with different quantities of textile laundry sludge and they were evaluated in terms of their mechanical characteristics, besides environmental issues verified through leaching and solubilization tests. The obtained results showed that sludge can be incorporate until a concentration of 20% (mass basis) producing suitable bricks in terms of its mechanical properties. Besides, the produced brick are safe and inert according to the applied leaching and solubilization tests.

Keywords: Textile laundry wastewater sludge; Clay; Bricks; Characterization

1. Introduction

Textile industries generally use several chemicals and dyes at high concentrations to get characteristics like softening, brightness, and color to clothes or textiles. In this way, textile industries generate huge quantities of hazardous wastewaters containing sand and grit, lint, free and emulsified oil and grease, heavy metals and volatile organic compounds [1].

The treatment of textile wastewaters is carried out in several stages. Before the biological treatment, particles are usually agglomerated from a micro emulsion using a process of coagulation and then flocculation where the particulates are made into ever increasing larger particles and finally into sludge. Especially high in sludge production are those treatment programs reliant on bentonite clay, ferric salts, and/or aluminum salts as the main driver in the emulsion breaking reaction. The generated sludge contains high quantities of pigments, besides the chemical coagulants used for the wastewater treatment.

Worldwide, regulations about landfilling of solid wastes are becoming more and more restrictive. The Brazilian standard ABNT 10004/04 [2] classifies this solid residue as non inert. It means that this sludge must be treated, correctly disposed or by-processed.

Solidification/stabilization (S/S) technologies are designed to improve waste-handling, to decrease the surface area necessary to inhibit heavy metals leaching, to reduce the solubility of contaminant compounds, and to detoxify hazardous constituents [3]. Besides, the valorization of wastes as secondary raw materials in the production of construction materials could allay the problems associated to the depletion of natural resources and to the disposal of industrial wastes [4].

Several papers have reported the successful application of solidification/stabilization processes to incorporate wastes like grog, water treatment sludge, steel-refining slag, non-fluxing ashes, mineral processing tails, galvanic sludge, catalyst reject, tannery sludge, and construction and demolition leftovers, into different kind of materials [5,6]. The choice of the material for the solidification/stabilization process is also an important task.

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Furlani et al. [7] pointed out that the red ceramic industry is a promising alternative to recycle waste materials due to the low residual porosity of the obtained bodies.

The main objective of this work is to evaluate the incorporation of textile wastewater sludge into ceramic bricks. The applied sludge and clay were previously characterized in terms of their composition, microstructure and thermal behavior. Ceramic bricks were fabricated with different quantities of textile laundry sludge and they were evaluated in terms of their mechanical characteristics, besides environmental issues verified through leaching and solubilization tests.

2. Materials and methods

2.1. Sludge and clay samples

The sludge used in this work was collected from the wastewater treatment plant of a textile industry located in the city of Maringa-PR (Brazil). In this industry, the laundry wastewater is treated with sodium hydroxide, iron and aluminum sulfates, and cation polyelectrolytes. The sludge was collected from the coagulation/flocculation tank once a week during 6 months. The material was sun-dried during 72 h and stocked after each collection. All the collected materials were homogenized. The dried materials were then milled and passed through a 0.6 mm sieve before use.

The used clays were supplied by a local pottery industry (Maringá-PR, Brazil). The samples of clay were also sun-dried during 72 h, milled, and sieved to 0.6 mm particles.

2.2. Characterization of raw materials

The characterization of clay samples were performed through particle size distributions, and liquidity and plastic limits analyses according to Brazilian standards NBR 7181/84 [8], NBR 6459/84 [9], and NBR 7180/84 [10], respectively. The liquid and plastic limits of a soil and its water content can be used to express its relative consistency or liquidity index and they are used to evaluate the weathering characteristics of clay-shale materials. Plasticity index was calculated from the arithmetic difference between liquid and plastic limits.

Moisture content of sludge and clay samples was determined gravimetrically. Samples were dried at 110 °C until constant weight. Brunauer–Emmett–Teller (BET) specific surface areas of sludge and clay samples were determined from the 77 K N_2 adsorption–desorption isotherms in a Quantachrome Autosorb Automated Gas Sorption System (Florida, USA). The cation exchange capacities (CEC) of clay and sludge samples were determined by conductimetric measurements at 665 nm with methylene blue.

Microstructures of the samples were examined by scanning electron microscopy (SEM) in a Shimadzu SS 550 microscope (Tokyo). Thermal behaviors of samples were examined by Thermal Gravimetric Analysis (TGA) curves using a Shimadzu equipment (model TA-50, Tokyo) working at N_2 atmosphere with a flux of 20 mL/min until 1000 °C. Differential scanning calorimeter (DSC) analysis were carried out in a Shimadzu equipment (model DSC-50, Tokyo), at a heating rate of 10 °C/min, and with N_2 flowing at 20 mL/min.

