https://dx.doi.org/10.22161/ijaers.5.12.39 ISSN: 2349-6495(P) | 2456-1908(O)

Use of Clay Sludge Water Treatment Plant Sludge to Produce Ceramic Brick

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***Abstract* — *In the search for solutions capable of minimizing the environmental impacts from waste water treatment plant (WWTP) and reduce costs related to the final destination of the waste generated, the present work aims to evaluate the potential use of WWTP sludge by incorporating clay slurry to the production of bricks, in order to present an alternative environmentally correct destination for this waste. Sludge and clay underwent chemical and mineralogical characterization, through granulometric distribution, X-ray fluorescence analysis, X-ray diffraction. The samples were homogenized in the proportions of 0%, 12%, 14% and 18% of sludge in red clay, pressed at 25 MPa, and later production of test specimens which were then burned to a temperature of 900ºC. After sintering, physical and mechanical tests were performed to evaluate the quality of the final product, by means of analysis of the properties of fire loss, linear retraction, water absorption, apparent specific mass, apparent porosity, flexural rupture stress, moisture and plasticity. The results of the laboratory tests with the residue proportions incorporated in the clay mass demonstrate influence on the physical and mechanical properties of the ceramic material. The results presented show a similarity to that recommended and that despite the 18% (sludge) samples, in relation to the clay showed a variation in the chemical composition due to its high organic matter content, it was observed that both had SiO_2, $Al2O_3$ and Fe_2O_3. Comparing the results with the parameters established in NBR 15270-1, 2 and 3/2005, it was verified that the WWTP sludge can be incorporated in up to 18% in the clay mass for the manufacture of bricks.*

Keywords— Waste water treatment plant, recycling, solid waste.

I. INTRODUCTION

The environmental impacts caused by the high solid waste index are consequences of the necessary economic expansion and the technological advance, imposing to society a consumerist rhythm. This consumption concept of modern society contributes to the various sources of waste generated, from the activities of industry, commerce, ore, fishing, water supply, residences, among others.

With the objectives of a cleaner production and commercial competitiveness, companies are increasingly concerned about the reduction and correct destination of waste generated in their production processes, and product life cycle. This concern is always aimed at minimizing the costs of solid waste treatment and the negative impacts on the environment.

The WWTP for public supply transform inadequate water for human consumption into sanitary safe water, in accordance with the drinking standard established in Brazil through decree nº 2,914 / 11 of the Ministry of Health [1].

The water supply industry, when using the complete or conventional treatment (coagulation, flocculation, decantation and filtration), carries out processes and operations such as the introduction of chemicals, which generate the residue [2].

This byproduct, generated from the addition of chemicals and water, is called sludge and is composed basically of soil particles, organic material drawn to raw water.

The sludge generated in the WWTP usually has an inadequate final destination, being exposed to the environment, contaminating it [3]. E a decomposição do solo e a contaminação de nascentes e lençóis são exemplos de danos ambientais causados pelo descarte inadequado de resíduos [4].

The fate of WWTP residue has long time been the water courses near the stations. However, current legislation is restricting and even prohibiting this practice. According to the National Policy on Solid Waste law 12.305/10 [5] e a NBR 10.004/871 [6], these sludges, classified as solid waste, should be minimized, reused and / or recycled, being prohibited in water bodies.

This material is a compound capable of causing environmental pollution, containing chemicals which, if disposed of in the environment without proper treatment, can cause serious environmental damage to the soil and aquatic life.

Heavy metals such as copper, zinc, nickel, lead, cadmium, chromium, manganese and, in particular, aluminum and iron, present in the sludge, have toxic actions [7]. The toxicity is also due to reactions during the process, form and retention time, characteristics of the watercourse, composition and impurities and other chemicals used in the treatment of water [8].

For [9], the generation of WWTP residues increased due to the use of higher concentrations of chemicals, since the water quality of the rivers is poor and the demand for drinking water is higher.

The sludge is considered the biggest environmental liability in the sector, since the concern with its correct disposal, without damaging the environment, has been gaining increasing attention, due to its composition, with the presence of metals [10].

Due to the high costs and environmental restrictions, many countries are looking for economically and ecologically viable alternatives for final disposal of waste generated at a WWTP. Although most developed countries have already adapted their systems to treat sludge, underdeveloped countries still release this material directly into the waterways and few stations have been concerned with waste disposal treatment [11].

One of the residues that present potential to be recycled in red ceramics are the sludge generated in the WWTP [12]. The physical and chemical characteristics of WWTP sludge are often similar to that of materials used in the manufacture of bricks: clay [11].

The raw material used in the production of ceramic bricks, ie the clay is usually composed of different mineralogical species that are mixed during the formation process. The characteristics and constitution of the ceramic products depend on these minerals or chemical compounds qualitatively and quantitatively and on other parameters of the raw material such as granulometry, thermal behavior and behavior in the presence of water.

The Polo Oleiro of Iranduba / Manacapuru, metropolitan region of Manaus in Brazil, presents 32 industries, whose production distribution varies in: Up to 100,000 bricks/month: 4 companies; from 100 to 400,000 bricks/month: 7 companies; more than 400,000 bricks/month: 9 companies [13].

The production of pottery depends directly on natural resources, especially clay and wood. Exploitation of the Iranduba polo clay deposit on the right bank of the Negro River is considered negative due to the environmental impacts [14]. The impacts promoted by the activity oleira stand out ditch opening for extraction of clay, deforestation for removal of wood, the production of smoke.

Both the extraction of clay and the generation of WWTP sludge cause serious environmental problems in water, soil and air. The problem of the disposal of the sludge would be solved if it successfully incorporated the clay sludge. In this case, the incorporation of sludge in the ceramic material, contributes to the reduction of pollution, and at the same time makes possible an alternative for the production of brick in the potteries [15].

