

Incorporation of industrial wastes as raw materials in brick's formulation



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ABSTRACT

This article presents a case study conducted as an experiment with the incorporation of different types of industrial waste in brick manufacturing process in laboratory scale. The main objective of this work is to incorporate large amounts of different types of waste as raw material in brick's formulation. Three types of wastes were mixed with clay: automotive industry waste sludge containing heavy metal concentrations; glass waste, from a galvanic plant, mainly consisting of glass microspheres; and wood ash, from the ceramic burning furnace. The formulation's materials were analyzed by X-ray diffraction, X-ray fluorescence and electronic microscopy. The dried samples were milled separately and then dry mixed. Water was added to the mixture in order to contribute to the compaction process. The samples were dried and then burned at temperatures similar to those used for brick firing furnace. The obtained ceramics were analyzed for their retraction and then submitted to flexural strength testing. Samples obtained value above 4 MPa were approved. Among the samples tested, the formulation that showed higher flexural strength was chosen. It was prepared sufficient sample to perform the solubilization and leaching tests. For tests, the samples were reduced to dust. The results of such analyzes did not identify the presence of elements described in the initial samples' formulation. Morphological analysis was performed using scanning electron microscopy. Tested sample showed glassy characteristic of material that has been sintered during the firing process. This effect is also a proof that the waste identified in initial sample's formulation were inerted. Obtained results characterizes that the tested formulation can be considered as an alternative for bricks manufacturing with incorporation of industrial waste and an activity non-hazardous to the environment.

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1. Introduction

The quest for continuous improvement has provided the industry in general, the development of its production processes in condition to make them ever more robust, but with lower investments. This allows to say that these processes are increasingly controlled and have higher incomes, if evaluated primarily from a financial perspective, development focused on reducing costs (Wiemes, 2013). The objective of solid waste recycling is to reduce raw material consumption, thus minimizing pollution problems and treatment costs (Mymrin et al., 2016).

The application of ISO 9000/14000 also greatly contributed to the development of industrial processes, favoring the continued increase in production. Evaluating this scenario in a broader perspective, there is an increase in consumption globally, driven by supply products that provide comfort, agility and speed, among other characteristics, to meet increasing demands from costumers. Although contradictory, considering the facts mentioned above, the situation is critical and many industries are contrary to what can be named sustainable performance or environmentally friendly company. The waste generation resulting from manufacturing processes is quite considerable and the destination is usually applied the provision in landfills or co-processing in cement ovens.

In a strategic context and considering an approach where environmental problems are treated as such, Barbieri (2007) states that a company should take advantage of market opportunities and neutralize threats arising from environmental issues. The

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development of cleaner processes has advantages, among which may be mentioned: reduction in waste generation, lower impact on the environment, improving company's image in supervisory bodies and reducing of costs manufactured helps organizations to develop preventive actions.

Fired clay bricks are construction materials, which have been used since ancient times and currently display different states of deterioration in numerous historic buildings (Cultrone et al., 2005). Nowadays, bricks are still being used for the same purpose (Karaman et al., 2006). Reducing waste is not the only reason to investigate the addition of certain residues into a clay matrix, although traditionally it has been the main purpose of research on this topic (Velasco et al., 2014). The current trend in bricks manufacturing has major emphasis on the use of post-consumer wastes and industrial by-products in the production process (Shakir and Mohammed, 2013). According to Reinosa et al. (2010), the building industry is the most suitable technological activity sector to consume solid wastes. Clay can also be used to immobilize harmful heavy metals ions (Churchman et al., 2006; Addy et al., 2012). Furthermore, clay minerals are silicate phases that can incorporate considerable amounts of metals in their structures. Therefore, the ceramic industry is one of the best candidates to consume large amounts of industrial wastes, such as combustion ashes, granite cutting sludge and wastewater sludges (Torres et al., 2004; Reijnders, 2007; Martínez-García et al., 2012; Mymrin et al., 2014b).

Vitrification is one of the techniques that has aroused great interest by many researchers. Mymrin et al. (2014a) define glazing as a common method of burning to the traditional pottery clay base. The vitrification process simulates the natural phenomenon of the glassing from volcanic rocks (ex. Basalt). These natural glasses contain toxic materials in their structure that have shown environmental inert as the time (Silva and Mello Castanho, 2004). As mentioned by Kim et al. (2005), this technology is implemented for processing radioactive waste and studied for inerting various types of waste. Piscicella et al. (2000) show that this technique is a viable solution to the environmental impact of various industrial activities and opens opportunities to assign value to waste.

An initial composition and heat treatment conditions, as described by Erol et al. (2007), are the most important parameters affecting the kind of crystalline phases occurred in the glass-ceramic and the final properties of the materials. These same authors also emphasize that glass-ceramics having desirable properties to meet many applications can be produced from waste materials through the application of appropriate heat treatments.

This paper presents the analysis carried out with the incorporation of three types of industrial waste in large quantities, with clay in brick formulation. In addition, the important point of the study considers the application of these formulations at similar temperatures those already applied in brick-making process. The structure of the work is based on the Cleaner Production methodology consisting eliminating pollution during the production process, after the generation of waste.