Sludge and clay samples were analyzed according to the presence of some metals frequently found in textile wastewaters and/or are limited by Brazilian standards, which are: Al, Cd, Pb, Cu, Cr, Fe, Mn, Hg, Na, Zn, Ba, Ca, Mg, and K. Samples were submitted to nitroperchloric digestion and metal contents were analyzed by atomic absorption in a SpectrAA-10 Plus spectrophotometer (Varian, Australia).

Leaching and solubilization tests were carried out in sludge and clay samples according to the Brazilian standards NBR 10005/04 [11] and NBR 10006/04 [12], respectively. Leaching procedure uses deionized water at a rate of 16:1. The sample with water is agitated for 24 h. Solid and liquid phases are separated by filtration. According to this procedure a waste is classified on a scale ranging between dangerous and non-dangerous. The solubilization test is used to classify a waste on a scale between non-inert and inert. The procedure uses deionized water without any pH correction and the liquid/solid ratio is 4:1. After mixing for 5 min the materials are left in the bottle for 7 days. After this period, the solid and the liquid constituents are separated by filtration. The obtained liquid extracts in leaching and solubilization tests were analyzed in this work to metal contents by atomic absorption in a SpectrAA-10 Plus spectrophotometer (Varian, Australia) for the same metals mentioned above.

2.3. Ceramic body production

Ceramic bodies for civil constructions, as shown in Fig. 1, were fabricated at laboratorial scale (73 mm × 35 mm × 55 mm) using a Gelenski machine (model MVIG-05

Fig. 1. Ceramic brick produced in this work.

647) in order to evaluate different quantities of sludge (0 (control), 5, 10, 15, and 20% in mass basis) that may be incorporated into the clay sample. For each assay, 15 kg of material (clay + sludge) were manually mixed with a quantity of water necessary to obtain a homogenous pulp.

In order to analyze the sludge incorporation at real scale, ceramic bricks were also produced in a local pottery (Maringá-PR, Brazil) as a cube with 20 cm of length, 10 cm of width, and 15 cm of height. The fabrication employed industrial machines available in this pottery at batches of 1000 kg with and without sludge incorporation.

All bricks were fabricated by the extrusion process. After that, the bricks were initially dried at 100 °C during 24 h. The temperature of the industrial oven was then increased from 100 to 900 °C during 3 days with increases at each 24 h.

Mechanical properties of the bricks were measured after the sintering process. The flexural rupture strength of the sintered samples was measured according to NBR 15270-3/05 (Appendix C) [13] with a universal testing machine (Brazil). Water absorption was measured by a water displacement method following the Brazilian standard NBR 15270-3/05 (Appendix B) [14].

Leaching and solubilization tests and scanning electron microscopy (SEM) were also carried out in the ceramic bodies as described for sludge and clay samples.

3. Results and discussion

3.1. Characterization of sludge and clay samples

Particle size distribution of the used clay was evaluated in order to verify the material quality. According to the results presented in Fig. 2 almost 50% of the particles are smaller than 0.002 mm, corresponding to the phase rich in clay minerals. The proportion of the clay fraction in the ceramic raw material is indicative of plasticity and workability [15]. About 27% of the material corresponds to the silt fraction (0.002 mm $\leq x \leq$ 0.06 mm), while the sand fraction (0.06 mm $\leq x \leq$ 0.6 mm) is about 24% in this sample. The obtained results show that this material is reliable for ceramic blocks fabrication. Coroado et al. [16] observed similar results for clay samples used to ceramic brick fabrication.

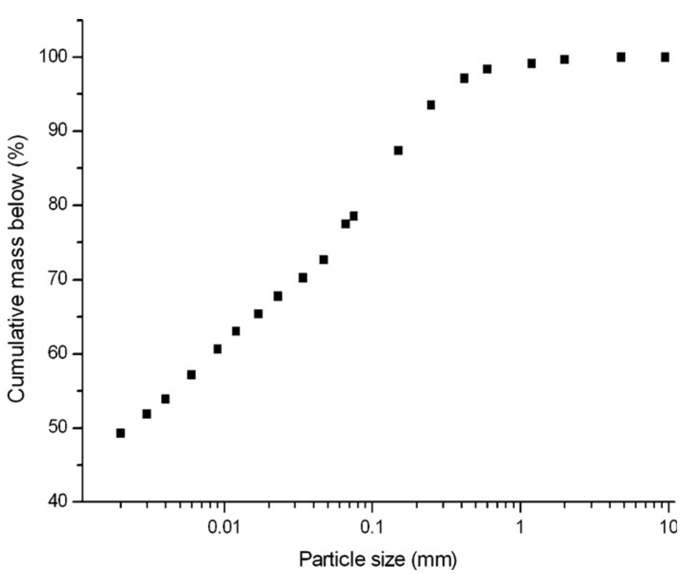

Fig. 2. Particle size distribution of the clay sample.

Table 1
Physical chemical properties of clay and sludge samples.

