

CHARACTERIZATION OF WASTE GENERATED AT THE TREATMENT PLANT EFFLUENT TEXTILE AND ITS INCORPORATION INTO RED CERAMIC

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***Abstract.** This study aims to analyze the feasibility of using solid sludge generated in wastewater treatment plant of textile effluent in the manufacturing process of tiles and bricks. To this end, it was characterized the solid waste generated in the process of treating wastewater from a textile industry. Then the percentages of incorporation of this solid sludge in the process of manufacturing red ceramic were identified and characterized. The solid compound was generated by the addition of silt to clay and then it was verified the level of toxicity of the materials. The toxicity analysis of the material after preparation of test samples incorporated with sludge from the treatment plant effluent from a textile company in the region of Joinville, Santa Catarina, showed no toxic characteristics to the environment.*

Key words: clay, red ceramic, characterization, and toxicity.

1. INTRODUCTION

The textile industries are a major factor in the Brazilian economy and the State of Santa Catarina has a great number of these industries, some of the most important of this sector in Brazil, operating mainly in the region of Itajaí Valley. The predominant economic activity in the region of Joinville is rooted in the industry, divided mainly in textile, plastic and metal mechanic. Increased industrialization in the last decade has resulted in a significant increase in waste generation.

According to Oliveira (2002), the solid waste is a major problem threatening life on planet earth, as well as it pollutes the soil, water and air, it also attracts animals as carriers of disease. According to Tenorio and Espinosa (2004) the more conventional classification of solid waste takes into account the origin of the waste that may be urban, health services, ports, airports, bus and rail, agriculture, radioactive, and industrial debris. The Brazilian Association of Technical Norms (ABNT) NBR10004 defines industrial solid waste any waste in solid or semisolid material resulting from industrial activities, including certain liquids and sludge whose characteristics make their launch infeasible in public sewers or water bodies. According to Tenorio and Espinosa (2004) industrial waste can vary from 65 to 75% of waste generated in an industrialized area.

It identifies that the responsibility for the managing and fate of this waste belongs to the industries that generate it, so industrialized societies are constantly being forced to fit the changes of guidelines for the management of sludge and waste. Thus, the industries seek the perspective of sustainable development, making it necessary to develop effective methods to replace the simple disposal of these in industrial landfills. From an economic standpoint, the waste generated by industry besides being considered waste can affect the image of the product. Seeking for new production perspectives, concepts and definitions are presented, it is possible to mention the example of a strategy based on the ideas of Natural Capitalism: the biomimicry. Hawken, A. Lovins and L. Lovins (2002) define biomimicry by the reduce of the waste of material, modifying industrial processes, materials eliminating toxicities, allowing constant recycling. Textile industries to suit the new reality are changing production processes in order to reduce the environmental impact. In addition, the possibility of recycling the waste considered inevitable contributes to this goal by eliminating the emission of substances into the environment. As identifies Almeida (2006), the main pollutants in the textile sector are present in wastewater. The analyzed company develops its products from the textile finishing phase which consumes a relatively large volume of water that can be biodegradable and not biodegradable. It can be found as pollutants of this sector organic substances (starch, dextrin, starch glucose, greases, pectin, alcohol, acid skeptical, soaps and detergents), inorganic chemicals (sodium hydroxide, carbonate, sulfate and chloride), sometimes composed heavy metals (such as chromium, cobalt, copper and zinc) in addition to hlogenados organic compounds and detergents tensoactives (ALMEIDA, 2006). It is observed that the characteristic of sludge generated is directly related to the production

process, in this sense, it is necessary to identify the characteristics of the waste generated to define the most appropriate destination. Based on this statement is that it is presented the main objective of this study: to characterize the residual sludge from an industry of the textile sector, which productive stage is characterized by the phase of completion, aiming at the feasibility of using this material as a feedstock for the production of red ceramic.

2. MATERIALS AND METHODS

To perform the characterization, it was collected sludge from the process of decantation and filter press of the Effluent Treatment Station (ETE) of a textile company in the region of Joinville. The analysis of X-ray fluorescence (XRF) of the residue was performed at the Laboratory of Ceramic Materials, in the State University of Ponta Grossa (UEPG), in Ponta Grossa, Parana. The thermal analysis and the X-ray diffraction (XRD) were performed at the Laboratory of Materials Characterization, in the State University of Santa Catarina (UDESC), Joinville, Santa Catarina.

To conduct the toxicity test were used sludge and argil used for red pottery making town of Joinville. The test was conducted at Biotechnology Laboratory of the University of Joinville Region (UNIVILLE), Joinville, Santa Catarina.

Initially, it was performed a preliminary analysis of waste in order to obtain their classification according to standard ABNT NBR10004/04.

For all elements studied, were preparation made and opening the sample separately, as described in Standard Methods for Examination of Water and Wastewater 20th Edition.

The FRX analysis of the sludge was performed with the Shimadzu equipment EDX-700 model. This analysis was chosen to perform the chemical characterization of sludge. To identify the crystal structure of the sludge it was used Shimadzu diffractometer (XDR 6000) with tube X-ray copper, voltage 40 kV and 30 mA current. The reading was held in the axis direction of 2θ between 5° and 80° with a speed of $2^\circ / \text{min}$. The sludge was also subjected to thermogravimetric (TGA) and differential term (DTA) analysis to evaluate the thermal behavior of the sample of sludge in a brand thermal analyzer Netzsch, model STA 449C Jupiter which operates under an argon flow ($30 \text{ mL} / \text{min}$) for heating and of a protective gas of nitrogen at a rate of $15 \text{ mL} / \text{min}$. The heating rate was $10^\circ \text{C} / \text{min}$ up to maximum temperature of 1200°C . The mass of the sludge sample used for thermal analysis was 28.55 g.