2. Methods

2.1. Materials and preparation of test samples

Raw materials used in this study are automotive waste sludge (AWS), glass waste (GW) from metal cleaning before galvanic process, wood ash (WA) and mixture of red pottery clay with sand (RPC), all provided by local industrial enterprises of Paraná state, Brazil.

Ceramic samples of various compositions are presented in Table 1. They involve homogenizing a mixture of the initial

Table 1
Formulation of ceramic samples.

Sample	Composition (%)			
	AWS	GW	WA	RPC
ETE-B1	50	20	0	30
ETE-B2	40	20	0	40
ETE-B3	40	10	10	40
ETE-B4	50	10	10	30
ETE-B5	4	0	10	86
ETE-B6	6	0	10	84
Clay	0	0	0	100

components (water contents of 12–15%), compressing at 3 MPa (wet samples are rectangular, 60 × 20 × 10 mm in size), drying to constant weight at 100 °C, sintering for 6 h (temperatures of 800, 850, 900 or 1000 °C), and cooling by natural convection. Testing conditions correspond to real ranges applied at local brick plants.

2.2. Methods

Raw materials and final ceramic were characterized according to their mineralogical and chemical composition by X-ray diffraction (XRD), X-ray fluorescence (XRF) and scanning electron microscope (SEM). Mineral composition (XRD) were studied by PANalytical brand, model Empireo with X'Celerator detector copper tube; chemical compositions (XRF) on PANalytical XRF equipment brand, Axios Max model with Rhodium 4kv tube; chemical micro analyses – by method of energy dispersive spectroscopy (EDS) on Oxford (Penta FET-Precision) X-ACT; morphological structures by SEM on FEI Quanta 200LV; solubility and leaching of metals from liquid extracts – by method of atomic absorption analysis (AAA) on Perkin Elmer 4100 spectrometer; mechanical resistance – by three-point flexural strength (FS) on EMIC universal testing machine; linear shrinkage (LS) – on Mitutoyo. Fire loss (FL) - calcined for 2 h in muffle furnace at 1000 °C. Values of mechanical properties were obtained as an average of 10 samples' measurements.

Samples were fired in laboratory furnace (Linn Elektro-Therm, thermocouple Pt-Pt/Rh and ranging from 5 °C), applying temperatures between tracks 800–1000 °C allowing to simulate the same conditions identified in a furnace a pottery. The firing temperature cycling and time adopted for each test burns were programmed to operate automatically, as represented in Fig. 1:

- Initiate the process until temperature 600 °C, with heating rate of 10 °C per minute.
- Temperature of 600 °C was kept constant for 30 min.
- Heating to desired temperature (800 °C for example), with heating rate 10 °C per minute.
- Operating temperature fixing (800 °C, for example.) for 6 h (360 min).

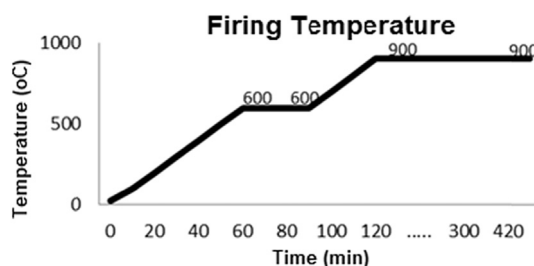


Fig. 1. Schematic representation of firing temperature applied to different samples tested.

Table 2
Compositions of raw materials.

Components	Raw material composition (%)			
	AWS	GW	WA	RPC
SiO ₂	3.1	67.8	45.3	56.9
Al ₂ O ₃	1.1	0.6	13.2	21.9
Fe ₂ O ₃	18.4	0.2	7.6	10.2
TiO ₂	1.1	—	1.2	1.6
ZnO	0.3	—	—	—
BaO	1.1	—	—	—
MgO	2.1	3.0	3.2	1.0
MnO	0.1	—	1.1	<0.1
CaO	25.9	6.9	15.0	0.2
K ₂ O	0.1	0.2	8.1	0.9
Na ₂ O	0.4	9.1	—	—
SO ₃	0.9	0.3	1.3	0.1
P ₂ O ₅	0.6	—	1.9	0.1
F.L.	44.5	12.0	1.3	7.0

F.L. – Fire Loss.

- Furnace shutdown (automatic, after burning the sample cycle) until room temperature.

After firing, samples were tested in flexural strength. It used universal testing machine EMIC - Model DL10000 (advance of 0.5 mm/min and 200 kN load cell - according to [NBR 13818, 1997](#)). The value of the analysis result on the display of the universal testing machine consists of a raw data processed and for that, considering each sample individually. The actual value of flexural strength after firing PCs was obtained by calculating flexural breaking strain (see Eq. (1)):

$$FS = (3FL)/(bh)^2 \quad (1)$$

where: FS – flexural strength (Mpa), F - load reached at the moment of rupture, L - distance between the support of cleavers (45 mm), b – sample's width (mm), h – sample's height (mm).