Parameter	Clay	Sludge
BET surface area (m^2/g)	47	36
Moisture (mass %)	3.2	15.0
CEC (mol/dm^3)	25.2	6.8

The obtained results of the Atterberg limits indicated that the clay sample has plastic and liquid limits equal to 20 and 50% (mass basis), respectively. Consequently, the plasticity index is equal to 30% (mass basis). These results show that the clay sample used in this work has optimal properties for pottery industries according to its workability properties [17]. The obtained results for clay characterization are quite similar to those published by Pinheiro and Holanda [18].

Table 1 presents physical chemical properties of clay and sludge samples regarding to BET surface area, moisture, and cation exchange capacity (CEC).

The isotherms obtained for clay and sludge samples are classified as type IV by the IUPAC classification. This means that both materials (clay and sludge) contain mainly mesopores. The clay sample presents higher CEC value than the sludge sample, which could be expected since clay is a well known adsorbent material. The sludge sample presented higher water content.

Fig. 3 presents SEM images for clay and sludge samples. Fig. 3(a) shows that the clay sample is a granular material. According to Fig. 3(b), there are materials of different shapes and sizes, as well as fibrous ones, in the textile sludge sample.

The thermal behavior of the raw materials (clay and sludge) was examined by TGA and DCS curves (Figs 4 and 5). Fig. 4a shows that there are two main peaks in the TGA curve of the clay sample. Similar result was presented by Özkan et al. [19] and Xu et al. [20]. The first smaller peak, at temperatures around 100 °C, is probably associated with adsorbed water. The second one, at higher temperatures, is associated with the dehydration of clay mineral, as also reported by Coroado et al. [16]. According to Fig. 4a, the weight loss was equal to 7.4% until 671 °C in the clay sample. Fig. 4b shows the DSC curve of the clay sample. Two main endothermic peaks are observed which are associated with adsorbed water and clay dehydration [16].

According to Fig. 5a there are three main thermal degradations for the sludge sample. The first one occurs until 223 °C and it probably corresponds to the loss of physically adsorbed water. Another significant thermal degradation can be observed until 304 °C with a weight loss of 75% that can be related to the dehydration of hydroxides [16]. This behavior is associated to the high water content in the sludge sample. A smaller smooth loss near 600 °C is observed in this curve (Fig. 5a) and can be attributed to volatile constituents. Fig. 5b

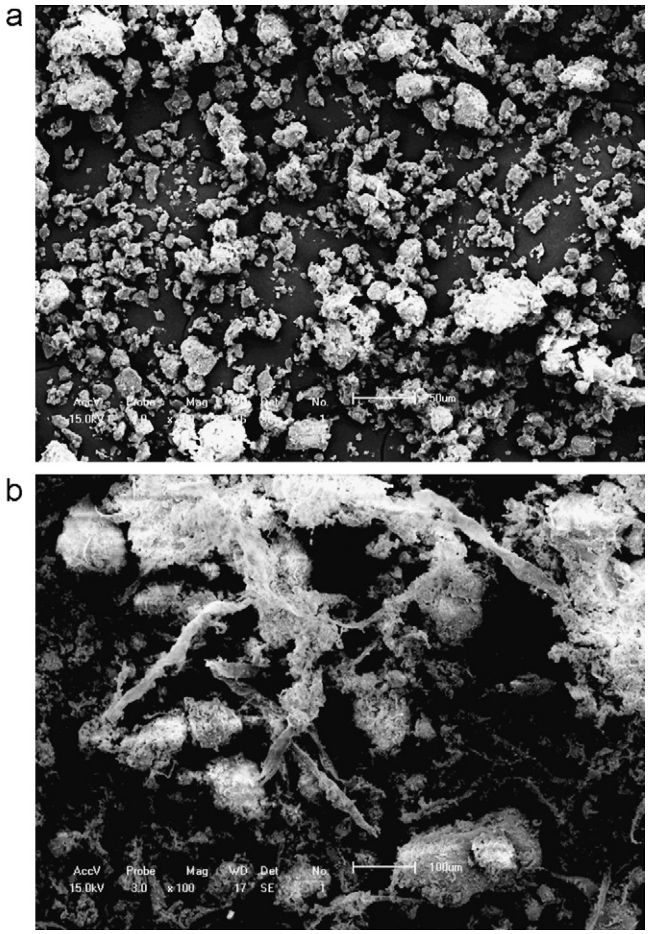

Fig. 3. Scanning electron microscope images of clay (a) and sludge (b) samples.