The need to reduce the environmental impacts generated by the solid waste produced by the water treatment plants in the City of Manaus shows that it is necessary to develop techniques for adequate final disposal and treatment of sludge. In order to do so, it is believed that the WWTP should be adapted to solve solid waste problems, since it is necessary to review the production and consumption models, adopting laws that aim to recover the waste at source, seeking reuse or recycling. Therefore, it will be necessary to seek partnerships, especially with companies of the nearby ceramic pole.

It is in the search of answer to the questions that define the objectives of this work, because it seeks to understand the environmental impacts generated by solid waste in the water treatment processes in the City of Manaus, since the incorrect disposal of these residues threatens streams, rivers and groundwater, and even the municipal landfill. The research proposes as an alternative for an adequate final destination of the waste generated in the decanting process in water treatment plants in the city of Manaus. The present research will be based on a series of standards and legislation, with Law No. 12.305/2010 being the main frame, as it deals with the National Policy on Solid Waste.

II. DEVELOPMENT

2.1 The Water Treatment

The water consumed by man is a source of well-being and health, requiring treatment for the removal of impurities that are in the form of suspended particles and particles in a colloidal state. Colloidal state is understood to mean particles with a diameter of less than 1 μm, and those with a diameter of more than 1 μm are known as slime. The removal of these particles is performed through the coagulation/flocculation process [16].

2.1.1 Chemical Coagulation

It is important to have clarity of the terms "coagulation" and "flocculation" and that there is a difference between them that cannot be confused.

The term coagulation comes from the Latin "coagulare" which means to join. This process describes the effect produced by the addition of a chemical to a colloidal dispersion which results in destabilization of the particles by reducing the forces that tend to keep them apart. In this

https://dx.doi.org/10.22161/ijaers.5.12.39 ISSN: 2349-6495(P) | 2456-1908(O)

case the particles begin to agglomerate allowing contact between them, forming particles of submicroscopic size.

Flocculation is characterized by the formation of sedimentable particles from destabilized particles of colloidal size. The term flocculation is also derived from the Latin "flocculare" which means to form a flake similar to a very fibrous porous structure. Unlike coagulation where the primary forces are electrostatic in nature, flocculation occurs through a mechanism of formation of chemical bridges (Van der Waals forces). Macroscopically flocculation transforms coagulated particles of submicroscopic size into more visible ones facilitating the sedimentation by gravity that occurs in the process of cleaning the water [17].

We have news of water treatment for human consumption, including the use of chemical substances (aluminum salts), since the year 70 BC. According to the historian of the time, named Pliny, aluminum salts were known as the clay of Italy, which was already an important commodity of world trade, since it had the capacity to transform "bitter waters into drinking water" [17].

In general, water presents impurities from soil decomposition, mineral dissolution and vegetation decomposition, all dissolved in water. Furthermore, the need for coagulation is increasing steadily as a result of increased water pollution and accelerated population growth in the world [18].

The impurities contained in the waters of natural origin or man-made origin, are of organic or inorganic origin. The inorganic ones are responsible for the variation of the turbidity being that the taste and odor are caused by the organic substances dissolved.

The particles that produce turbidity are classified according to their size, the molecules have an average size of 50μm. The fraction of molecules with a diameter greater than 1μm are known as slime and settle easily when the water is at rest. On the other hand particles with a diameter smaller than 1 μm known as colloidal remain in suspension for long periods of time and therefore coagulation is necessary for its elimination [19].

Therefore, the need for coagulation is for the colloidal particles to form larger aggregates and thus increase their sedimentation rate. The formation of larger particles from smaller ones is important for a phase of cleaning the water known by filtration [20].

2.1.1.1 Factors that influence coagulation

To achieve a suitable water coagulation pattern, a complex network of variables such as pH, turbidity, water chemical composition, type of coagulant, and physical factors such as temperature and mixing conditions must be taken into account. These interactions are so complex that it is theoretically impossible to determine an appropriate coagulant pattern for a given water sample, so the amount of coagulant is determined empirically for each type of water [21].

2.1.1.2 The Effects of pH

There is a pH scale for a given sample of water within which good coagulation/flocculation occurs in the shortest possible time. The variation of this scale is influenced by the type of coagulant that can be used, by the chemical composition of the water and the concentration of the coagulant [20].

The most commonly used coagulants are aluminum salts (aluminum sulfate) or iron salts (iron chloride) called metal coagulants. Os coagulantes de metais precipitam e coagulam mais rapidamente, e o pH deve estar entre 5.8 e 7.8. Depending on the turbidity and the presence of ions in the water this variation rises to 6 to 7.8 [22].

2.1.2 Sludge generation in WWTP

Water treatment plants collect water from rivers, carry out adequate treatment and distribute it as drinking water to the centers for human consumption. The processes used in these stations are, as a rule, the following [23]:

- Oxidation
- Coagulation
- Flocculation
- Decanting
- Filtration
- Disinfection
- pH stabilization
- Fluoridation

In the oxidation, chlorine is injected into the raw water collected to oxidize the dissolved metals, especially iron and manganese. Coagulation adds lime to maintain the pH at the appropriate level and then after aluminum salts or iron salts as the primary coagulant to form the impurities flakes. In contact with water this coagulant reacts almost instantly, promoting a hydrolysis reaction, resulting in the formation of certain compounds that will be, together with the impurities present, constitute the flakes, which will be separated later in the settling and filtration units [17].

In flocculation, the water is mixed in tanks, with flakes of larger impurities beginning the decantation phase. After decantation, the water passes through filters with anthracite, sand, and gravel retaining the impurities that were not sedimented in the previous steps [23]. These impurities withdrawn from the water, mainly from the decanters and from the washing waters of the filters are called sludge from the WWTP sludge water treatment plant. The characteristics of these impurities depend on the conditions of the raw water withdrawn from the source, the dosages and chemical products used and the way of cleaning the decanter filters.

The sludge lagoon is the place of disposal of the sludge after its removal from the decanters and then it is sent to the drying beds where the free water is drained and can be

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returned to the treatment system, depending on the treatment plant.