3. Results and discussion

3.1. Raw material's characterization

The raw material's characterization is available in [Table 2](#).

Chemical composition of AWS involves Fe₂O₃ – 18.4%, CaO – 25.9%, MgO – 2.1%, P₂O₅ – 0.6%, Al₂O₃ – 1.1%, TiO₂ – 1.1%, K₂O – 0.1% and Na₂O – 0.4%. It also includes heavy metals in very high contents: Ni – 0.1%, Zn – 0.3%, Pb – 0.23%, classified as hazardous material. Very high fire loss (44.46%) can be explained by the presence of organic components (mainly oils, paints, etc.). Fluxes' material presence is also identified in its formulation.

The main components of the GW are SiO₂ (67.8%), Al₂O₃ (0.6%) and Fe₂O₃ (0.2%) with a high content of Na₂O (9.1%), CaO (6.9%) and MgO (3.0%) which are extremely valuable as a fluxes' material for ceramic production. Fire loss (11.98%).

Chemical composition of the WA are SiO₂ (45.3%), Al₂O₃ (13.2%),

Fe₂O₃ (7.6%) and TiO₂ (1.2%) with a high content of K₂O (8.1%), CaO (15.0%) and MgO (3.2%) which are extremely valuable as fluxes material for ceramic production. Fire loss (1.28%).

The chemical components of RPC are SiO₂ (56.9%), Al₂O₃ (21.9%), Fe₂O₃ (10.2%) and TiO₂ (1.6%) with a high content of K₂O (0.9%), CaO (0.2%) and MgO (1.0%). Fire loss (6.95%). Clay is responsible to promotes plasticity to wet mass.

[Fig. 2](#) represents SEM micro image of four raw materials under study. Figure with letter A represents how AWS exhibit different particles' size and shapes, with compact and uniform characteristic. Figure with letter B shown that WG are almost perfect spheres, with different sizes and many small particles, also with irregular format, characteristic of break of them. Figure with letter C represents WA residue and two specific morphological structure observed, with characteristic shape of unburned wood shafts and regions rather fragmented. And figure with letter D is RPC as it was collected directly *in natura*, also with compact and uniform characteristic, and with small sheets or superposed sheets.

3.2. Mechanical properties of developed ceramics

Most of tested samples were above the minimum set value. Some of the compositions developed have rather high flexural strengths (from 4.07 to 8.14 MPa, [Fig. 3](#)). [Santos \(1989\)](#) states the demanded flexural strength is 10 MPa for clay tiles and 1.5 MPa for bricks.

The ceramics' resistances increase ([Fig. 3](#)) as melting temperature increase, and because of the presence of GW and WA in the formulation. The addition of WA in the formulation is that the material has good water absorption, and which can provide uniform porosity throughout the firing process. Tested samples presented good results in mechanical properties. The best results was obtained with sample ETE-B3 that used 60% of wastes in its formulation. Only ETE-B1 samples did not achieved the established value of flexural strengths. ETE-B2 samples tested at 800 and 850 °C were at the limit of specifications, as represented in [Fig. 3](#). Addition of more than 40% of AWS does not promote positive values in flexural strength.

Addition of WA promoted a very good result in flexural strength, especially in sample ETE-B3 comparing to ETE-B4. Thus was

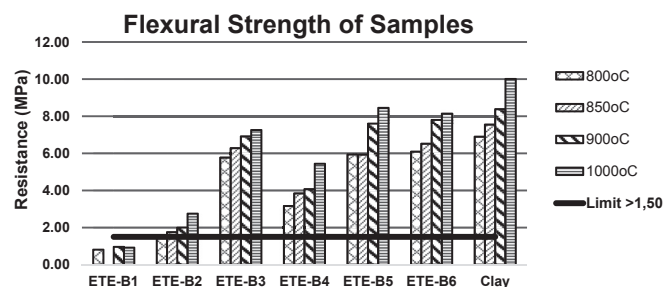


Fig. 3. Results of three points flexural strength.

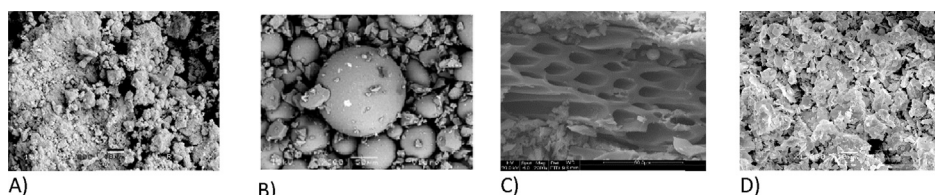


Fig. 2. SEM micro-image A) AWS, B) GW, C) WA and D) RPC.