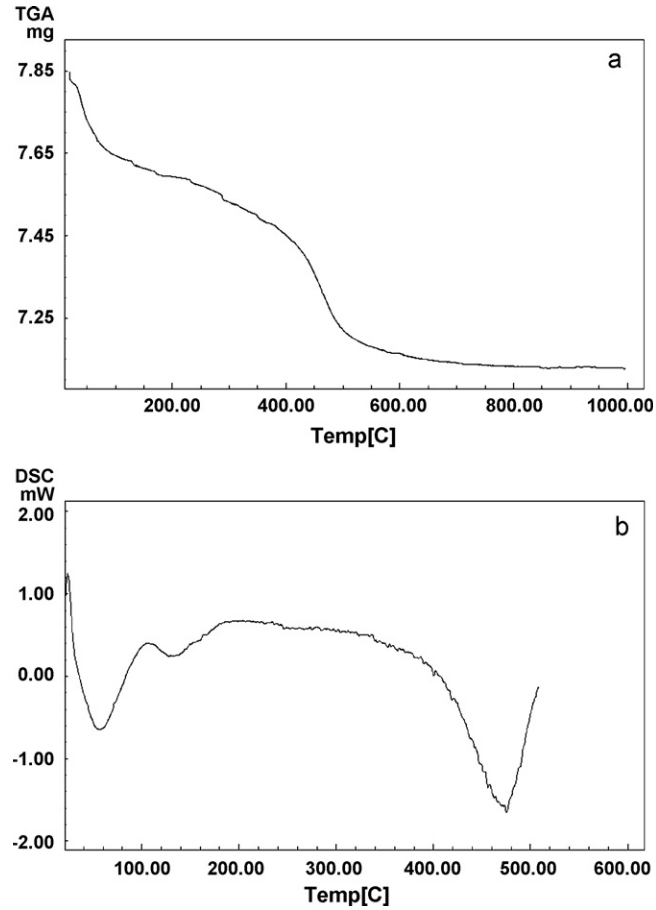

Fig. 4. TGA (a) and DSC (b) analyses of clay sample.

shows the DSC curve for the sludge sample where a pronounced endothermic peak around 100 °C is observed. This peak is related to water loss.

Table 2 presents the quantity of metals identified by the atomic absorption analyses of clay and sludge samples.

The high content of metals like aluminum, iron and calcium is an indicative of the oxide composition of base-clay materials. This clay sample is referred as red-firing clay due to its high iron content [19]. The clay sample also presents significant quantities of K and Na (alkalines), Mg, Ca, and Ba (alkaline-earth), and traces of Cu, Zn, Cr, Mn, and Co. In general, clays are associated with these metals forming quartz, pyrite, mica, calcite, dolomite, among others compounds.

The analyzed sludge also presented high quantities of aluminum and iron. These elements are constituents of the products used during the process of water treatment, as well as Na, Mn, Ca and Mg. Quantities of Ba, Zn, Cr, Cu, and Ni are probably derived from the dyeing process applied in the textile industry. Jørgensen et al. [21] also analyzed the composition of a laundry sludge and detected high content of heavy metals, like Cu and Zn. These characteristics make impossible the direct disposal of this sludge on the soil, or even the use of the sludge in agriculture, either directly or after composting.

Table 3 presents the metal content analyzed in the extract after leaching and solubilization tests. This table also shows the limits for each metal established by Brazilian standards [2].

Results presented in Table 3 show that the amount of metals present in the extract after leaching test does not exceed the Brazilian standard [2]. Similar results were reported by Basegio et al. [22] for raw clay samples. However, the concentration of aluminum and iron in the extract after the solubilization test exceed the limit established by the Brazilian standard [2], as shown in Table 3. This means that the clay and the sludge are not inert materials. Clay materials commonly have high contents of aluminum and iron due to their mineralogical characteristics. The presence of aluminum and iron in the solubilized extract of sludge is probably due to the application of aluminum and iron sulfates during the wastewater treatment.

3.2. Ceramic bricks at laboratorial scale

Fig. 6 presents flexural rupture strengths measured for ceramic bricks obtained without sludge (control) and with 10, 12, 15, 16, 20, and 24% (mass basis) of sludge. These values are the average of 10 measurements. An analysis of variance (ANOVA) of these data showed that there is significant difference between the produced bricks ($p = 0.0309$) considering a confidence limit of 95%.