A maioria das estações de tratamento de água realiza limpezas periódicas, em média uma vez por mês, manualmente, isto é, esvaziando o decantador e lavando o fundo. Em seguida, temos a seguinte seqüência de tratamento de água em uma WWTP: coagulação, floculação, sedimentação, filtração, remoção do lodo - fluoretação [24].

2.1.2.1 Characteristics of WWTP sludge

Due to the addition of aluminum salts or iron salts to cause the coagulation of the existing residues in the raw water, the hydroxides of these salts become the main chemical components of the sludge and, in addition, organic and inorganic particles. In the absence of algae and other organic materials in the fountain, the organic fraction of the sludge becomes negligible and the sludge has chemical stability characteristics, being composed only of inorganic matter, such as fine sands, limes and clays [25].

2.1.2.2 Water removal

The hydroxides formed due to the salts added during the coagulation process hinder the dehydration of the sludge during the drying phase making it gelatinous and bulky and of thixotropic viscosity. A widely used parameter for assessing the difficulty of removing water is "specific filtration resistance". The higher the value of this parameter, the more difficult it is to remove the sludge water by filtration [20]. Sludge produced in WWTP using aluminum salts or iron salts as coagulants presents specific resistance ranging from $5x10^{12}$ a $50x10^{12}$mg/kg. Sludge with specific resistance greater than $5x10^{12}$mg/kg are difficult to dehydrate and those with values less than $1x\ 10^{12}$m/kg are easy to dehydrate, so the sludge generated in the WWTP is difficult to dehydrate [26].

All sludge consists of a combination of a solid phase with a liquid phase. The different physical forms of water in the sludge exert a marked influence on the greater or lesser difficulty of separating the liquid phase from the solid [26]. The physical states of the water are defined. in order of increasing difficulty of dehydration, that is, they present greater free water withdrawal facilities followed by interstitial water, vicinal water and finally water of hydration because they have chemical bonds with the surface of the solid particles [26].

A better efficiency in the operations of reducing the amount of water in the sludge is through the addition of lime or solutions of synthetic polymers known as polyelectrolytes [26]. Synthetic polymers are classified into anionic polymers, cationic polymers, nonionic polymers, being related to the charges presented by their molecules in aqueous solution [20].

2.1.3 Final disposition of WWTP sludge

The sludge treatment system involves techniques whose pertinence are influenced among other factors by the characteristics of the sludge, available area, local climate and environmental conditions of the region. Most treatment plants discharge it directly into the sewage system, without any kind of treatment, other than the presence of a tank for control and regularization of the discharge flow [26]. An alternative usually adopted is the use of ponds or drying beds, and after drying the sludge is sent to landfills in the treatment plant [20].

In temperate regions where sludge is difficult to dehydrate, sludge ponds are transformed into a drying bed, placing drains at the bottom of the ponds. After the drying period, both the ponds and the beds should be removed by dredging of the concentrated sludge at the bottom of the units for subsequent final disposal, which depending on a technical and environmental analysis may occur in several ways [26].

2.2 Solid wastes

The brasilian standard NBR 10.004 (ABNT, 1987) defines solid waste as solid and semi-solid waste that results from community activities of industrial, domestic, hospital, commercial, agricultural, service and sweeping origin.

Included in this definition are sludges from water treatment systems, those generated in pollution control equipment and facilities, as well as certain liquids whose peculiarities make it unfeasible to be released into the public sewer or water bodies, or require for this technical and economically unviable solutions in the face of the best available technology. It is also verified that the commercial residues have composition according to the type of generating trade.

2.2.1 Classification of Solid Residues

The Brazilian Association of Technical Standards - ABNT, through NBR 10004/04 [6], defines solid waste as follows: Solid and semi-solid waste, which results from activities of industrial, domestic, hospital, commercial, agricultural, public service, such as sweeping, pruning of trees, among others. Solid waste is also considered to be the sludge from water treatment systems, those generated in pollution control equipment and facilities, as well as certain liquids, whose particularities make it unfeasible to be introduced into the public sewage system or water body, or require technical and economically unviable solutions in the face of the best available technology Brazilian Technical Standards Agency (ABNT, 1987)[6]. For all these wastes there are several classifications, as waste class: I - Hazardous; Class II waste - Non-hazardous; Class II A waste - Not inert; Class II waste B - Inert, hazardous and non-hazardous, inert and non-inert (Fig. 1).

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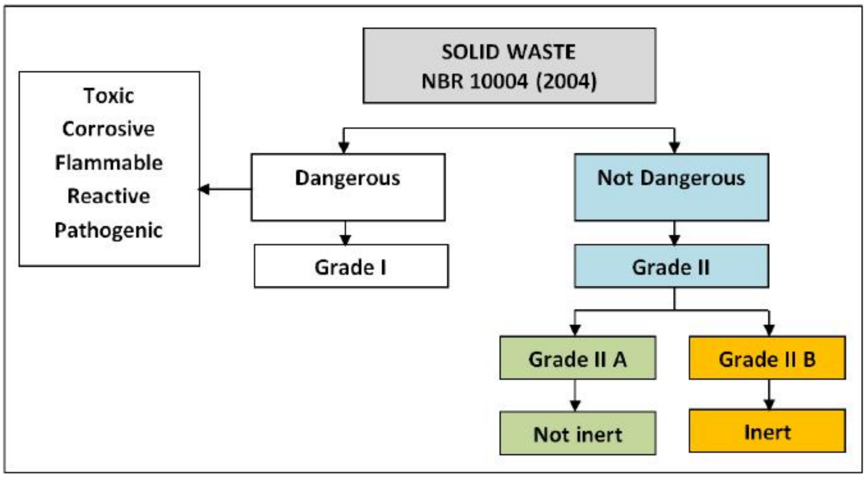

Fig. 1: Classification of solid waste.
Source: NBR, 10.004/2004(adapted from)[6].