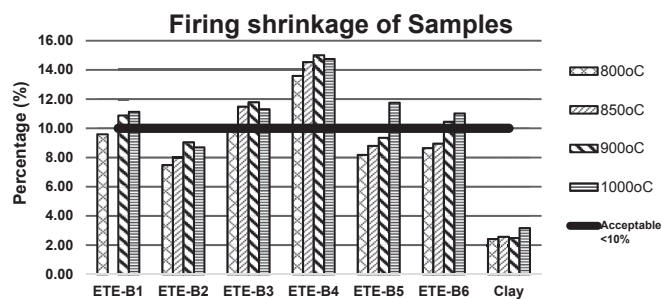


Fig. 4. Results of firing shrinkage (%).

possible by the presence of high quantity of SiO_2 , Al_2O_3 , CaO and K_2O , these last two, which act as flux materials with high temperature. In addition to the glass phase fused-bond with the clay brick bodies, the fusion of crystalline quartz in clay also played an important role in enhancing the properties of clay bricks. (Phonphuak et al., 2016). As an additive, WG, when incorporated into a mixture, could induce the vitrification in clay bricks, resulting in higher density, less water absorption, and lower drying shrinkage (Loryuenyong et al., 2009). WA also acts as a flux owing to Na_2O content and its noncrystalline composition, thus lowering the temperature required for sintering bricks. In addition, the

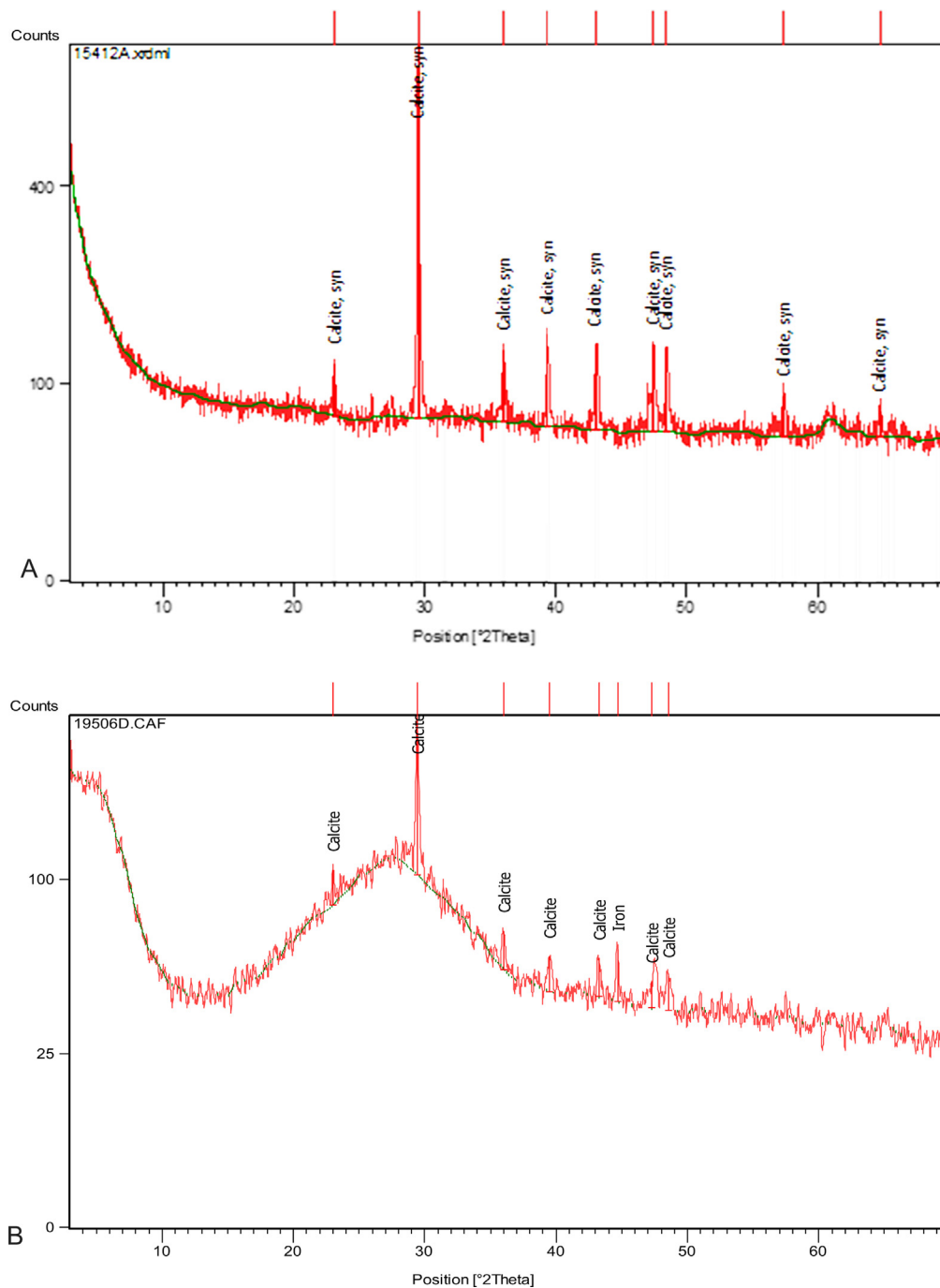


Fig. 5. Diffractogram patterns of raw materials used: A) AWS, B) GW; C) WA and D) RPC.