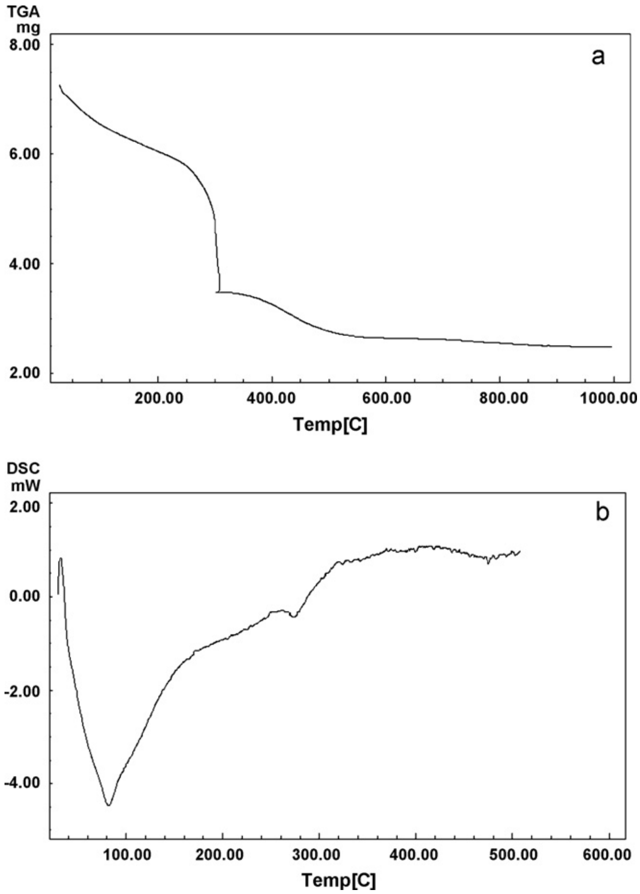

Fig. 5. TGA (a) and DSC (b) analyses of sludge sample.

The obtained results show that the flexural rupture strength decreased as the sludge concentration increased. This decrease is more pronounced in the sample with 24 wt% of sludge. Anyway, all the obtained flexural strengths are according to the Brazilian standard for ceramic bricks, which is equal to 1.5 MPa [13]. Montero et al. [17] also observed a flexural rupture strength decrease in ceramic tiles incorporated with sewage and marble sludges.

Table 2
Results of atomic absorption analysis of clay and sludge samples.

Metal	Clay (g/kg)	Sludge (g/kg)
Al	1.986	15.886
Ba	9.50	5.40
Ca	5.724	9.809
Cd	[a]	[a]
Co	0.001	[a]
Cr	0.015	0.002
Cu	0.033	0.049
Fe	13.51	15.98
Hg	[a]	[a]
K	2.594	1.187
Mg	1.150	2.078
Mn	0.009	0.038
Na	0.040	0.300
Ni	[a]	0.01
Pb	[a]	[a]
Zn	0.012	0.07

[a] Not detected.

Fig. 7 presents the values measured for water absorption in the samples with and without sludge incorporation. These values are the average of 10 measurements. An analysis of variance (ANOVA) of these data showed that there is significant difference between the produced bricks considering a confidence limit of 95%.

Results presented in Fig. 7 show that water absorption increases as the sludge percent is increased. This behavior is associated with the characteristics of the incorporated material and also with the employed firing temperature. Basegio et al. [22] showed that water absorption of clay products decreases with the increase in the firing temperature. Pinheiro and Holanda [18] noticed that the water absorption decreases with the encapsulated petroleum waste addition into clayey formulations. On the other hand, Shih et al. [23] indicated that the water absorption of the bricks increased when the slag content increased.

Although the water absorption have increased according to the sludge incorporation, excepted for the brick sample with 24 wt% of sludge, the obtained values are in accordance with the limit established by the Brazilian standard [14] (from 8 to 22 wt%).

Fig. 8 presents the SEM images of the ceramic bricks built up with sludge incorporation ranging from 0 to 24%. All samples are rough and porous, as expected for clay ceramic fired at temperatures around 1000 °C [5]. Results presented in Fig. 8 show that, in general, the surface smoothness increases as the concentration of sludge is increased, and different king of materials can be observed onto the surface.

Table 4 presents metal concentrations detected in the extract obtained after the leaching test of the ceramic bricks produced at laboratorial scale with different quantities of textile sludge. All the proposed metals were analyzed, but this table presents only the detected ones.

These results show that the metals Ba and Al, which were detected in the lixiviated extract of raw clay and sludge (Table 3), were detected in small concentration, indicating that these metals were retained in the ceramic bricks or they were eliminated during the sintering process. The concentration of Ba diminishes as the quantity of sludge is increased since its concentration is higher in the clay than in the sludge. Concentrations of Cr and Cu increase as the quantity of sludge are increased. Concentrations of Ba, Cr, Cd, and Pb are in accordance with the limits established by the Brazilian standard [2].

Although the ceramic bricks had been classified as non dangerous according to the lixiviation test, it is still necessary to carry out the solubilization test to verify if materials present in the ceramic bricks are inert. Table 5 presents metal concentrations detected in the extract obtained after the solubilization test of the ceramic bricks.

These results show that the metals Fe and Al, which were detected in the solubilized extract of raw clay and sludge (Table 3) at higher concentrations than the concentration established by the Brazilian standard [2], were detected at small concentrations in the solubilized extract of ceramic bricks built with sludge, indicating that these metals were retained in

Table 3
Comparison of metal concentrations measured in the extract after leaching and solubilization tests of clay and sludge samples with the limits established by the NBR 10004 [2].