Entre os resíduos de alto impacto ao meio ambiente, destacam-se os resíduos da estação de tratamento de água, o lodo WWTP. This waste is classified as Class I Hazardous Waste: where these can, in view of their flammability, corrosivity, reactivity, toxicity and pathogenicity characteristics, present risks to public health, causing or contributing to the increase in mortality or incidence of diseases or to present effects high impact on the environment when handled or disposed of improperly.

III. MATERIAL AND METHODS

For the present study, two raw materials were used: waste from the water treatment plant for Manaus and clay pottery Cerâmica Montemar, from the municipality of Iranduba, State of Amazonas in Brazil.

3.1 Preparation of samples

The clay was subjected to the drying process in an oven at 110ºC for 24 hours and then milled in a ball mill for 30 minutes in order to reduce the clods of clay.

The solid residue was dried at 50 ° C for 48 hours. Then, ball milling was performed for 30 minutes. 15 grams of the residue was calcined at 900 ° C for 3 hours in an electric furnace Linn Elektro Therm LM 312.06, for further analysis.

The grinding of both the clay and the residue was carried out in a ball mill for the WORK INDEX test of the brand QUIMIS, series 005 and model MA 08/20. After grinding the raw materials were characterized.

3.2 Characterization of raw materials

For the characterization of the raw materials (clay and residue), the chemical analyzes of the clay, the ground residue and the calcined residue were carried out by X-ray fluorescence, and for the analysis of the structure and identification of the mineralogical composition of the clay was used an X-ray diffraction apparatus.

The granulometric analyzes of the clay and the residue were done by wet sieving, using the opening sieves, according to ABNT: # 16, #30, #40, #50, #100, #200.

3.3 Preparation of test specimens

For the preparation of the ceramic masses, were first measured the humidity of the clay and the residue, which were 4% and 7%, respectively, to make the correct corrections in the mixtures. Using an Adventurer OHUS brand analytical balance, model AR 3130 Class II, the raw materials were weighed by varying the residue concentrations by 0%, 12%, 16% and 18%. Then, water was added to the formulations in 10% of the mass, to facilitate the conformation of the proof bodies. Each mixture was subjected to a homogenization process for 1 hour in a ball mill.

Afterwards, the masses were subjected to the pressing process using a steel mold in the form of prismatic blades with dimensions of 100x50mm and taken to a hydraulic press, brand MARCONI, series 011639, with a maximum capacity up to 20 tons, using pressure of 2 (two) tons. 48 (forty-eight) test specimens were made, of which 12 (twelve) for each formulation, with dimensions on the order of 72,03 x 31, 67 x 7,77 mm.

After pressing, we measured the masses of the prepared test specimens, with analytical balance Adventurer OHUS, model AR 3130 Class II, being denominated wet mass (Wm) and length value (L_0), with a digital analog caliper 200mm / 8 precision, ZAAS Precision brand, Stainless Hafoi model, 1/129 "0.05" mm graduation, accuracy of ± 0,05, before going through the drying process.

The molded samples were placed in a model MA033 oven of MARCONI brand, with temperature around 110ºC, for 24h. After drying, the masses of the test specimens, with analytical balance Adventurer OHUS,

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model AR 3130 Class II, denominated dry mass (Dm), were measured.

The process of burning of the test specimens was carried out in an oven micro processed muffle furnace, QUIMIS brand, at a temperature of 900ºC, with a temperature range of 3 (three) hours and a heating ramp of 10ºC/min. The mass of the test specimens after burning (Mb), with analytical balance Adventurer OHUS, model AR 3130 Class II, besides the value of the length (L1), the thickness (h) and the width (b) of these bodies were measured, with ZAAS Precision caliper, Stainless Hafoi model, graduation of 1/129 "0,05" mm, accuracy of ± 0,05.

3.4 Evaluation of the ceramic properties of test specimens

The following tests were performed on the test specimens: flexural rupture stress (FRS), Linear retraction after burning (LR), water absorption (WA), Apparent porosity (AP), apparent specific mass (ASM).

These physical and mechanical tests are of extreme importance, and through these tests the quality of the material is determined. The formulas used for the determination of these properties are presented by [27]:

Flexural Rupture Stress (FRS)

$$FRS\,(Kgf/cm^2) = \frac{3}{2}\,x\,\frac{P.L}{b.h^2} \tag{1}$$

Linear Retraction após a queima (LR)

$$LR(\%) = \frac{L_0 - L_1}{L_0}x100 \tag{2}$$

Water Absorption (WA)

$$WA\,(\%) = \frac{P_U - P_S}{P_S}x100 \tag{3}$$

Apparent Porosity (AP)

$$AP\,(\%) = \frac{P_U - P_S}{P_U - P_i}x100 \tag{4}$$

Apparent Specific Mass (ASM)

$$ASM\,(\%) = \frac{P_S}{P_U - P_i}x100 \tag{5}$$

Where,

P = water mass + container mass (Kgf)

L = distance between the device support points (cm)

b = width of the test piece (cm)

h = thickness of the test piece (cm)

L_0 = Length before drying (cm)

L_1 = Length after burning (cm)

Pu = mass of the moist body (g)

Ps = dry body mass (g)

Pi = body mass immersed in water (g)

3.5 X-ray diffraction analysis in ceramic products

Para identificação da composição mineralógica dos produtos cerâmicos, as análises de difração de Raios-X foram realizadas nos corpos de prova com formulações de massa cerâmica de 0%, 12%, 16%, e 18% de resíduo incorporado, sinterizados a temperatura de 900ºC. Estes corpos de prova foram pulverizados em moinho de bolas WORK INDEX, da marca CIMAQ, série 005 e modelo MA 08/20, até textura de pó.

IV. RESULTS

4.1 Characterization of samples

4.1.1 Análise Granulométrica da Argila

According to the results of the granulometric analysis (Fig. 2), it is observed that more than 80% of the clay has a particle size less than 0.075mm (# 200 ABNT).