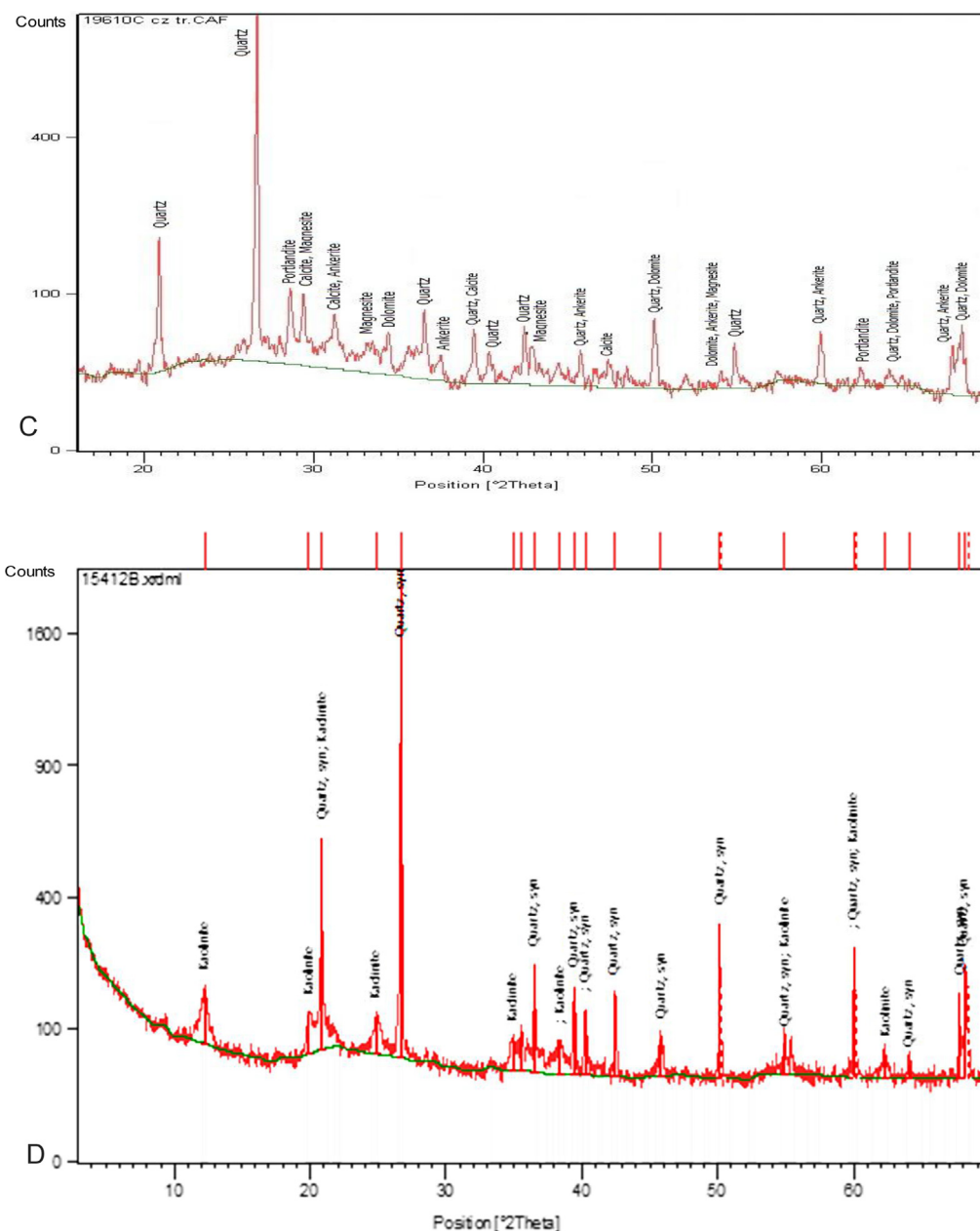


Fig. 5. (Continued).

increased glassy phase in the finished brick has the potential for improvement in both structural and durability properties, while reducing manufacturing costs (Chidiac and Federico, 2007).

It is important to note that increasing amount of AWS in the formulation of ceramic material, greater will be the amount of CaO present in the residue (due to its origin - treatment plant of industrial waste) but mainly because FL of this raw material is very high, which harms the reaction kinetics. So that, the reduction of the amount of AWS percentage (from 50 to 40%) in the formulation, improvement occurs in its resistance to bending.

Sample identified as clay were prepared with no addition of waste and did better results in flexural strengths.

There is an increased variation in the firing shrinkage of the test samples with increasing temperature, with the same composition

range of the mixture, as can be seen in Fig. 4. This phenomenon occurs because the glass tends to bind the particles with the other components of the formulation, subsequently providing the effect of glazing material obtained.