Metal	Leached extract			Solubilized extract		
	Clay (mg/L)	Sludge (mg/L)	Limit (NBR 10004 [2])	Clay (mg/L)	Sludge (mg/L)	Limit (NBR 10004 [2])
Al	0.35	7.69	[b]	1.0	1.5	0.2
Ba	5.0	3.0	70.0	[a]	[a]	0.7
Ca	[a]	[a]	[b]	[a]	[a]	[b]
Cd	[a]	[a]	0.5	[a]	[a]	0.005
Cr	[a]	0.03	5.0	[a]	[a]	0.05
Cu	0.05	0.01	[b]	[a]	[a]	2.0
Fe	0.6	0.46	[b]	0.5	0.4	0.3
Hg	[a]	[a]	0.1	[a]	[a]	0.001
K	0.01	0.01	[b]	[a]	[a]	[b]
Mg	0.04	0.04	[b]	[a]	[a]	0.1
Mn	0.06	[a]	[b]	[a]	[a]	0.1
Na	0.7	0.06	[b]	[a]	[a]	200.0
Ni	[a]	[a]	[b]	[a]	[a]	[b]
Pb	[a]	0.05	1.0	[a]	[a]	0.01
Zn	[a]	[a]	[b]	[a]	[a]	5.0

[a] Not detected.
[b] Not limited by this normative.

the ceramic bricks or they were eliminated during the sintering process. The concentration of Al increases as the quantity of sludge is increased in the ceramic bricks. However, the observed values do not extrapolate the limit established the Brazilian standard [2]. Similar results were observed by Basegio et al. [22] for leaching and solubilization tests with ceramic bodies incorporated with tannery sludge. In this way, the ceramic bricks built up with sludge of textile wastewater are classified as non inert.

3.3. Ceramic bricks in real scale

The results obtained at laboratorial scale showed that textile sludge can be incorporated into ceramic bricks at 20% related to the total mass. This limit was established mainly due to the water absorption and the flexural rupture strength properties obtained for the produced ceramic bricks at laboratorial scale. In this way, ceramic bricks were produced in real scale without sludge incorporation (control) and with 20% of sludge (mass basis).

Flexural rupture strengths were measured for 10 samples of each ceramic brick (with and without sludge). The obtained averages were 4.62 and 3.73 MPa for the ceramic brick with and without sludge, respectively. According to the variance analysis (ANOVA) these results are different ($p = 0.0275$) considering a confidence limit of 95%. Sludge incorporation decreased the obtained flexural rupture strength, but it is still in accordance with the limit established by the Brazilian standard (1.5 MPa) [13].

Water absorptions for the ceramic bricks with and without sludge were equal to 10.10 and 15.73% (average of 10

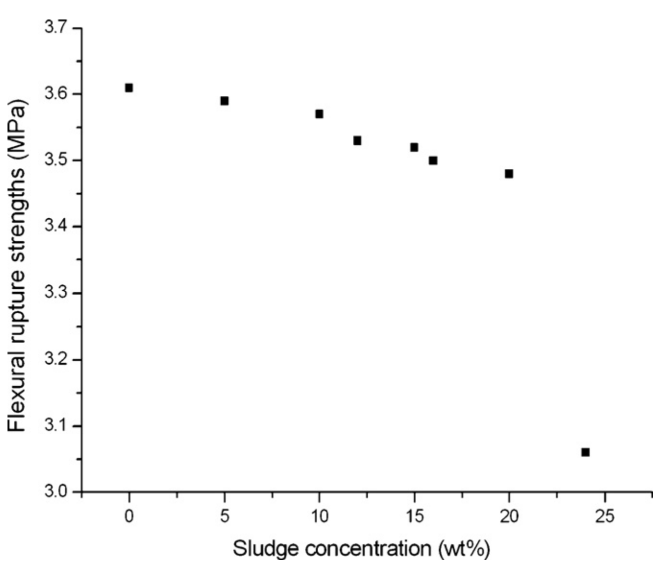

Fig. 6. Average values of flexural rupture strengths of ceramic bricks incorporated with sludge.

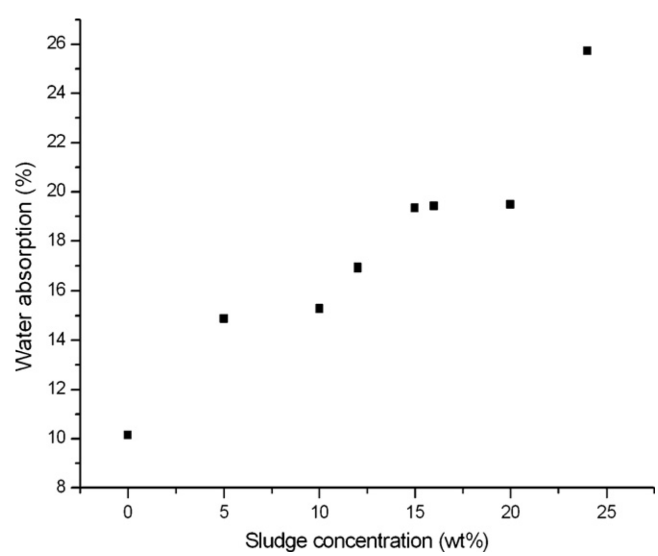

Fig. 7. Average values of water absorption of ceramic bricks incorporated with sludge.