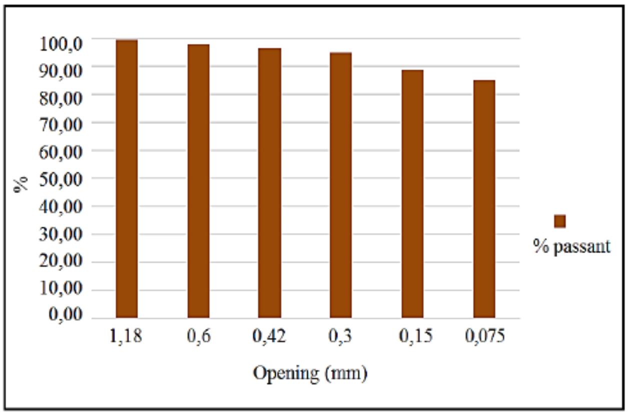

Fig. 2: Granulometric distribution of clay.
Source: Authors, 2018.

The ABNT NBR 6502/ 95 [28], classifies the soil particles according to the grain diameter, being that the clay soil has a granulometry of less than 0.002 mm, this type of fine granulation soil, well grouped, has characteristics of high plasticity, that after wet, facilitates the molding of the bricks. In a study carried out with the soil, from the same region of Iranduba / Am, analyzed in this work, that clay, which in turn is a chemical compound that has a very fine granulometry and, when moistened with water, form a mixture with a certain plasticity [29].

Particle size analysis of the Pará region, performed by [30] observed that more than 70% of the clay had grain less than 0.038mm (# 400 ABNT) and these characteristics were important for the incorporation of the sludge into a ceramic mixture.

4.1.2. Granulometric analysis of the residue

In order to conduct the granulometric characterization of the sludge, the standard ABNT NBR 7181/ 84 were used [31].

It is possible to observe in the results of the granulometric analysis of the WWTP sludge in (Fig. 3), where it presents the percentage of the passing material according to the openings of the sieve, that the residue has continuous graduation in the sieves of 0.3 to 0.075, that is the material analyzed is well graded.

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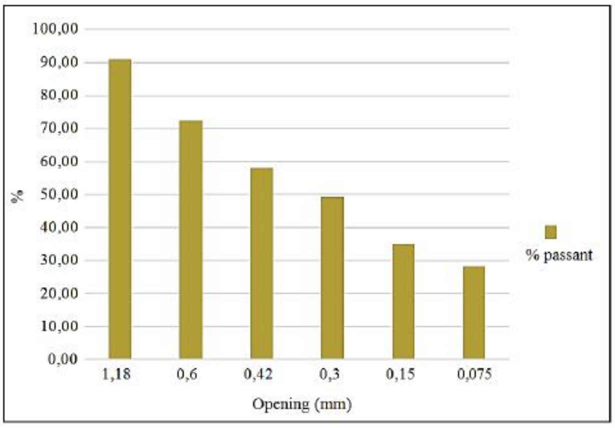

Fig. 33: Granulometric distribution of the residue. Source: Authors, 2018.

Possibly without void spaces which avoids the porosity of the material and increases its mechanical strength. It is also observed that more than 50% of the material have dimensions lower than 0.42%, which facilitates the homogenization between the grains of clay and residues.

In the particle size analysis of WWTP residue, performed by [32], He verified that the particle size distribution was in the range of 0.001 to 1.2mm, these results showed a wide range of particle size distribution, as it chose to use the residues that left the crusher without prior classification of the granulometry. The author [32], still checked that the sludge particles had an irregular profile due to not having obtained a standard granulometry in the process of preparation of the sludge particles. In this study, the well-graded and continuous WWTP sludge residue is considered a positive result, because, with regular decreasing of grain sizes, it facilitates the homogenization and aggregation of soil particles during cooking.

4.1.3 Real grain density (GD)

The results of the real density of clay and sludge grains (Table 1) reflect the presence of minerals and organic matter present in the soil.

Table 1: Real grain density of clay and sludge samples.

Standard: DNER-ME 093/94	Clay	Sludge
Picnometer	1	2
Temperature °C	25	26
Weight pycnometer (g)	47.46	47,46
Weight of the pycnometer + dry sample (g)	67, 47	66,15
Weight of the pycnometer + sample + water (g)	159,55	160,01
Weight of the pycnometer + water (g)	151,19	152,20
Weight of dry material (g)	20,0	20,0
Real grain density (g/cm^3)	1,717	1,718

Source: Authors, 2018.

The limits of actual density vary between 2.3 and 2.9 g / cm³, it can be observed in Table 4 that both clay and residue have low real grain density, between 1,717 and 1,718, respectively, which characterizes a soil with high organic matter, positively influencing the porosity, because few porous cavities do not weaken the ceramic material.

4.1.4 Fluorescence of X - rays

It is observed that X-ray fluorescence results in both clay and residue (sludge) presented a higher concentration of aluminum oxide (Al_2O_3) compounds at 51.33% and Silicon Oxide (SiO_2) at 55 , 41 (Table 2). therefore the presence of this compound in the analyzed materials was expected, as for Al_2O_3, is mainly due to the type of coagulant (aluminum sulphate) used during the purification of water in the Treatment Station of PROAMA. This fact was verified by [33], in analyzes of the clay and sludge elements, where it found higher concentration of Aluminum Oxide (Al_2O_3) and Silicon Oxide (SiO2), due to the type of coagulant used to decantation and mineralogical sedimentation characteristics of the Amazon region. The greatest predominance of elements in the sludge was aluminum, iron and silica; being that the highest content was aluminum, due to the use of aluminum sulphate as a coagulant [34]. This chemical similarity between the materials corroborates the addition of the clay sludge to the ceramic brick making, because they prevent undesired reactions during the burning process of the test specimens.

Table.2: Chemical composition in percentage of clay, residue and ceramic mass with 18% of residue.