Firing shrinkage is related to the loss of water between clay particles resulting in the closer packing of clay particles and resulted shrinkage. To minimize shrinkage, firing temperature which is an important parameter affecting the degree of shrinkage must be controlled during the firing process (Karaman et al., 2006). Normally, a good quality of brick exhibits a shrinkage below 8% (Okunade, 2008). In this study, fired clay bricks were fired at temperatures of 800, 850, 900 and 1000° C. The firing shrinkage increased with increasing firing temperature and decreased with increasing amounts of waste glass.

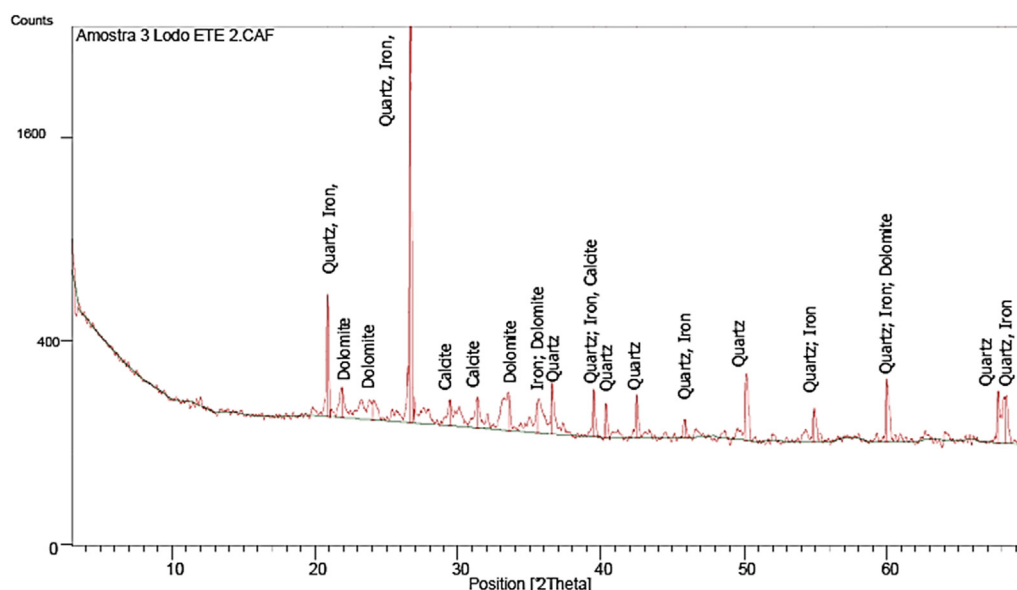


Fig. 6. XRD analysis of obtained ceramic (ETE-B3) sample.

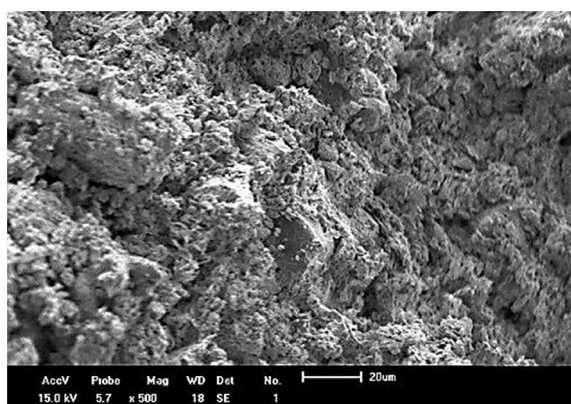


Fig. 7. SEM micro image of obtained ceramic (ETE-B3) sample.

Table 3
Solubility analysis of ETE-B3 sample.

	Sample ETE-B3 (mg/L)	ABNT NBR 10.004 (2004) attached G (mg/L)
Al	NC	0.2
Ba	<0.01	0.7
Cd	<0.001	0.005
Pb	<0.01	0.01
Cu	NC	2.00
Cr	<0.01	0.05
Fe	NC	0.3
Mn	NC	0.1
Zn	0.01	5.00

Table 4
Leaching analysis of ETE-B3 sample.

	Sample ETE-B3 (mg/L)	ABNT NBR 10.004 (2004) attached F (mg/L)
Ba	2	70
Cd	<0.001	0.5
Pb	<0.01	1
Cr	<0.01	5
Hg	NC	0.1
Se	NC	1

3.3. Physicochemical processes of ceramics structural formation

The best mechanical properties are exhibited by composition ETE-B3 (Table 1) with flexural strength reaching 5.77 MPa after sintering at 800 °C. Therefore, composition ETE-B3 was chosen for the XRD, SEM and EDS analyses of the processes involved in the ceramics' structural formation. In addition, the choice of 800 °C temperature is related to lower generation of environmental impact (due to its lower energy consumption) and the most critical condition in terms of sample burning. The optimum firing temperature was the lowest temperature used to produce bricks with required properties (Phonphuak et al., 2016). Fig. 5 presents the XRD analysis of samples used in the formulations tested.

According to XRD analysis (Fig. 6), the process of sintering causes synthesis of new minerals that are absent in the initial mixtures.

Fig. 6, shows the diffraction pattern of XRD analysis of the ETE-B3 Ceramics, which was the best result of resistance to bending, obtained from the tested samples, and morphological analysis by scanning electron microscope.