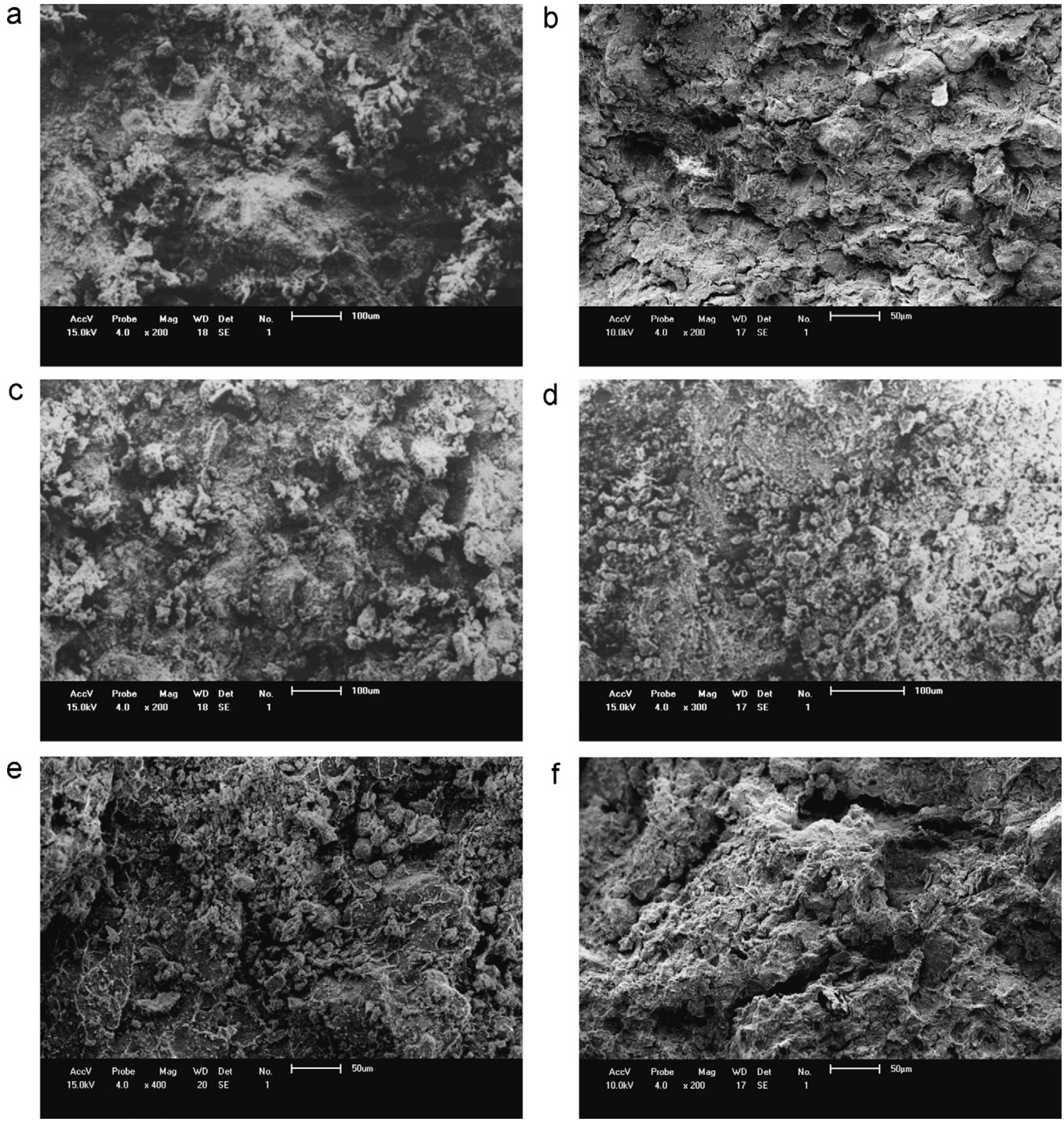

Fig. 8. Scanning photomicrographs of ceramic bricks fabricated at laboratorial scale with different sludge concentration (0% (a), 5% (b), 12% (c), 16% (d), 20% (e), and 24% (f)).

measurements). According to the variance analysis (ANOVA) these results are different considering a confidence limit of 95%. Although the water absorption has increased for the ceramic bricks with 20% of sludge, this value is still in accordance with the limit established by the Brazilian standard (from 8 to 22%) [13].

The ceramic bricks produced at real scale presented higher flexural rupture strength and smaller water absorption values than those produced at laboratorial scale with the same sludge quantity, probably due to better homogenization and compaction of the samples during the mixing in the industrial scale.

Table 6 presents metal concentrations measured in the extract obtained after the leaching test of the ceramic bricks produced in real scale.

The ceramic brick fabricated with 20 wt% of textile sludge is classified as non dangerous for the environment according to

Table 4
Metal concentrations (mg/L) measured in the leached extract of ceramic bricks fabricated at laboratorial scale.