X-Ray Fluorescence			
	Concentration (%)		
Compost	Clay	Residue	Ceramic mass with 18% of waste incorporated
Al_2O_3	51, 33	55,41	51,40
CaO	0,01	3,95	0,15
Cl	0,006	0,04	0,005
Cr_2O_3	0,008	-	-
Fe_2O_3	2,71	6,61	3,30
K_2O	0,37	1,25	0,41
MgO	0,18	0,48	0,21
MnO	0,005	0,03	-
Na_2O	0,14	0,31	0,22
Nb_2O_5	0,003	0,02	0,003
P2O₂	0,06	0,31	0,04
Rb_2O	0,002	-	-
SiO_2	45, 74	36,38	42,16
SO_3	0,05	1,44	0,08

Source: Authors, 2018.

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4.1.5 Diffraction of X-rays

The results of the microstructural characterization of crystalline materials, performed by means of X-ray diffraction, in the clay are shown in Fig. 4.

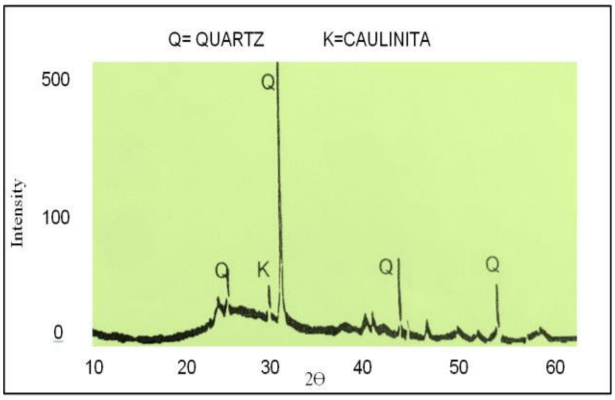

Fig. 44: X-ray Diffractogram of clay.
Source: Authors, 2018.

It is possible to observe that the most pronounced phases are: quartz (SiO2) with a major peak, followed by kaolinite ($Al_2Si_2O_5(OH)_4$), this is due to the fact that most of the clay minerals in the state of Amazonas originate from residual deposits formed from alterations of rocks of the Alter do Chão Formation composed, among others, by quartz and kaolinite.

Most of the region of the Manacapuru-Iranduba ceramic pole is constituted of those of the residual deposits. Some studies of the clay extracted from the banks of the Guamá River, in Pará [30] indicate that the mineral quartz is the majority, followed by kaolinite that was also found in that region.

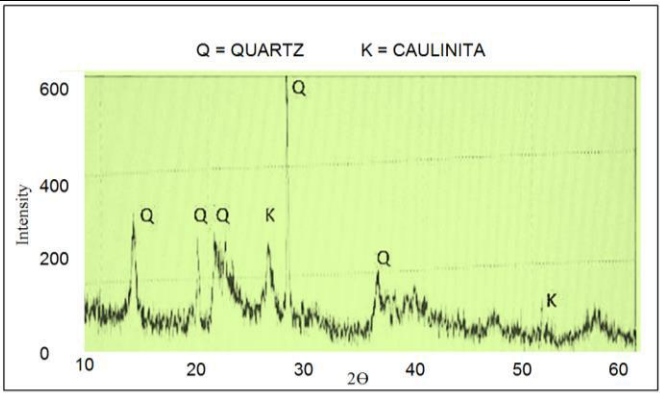

Fig. 55: X-ray diffraction of the sludge.
Source: Authors, 2018.

It is possible to notice that the main mineral found in the residue, as well as in the clay analysis, is quartz, followed by kaolinite (Fig. 5).

This mineral similarity between clay and sludge is of extreme importance due to the physical and mechanical benefits of incorporating the residue into the clay mass [35].

The quartz mineral, found in this study, confirms the chemical composition found in X-ray fluorescence analysis, since the chemical composition of quartz is silica dioxide, which in turn influences thermal expansion during cooking. In Fig. 6 shows the results of the X-ray diffraction in the test specimens with a proportion of 18% of the residue after burning at 900 ° C. It is possible to notice that, there was a favorable increase of the quartz peaks and reduction of the intensity of kaolinite. The reduction of kaolinite occurred due to the sintering of the test specimens, with the linear organization of the minerals present, improving the quality of the final product [33].

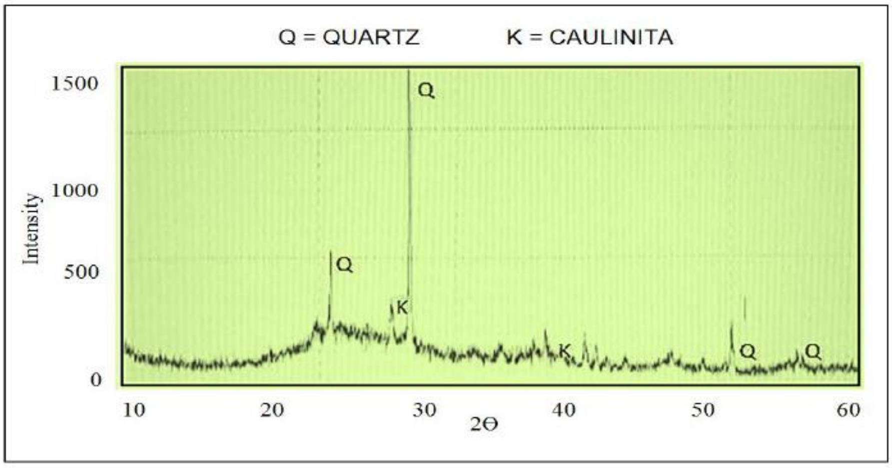

Fig. 6: X-ray diffraction of test specimens after burning, with 18% of residue.
Source: Authors, 2018.

This behavior occurs due to the addition of residue that favors the appearance of hematite and the more residue added, the iron present in the sludge tends to accelerate the solid state reactions, giving rise to a new crystalline

https://dx.doi.org/10.22161/ijaers.5.12.34 ISSN: 2349-6495(P) | 2456-1908(O)

phase, cristobolite [20].