The results of SEM micro image for the ETE-B3 material are presented in Fig. 7.

Sample ETE B3 displayed in Figs. 5 and 6, presented a homogeneous appearance without the presence of phases, which characterizes melting and complete interaction of mixed components, as states Kim et al. (2005). A glazed structure justifies the presence of the amorphous substance obtained in the XRD test and explains the good results obtained with flexural strength in respective sample. The structure observed was glassy and uniform appearance. According to Borlini et al. (2005), presence of alkali and alkaline earth oxides along with clay, contributes to porosity reduction by the formation of liquid phase in ceramic matrix. Considering these aspects, is possible to say that a new material with a condensed and homogeneous mass formation, characteristic of the merger of the materials that composed the proof bodies was obtained. The presence of porosity in samples is due to the fact that gas formation during the burning of organic material present in the sample, and that has its origin in both clay as sludge from the ETE industry, as well as the presence of traces of oils and greases present in its residue.

Table 5
Cost-benefit analysis – comparison with two process (brief descriptions).

Evaluation items	Actual flow (100% clay)	Proposal flow (60% wastes +40% clay)
Raw Material Extractions	Machines, Labor, Trucks	Reduce use of Machines, Labor, Trucks, and use of wastes, coming from industry.
Transport Impact	Known	Must set transportation route and validate with environmental agency
Process Validation	Ok – Process validated	Necessary process validation
Final Product	Ok - Conform	Conform, but necessary confirmation of bricks' characteristics
Environmental Agency	OK – Process and Product conform	Not Ok, process and product must be validated
Storage Conditions	OK – not necessary specific conditions	Necessary to build a suitable site for waste storage
Labor Safety/Work Conditions	Process OK	Necessary use of specific gloves to avoid contact with wastes
Atmospheric Emissions	Known	Unknown – necessary to realize measures to identify environmental impact
Waste Destination (from industry)	Coprocessing in cement kilns	Use only in potteries, according to this study
Waste Destination Costs	Necessary to pay	Possibility to reduce destination costs

The results of leaching and solubilization showed in [Tables 3 and 4](#), presents values below to those specified by the Brazilian standard, and thus is proven that the materials manufactured with the formulation of the ETE-B3 sample, present conditions to be applied in the production process of a pottery that meets the minimum requirements necessary to ensure the length of time the temperature of 800 °C.

Samples ETE-B1 and ETE-B2 did not show values that could be characterized as significant before the specification (minimum 1.5 MPa). Therefore, these formulations cannot be applied to the manufacture of ceramic materials (bricks) at the tested temperatures. The ETE-B1 sample, tested at 800 °C was damaged (molten) that's why flexural strength measurement was not performed. In this same graph it was also presented the material containing only clay (called as clay). For this sample, the obtained values were significant with regard to resistance to bending. In the tests, it was shown that all formulations tested exhibited resistance to bending than the lowest value observed in the sample preparation only clay, although satisfying the minimum requirement of 1.5 MPa. Due to the use of crushed glass, the samples described in [Table 1](#) show relevant values for resistance to bending. For ETE-B3 samples, ETE-B4, ETE-B5 and ETE-B6 with added ground glass sphere, provide relative increase in the flexural strength of the bodies evaluated evidence. ETE-B4 sample showed high amount of residues (70%) and this favors the formation of a more brittle material after firing in oven. That is the evidence that values obtained, especially for

burned samples between 800 °C and 900 °C showed intermediate values.

ETE-B3 and ETE-B4 samples were formulated with amounts of sludge, ranged from 40 to 50%. In addition, it was added 10% ash and 10% of ground glass sphere (which favors vitrification process). Important to note that during rehearsals, proof bodies were prepared (Samples ETE-B5 and ETE-B6) with similar formulation applied only to the residues of the Industry A. It was found that ETE-B5 sample showed good results considering the proposed wording, reaching to 5.93 MPa at 800 °C.

For microstructure analysis, it is possible to identify whether the obtained material showed uniform visual appearance, without concentration or regions of non-vitrified materials or even non-agglomerated sintered ceramic materials. From the definition of the sample that showed the best characteristic flexural strength, solubilizing assays were performed ([NBR 10006, 2004](#)) and leaching ([NBR 10005, 2004](#)) to verify the chemical elements present in the sample analyzed after firing oven.

The cost-benefit analysis can be considered from the point of view of the proportions of each residue in the applied formulation brick tested in the laboratory compared to brick made of a pottery. [Table 5](#) provides a brief description of the main points that can be compared in qualitative aspect.