Metal	Sludge concentration (wt%)							
	0	5	10	12	15	16	20	24
Al	0.2	0.5	0.6	0.5	0.7	0.5	0.4	0.3
Ba	5.8	5.5	5.3	2.6	5.1	5.2	2.6	1.6
Cr	[a]	[a]	[a]	[a]	[a]	[a]	0.02	0.01
Cu	[a]	[a]	[a]	0.01	0.02	0.01	0.02	0.02
Fe	0.4	0.6	0.7	0.5	0.9	0.6	0.5	0.6
K	[a]	0.01	0.04	0.01	0.02	0.01	[a]	[a]
Mg	0.02	0.02	0.03	0.02	0.02	0.03	0.03	0.04
Na	0.03	0.04	0.08	0.04	0.07	0.04	0.01	0.01

[a] Not detected.

Table 5
Metal concentrations (mg/L) measured in the solubilized extract of ceramic bricks fabricated at laboratorial scale.

Metal	Sludge concentration (wt%)							
	0	5	10	12	15	16	20	24
Al	[a]	[a]	[a]	[a]	0.02	0.02	0.05	0.08
Cu	0.01	[a]	[a]	[a]	[a]	[a]	[a]	[a]

[a] Not detected.

the Brazilian standard [2]. A better compaction of the materials is carried out in the fabrication of the bricks at real scale, warranting that metals like Cr, Cu Mg, and K were no more detected in the leached (these metals were detected in the leached extract of samples fabricated at laboratorial scale, as shown in Table 5).

Only aluminum at a concentration of 0.01 mg/L (below the limit established by the Brazilian standard [2]) was detected in the extract obtained after the solubilization test of the ceramic bricks produced at real scale with and without sludge addition. The higher compaction employed in the real scale ensured the immobilization of Cu, which was detected in the solubilized extract of the bricks fabricated at laboratorial scale (Table 6).

Fig. 9 presents the SEM images of the ceramic bricks fabricated with and without sludge incorporation. These figures show a pore size increase for the sample fabricated with sludge, confirming the results obtained for resistance and water absorption.

Table 6
Metal concentrations (mg/L) measured in the leached extract of ceramic bricks fabricated at real scale.

Metal	Sludge concentration (wt%)	
	0	20
Al	0.1	0.4
Ba	4.5	4.0
Fe	0.4	0.5
Na	[a]	0.03

[a] Not detected.

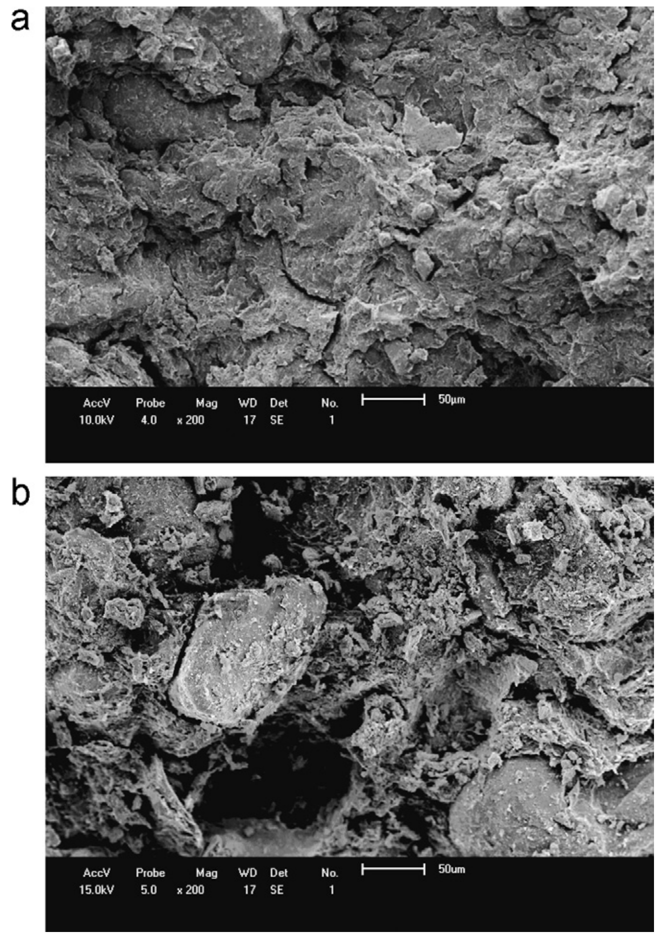

Fig. 9. Scanning photomicrographs of ceramic bricks fabricated at real scale without (a) and with (b) sludge incorporation.

4. Conclusion

The obtained results showed that an incorporation of textile wastewater sludge until a concentration of 20% (mass basis) leads to good quality bricks. In general, the mechanical properties of the obtained ceramic bricks were quite satisfactory under Brazilian legislation. Besides, leaching and solubilization tests showed that the ceramic bricks fabricated with textile sludge are inert, by means that the chemical compounds detected in the raw samples are not solubilized. The use of textile wastewater sludge in ceramic bricks is an advantage since it is converted from a hazardous residue into a secondary raw material.

Acknowledgments

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