4.2 Physical and mechanical analysis after burning

4.2.1 Linear Retraction (LR)

It is possible to observe in the results of the linear retraction, after the burning in temperature around 900 ° C, as a function of the percentages of 0%, 12%, 14% and 18%, of residue added in the formulation of the clay mass (Fig. 7), that the linear retraction increased with the increase of the clay content to the clay mass in all the percentages, this fact is due to the sintering process of the heated test specimens at high temperatures.

The grouping of the molecules is an important and necessary factor in the cooking of the ceramic materials, as it causes hardening to take place and, consequently, to give resistance of the final product.

During the cooking the pieces contract due to the occurrence of coalescence of the powder particles by the sintering process [36].

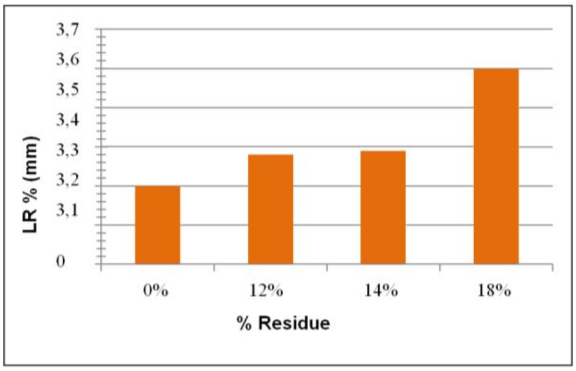

Fig. 7: Linear retraction in test specimens.
Source: Authors, 2018.

The driving force of sintering is the reduction of the surface of the powder [37]. This behavior indicates a favoring in the process of sintering of the test specimens [30]. This sintering process, which occurs during firing, creates a change in the microscopic structure due to the minerals, such as quartz, causing the part to become solid, which results in good resistance of the ceramic product.

All retraction values are in accordance with the limits established by the Brazilian standard [38] which is below 8%. The incorporation of WWTP sludge in the ceramic brick manufacturing process, in the proportions of 0%, 12%, 16% and 18% of residue, showed an increase in the linear retraction, at sintering temperature at 900°C, possibly due to the presence of organic matter that volatilizes in the burning. This behavior indicated that the organic matter volatizou in the burning of the ceramic material at high temperatures [39].

4.2.2 Water Absorption (WA)

The results of the water absorption test of the test specimens as a function of the percentage of residuals added in the clay of 0%, 12%, 14% and 18%, can be observed in Fig. 8.

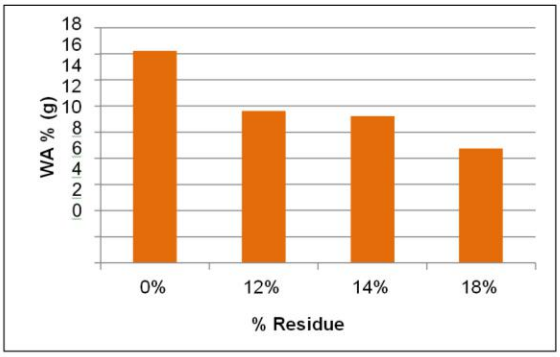

Fig.8: Water absorption of test specimens.
Source: Authors, 2018.

It is possible to note that the larger the amount of residue incorporated into the clay, the lower the occurrence of water absorption. Each of the raw materials influences the changes that occur during the cooking process and the characteristics of the finished piece [36]. In this case, the water absorption decreases with the increase of the sludge content to the clay mass in all proportions, confirming that the more sintered the material is in function of the firing temperature, the greater the linear retraction, and the lower the water absorption, due to the grouping of the molecules. The decrease in WA is expected due to the large presence of clayey property and low presence of sand. Organic matter, as well as moisture, are important parameters that can influence the final quality of the ceramic blocks [39].

The humidity is important to determine the handling of the sludge, since a high moisture content can hinder the routing of the manufacturing components, obstructing passages or adhering to parts of the system [1].

The maximum and minimum water limit is 8% to 22%, respectively [13]. It is noted that the results of moisture content are within the limits allowed by the standard, implying a good result of the material studied.

4.2.3 Apparent Porosity (AP)

After compacting the powder particles into the desired shape, there will be pores or empty spaces between the particles and that, after heat treatment, most of this porosity will be eliminated, however some residual porosity remains [36]. The results of the averages of the apparent porosity, after burning at 900 ° C, as a function of the formulations with residue percentage at 0%, 12%, 14% and 18%, added to the ceramic mass, is presented in Fig. 9.

https://dx.doi.org/10.22161/ijaers.5.12.34 ISSN: 2349-6495(P) | 2456-1908(O)

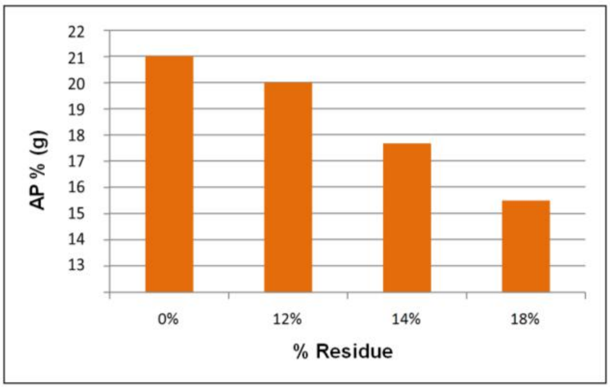

Fig. 96: Apparent porosity of test specimens.
Source: Authors, 2018.

It is noticed that at a temperature of 900°C, there is a contraction in the tests specimens provoking the reduction of the porosity. These microstructural shrinkage and pore reduction changes occur during cooking. During the cooking, the shaped piece shrinks and exhibits a reduction in its porosity, together with an improvement in its mechanical integrity [36]. The apparent porosity decreases with increasing temperature and the decrease in water absorption is similar to that of porosity reduction.

For the present study, the percentage of porosity in the pieces is very important, because the higher the porosity and the water absorption, the lower the resistance and the quality of the ceramic brick, besides other advantages. Because the low porosity has as positivity, greater thermal comfort and less possibility of infiltration for the proper purposes of use in the constructive process and structuring.