[Fig. 8](#) shows a synthetic way the main steps of the manufacturing process of a ceramic formulation. In this figure are shown two productive flows, considering the current process flow

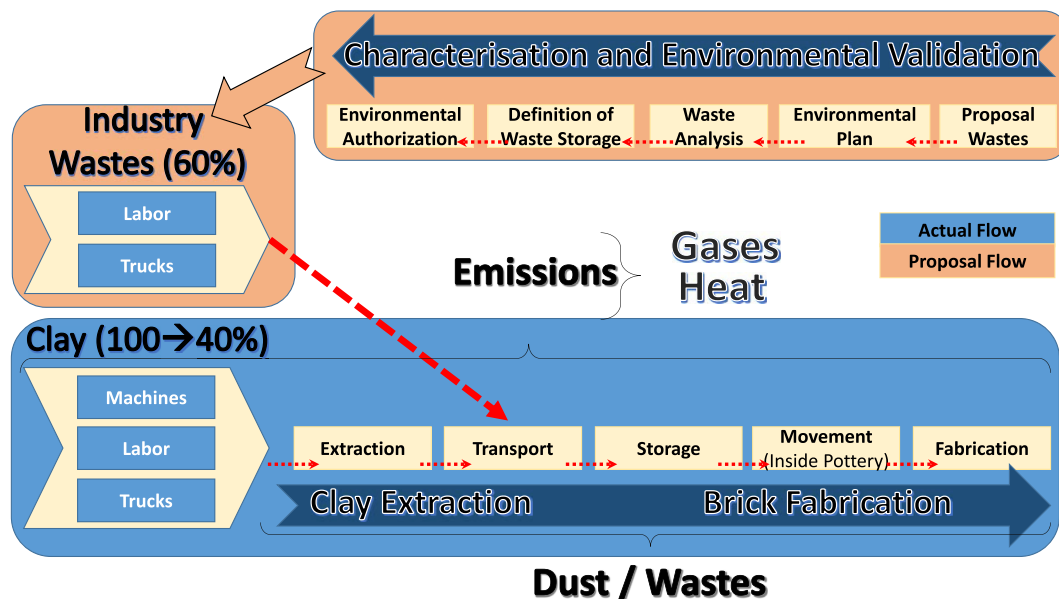


Fig. 8. Schema of brick's fabrication – Considering Actual and Proposal Flow.

and the other considering a proposed flow (which is considered the use of industrial waste by up to 60%). However, the cost benefit balance is too far away to identify whether the process is advantageous or not.

4. Discussion and conclusion

The main objective of this paper was to evaluate the possibility of use wastes containing heavy metals from an automotive industry and incorporate them in a formulation of a ceramic mass to develop new ceramic composition using wastes as raw material. Several formulations were used and many tests and samples analyzes were conducted to identify the best configuration to obtain highest percentages of waste in brick.

There are no practical results, because the whole study was conducted only in laboratory. The most important result expected was about the conditions that samples were tested and about the results it was obtained.

According to Cleaner Production theory, what the authors intended to do was to exchange materials in the bricks' formulation to identify the opportunity to incorporate wastes as raw materials in the composition of a new ceramic. In the concept of cleaner production, the work was directed at waste minimization line with a change in the process, making replacement materials, in this case the clay by industrial waste. The results obtained and described above showed that a significant amount of incorporation wastes from an automobile industry, more addition of GW and WA is considered technically feasible to obtain a red ceramic (brick) in firing temperature to 800 °C for 6 h.

The addition of WA in the new formulation was because the material has good water absorption, and which can provide uniform porosity throughout the burning process. Furthermore, the presence of elements such as K₂O and MgO in the material configuration may characterize them as flux materials.

In view of the various authors investigated, it is unanimous the condition of use flux materials (like K₂O, MgO and Na₂O) with wastes and clay materials in order to promote the manufacture of other materials (red manufacturing ceramic) to achieve the inertization process of materials containing toxic wastes. In addition, Brito et al. (2007) state that analysis of these results should consider the factors that contribute to the densification of the ceramic body during the firing step. This factor was also observed in this study because it has helped to reduce porosity of the final product.

As stated by Pisciella et al. (2000), Obrador apud Xu et al. (2008), Silva and Mello Castanho (2004) and other authors, vitrification processes and recycling of industrial wastes are combined processes that represent a viable solution to the environmental impact of various industrial activities and opens opportunities to assign value to waste.

Some parameters observed in this study are from relevant interest and can provide good characteristics in obtained ceramic, like particle size, compression force, increase temperature and firing temperature. Compression force promotes good contact between particles, which generates good densification of the sample. Increase temperature rate is responsible for good border interaction and will generate liquid phase between particle surfaces. Moreover, associate with constant temperature, will produce ceramic with good flexure resistance. All these characteristics were observed in tested samples.

According to results presented, it is possible to obtain bricks (red ceramics) through the addition of industrial waste in significant quantities, as shown in the work. Special attention should be given to initial sample preparation (mixture of materials), followed by compaction, that gives a homogeneous product, and free of clumps,

which can prejudice the outcome of the final product.

In SEM analysis, evaluated sample presented vitreous phase, characteristic of ceramic. Leaching and solubility tests demonstrated no presence of heavy metals in composition, that confirm the presence of vitreous phase in the obtained ceramic. These conditions permit to stablish the proposed idea to encourage the development of environmentally friendly product, with decreasing exploration of clay mines, reduced power consumption and less impact on associates costs production.

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