However, the total porosity changes negatively, the mechanical properties of the ceramics; however, on the other hand, points out that porosity may be useful to increase resistance to thermal shock [37].

It is possible to admit that, due to these low porosity characteristics, the proportion of 0%, 12%, 14% and 18% of residue incorporated in the ceramic mass will not compromise the flexural strength, so what is intended in this study is the successful incorporation of sludge in the clay for brick making, since what is expected is a non-shock resistant material shock resistant, but a product that supports and resist the stress applied to it in accordance with the standard [38].

4.2.4 Apparent Specific Mass (ASM)

The results of the apparent specific mass of the ceramic products were analyzed as a function of the firing temperature for the formulations of 0%, 12%, 14% and 18% of residue incorporated into the clay (Fig. 10).

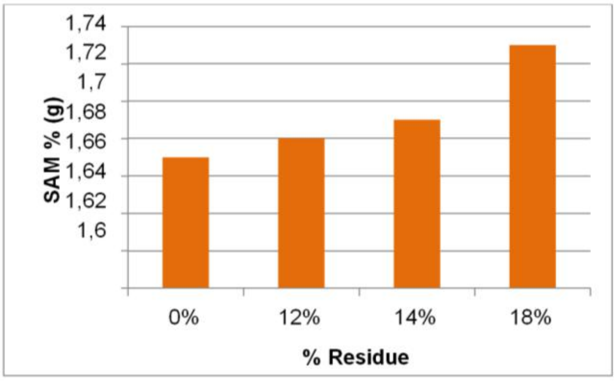

Fig. 107: Apparent specific mass of test specimens.
Source: Authors, 2018.

It is possible to note that in temperature at 900 ° C there was an increase of ASM in all formulations. This fact, too, was evidenced in other similar studies [1, 26, 30, 39], which had the ASM increased under the influence of elevated temperatures. It is possible to notice that the MEA of the proportions was between 1.85% and 2%. This small difference between the values demonstrates a good performance of the residue added to the clay mass. For, the increase of the mass would occur due to the function of the density of the incorporated sludge, as well as the organic matter that dissipates in the process of burning of the test specimens.

The apparent porosity and apparent specific mass are associated with the absorption of water [3]. There was an increase in the mass and decrease of the water absorption, as well as the apparent porosity, these characteristics are expected, as they positively favor the resistance of the final product.

4.2.5 Bending rupture stress (BRS)

It can be noted that the results of the flexural strength tensions (BRS) as a function of the test specimens burning temperatures of 900°C in the 0%, 12%, 14% and 18% formulations (Fig. 11) there was a decrease in BRS with the incorporation of the residue.

This fact was expected, since a good sintering was observed, that is, good union of the existing particles in the test specimens, which were repeated in the linear retraction, water absorption, apparent specific mass and confirmed with the test rupture, since the values found are within what is defined by the standard [38].

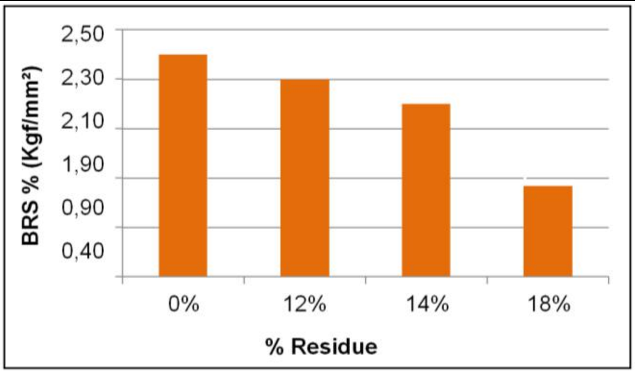

Fig. 118: rupture stress of test specimens.
Source: Authors, 2018.

The decrease of the BRS due to the increase of residue to the ceramic mass was expected, however, does not negatively influence the test specimens. To [39] in tests, showed that the BRS decreased with the concentration of sludge added to the ceramic mass, confirming a good final result of the material.

V. CONCLUSION

By means of the granulometric characterization of soil samples, it can be concluded that clay has more than 80% of particle size less than 0.075mm (#200 ABNT), being classified as clay soil.

In relation to the sludge residue, more than 50% of the material has dimensions lower than 0.42% of continuous grade, that according to ABNT NBR 7181/82, which facilitates the homogenization between the clay and residue grains.

Due to the type of coagulant used in the decantation and mineralogical sedimentation characteristics of the Amazon region, the chemical analysis by X-ray fluorescence, both in the clay and in the WWTP sludge residue, the elements found in the highest concentration were Aluminum Oxide (Al_2O_3) and Silicon Oxide (SiO_2).

The presence of quartz (SiO_2), Caulinite ($Al_2Si_2O_5(OH)_4$) in the clay and in the residue was identified in the X-ray test. This is due to the fact that most of the clay minerals of the state of Amazonas comes from residual deposits formed by alterations of rocks of the Alter do Chão Formation composed, among others, by quartz and kaolinite. This facilitates, due to the similar characteristics of the two raw materials, the mixture for the manufacture of brick.

Physical and mechanical analyzes such as water absorption, apparent porosity, apparent specific mass and bending rupture stress, show that WWTP sludge in the proportions of at most 18% can be incorporated in clay mass for the manufacture of brick with satisfactory results, since they are in conformity with the parameters established by ABNT (NBR 15270-1,2 e 3 de 2005).

To contribute to the reduction of the mineral extraction of the clay, as well as the reduction of the discharge of the sludge in dumps or bodies of water, will bring benefits to the environment with the reduction of the impacts generated by the potteries and water treatment plants for public supply in the capital of the state and municipality of Iranduba.

ACKNOWLEDGEMENTS

To the Graduate Program in Science and Environment (PPGCMA), linked to the Institute of Exact and Natural Sciences (ICEN) of the Federal University of Pará (UFPA) and to the Institute of Technology and Education Galileo of the Amazon (ITEGAM), by support to this research.

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