2.1. Fabrication of test samples for toxicity testing

To conduct the toxicity test it was used test specimens molded with the highest concentration of sludge evaluated in this project, which is 20% of sludge, burned at a temperature of 800°C . The mechanical tests were evaluated in the compositions of 10, 15 and 20% of sludge in the mass of argil and at the temperatures of 800, 900, 1000 and 1150°C .

The test specimens, with dimensions of $70 \times 30 \times 5 \text{ mm}$ were molded using the procedures indicated by the Brazilian ceramic industry.

To check the toxicity it was evaluated temperature, dissolved oxygen (DO), conductivity and PH of the cultivation medium of the microorganisms *Daphnia Similis* and also of soluble materials.

It was performed the test for acute toxicity using a solubilized material as described in the NBR 10006, procedure for obtaining solubilized extracts of solid waste. It was prepared 6 concentrations of soluble, where 2 for argil, 2 for sludge and 2 for the composite of 20% sludge embedded in clayey mass.

During tests, microorganisms are exposed to different concentrations of solubilized material, corresponding to different concentrations (0.01%, 0.1%, 10% and 100%).

The dilution water used was the water of maintenance of bodies in the lab. On the day before the tests, ovigerous females of *D. Similis* (maintained in culture through food for 105 cells / mL of algae) have been separated for obtaining neonates aged less than 24 hours.

For each dilution it was used 20 *Daphnia Similis* microorganisms. It was performed four duplicates in each treatment, each duplicate containing five microorganism. The duration of each test was 48 hours and the containers that were used are beakers with 200 ml. It was not made renewal of the solution, ie, of the solubilized and the maintenance water of the bodies *Daphnia similis*. During the tests the organisms were kept without food at a temperature of $20 \pm 2^\circ \text{C}$.

After the test period was conducted to count the survivor microorganisms and the results expressed in percentage of organisms killed or immobilized in each treatment. All data were treated statistically using the Tukey test with significance level of 5%.

3. RESULTS AND DISCUSSION

The analysis for classification of the waste showed that the sludge according to the Annex A of NBR 10004/2004, waste from sewage treatment plant effluent, although within acceptable limits by the standard, should be regarded as toxic waste. Thus, according to the analysis result of leaching, the sludge cake of this textile industry has indicated values within the acceptable and their classification is given as class II A.

3.1. Material characterization

The FRX analysis is presented in Figure 1, compared with the chemical composition of argil used in this work. In this figure, it is possible to verify similarities between the chemical compositions, indicating the possibility of incorporation of the sludge to the argil.

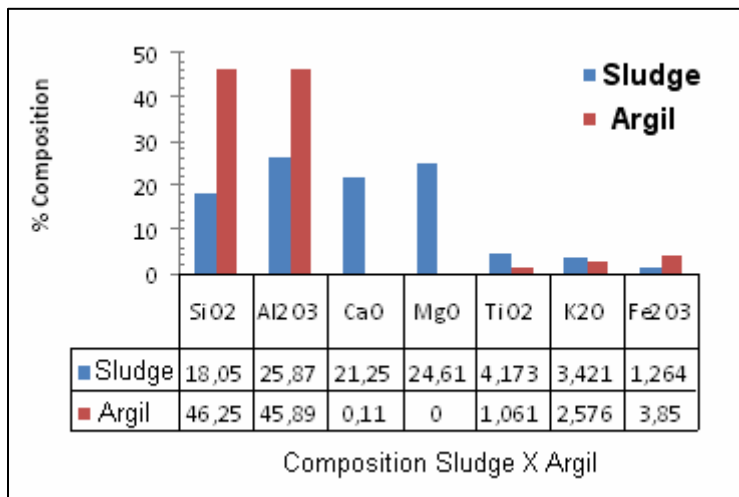


Figure 1. Comparison of the chemical composition of the materials.

It can be observed that the sludge has predominance in the chemical composition of silicon oxide (SiO₂) and alumina - Al₂O₃ as well as the argil. The sludge has a composition more diverse, the high concentration of magnesium oxide and calcium, can be related to the use of these products in the process of wastewater treatment of the company.

The diffractogram of the sludge shown in Figure 2 shows characteristic peaks of crystalline phase, it can be said that there are crystals of alumina (Al₂O₃) and quartz (SiO₂). It observes the occurrence of lime (CaO) used to reduce the pH in the physical chemical treatment, titanium oxide (TiO₂) and potassium oxide (K₂O). The minerals identified are presented compared with patterns from the database of the International Center for Diffraction Data - ICDD.

The argil has a crystalline structure more defined in relation to sludge, with the predominant presence of the peaks of kaolinite and quartz. This condition is consistent with the analysis of X-ray fluorescence (XRF), which presented more than 90% of its composition silica oxide (SiO₂), aluminum oxide (Al₂O₃), which consequently resulted in predominant peaks of quartz and kaolinite

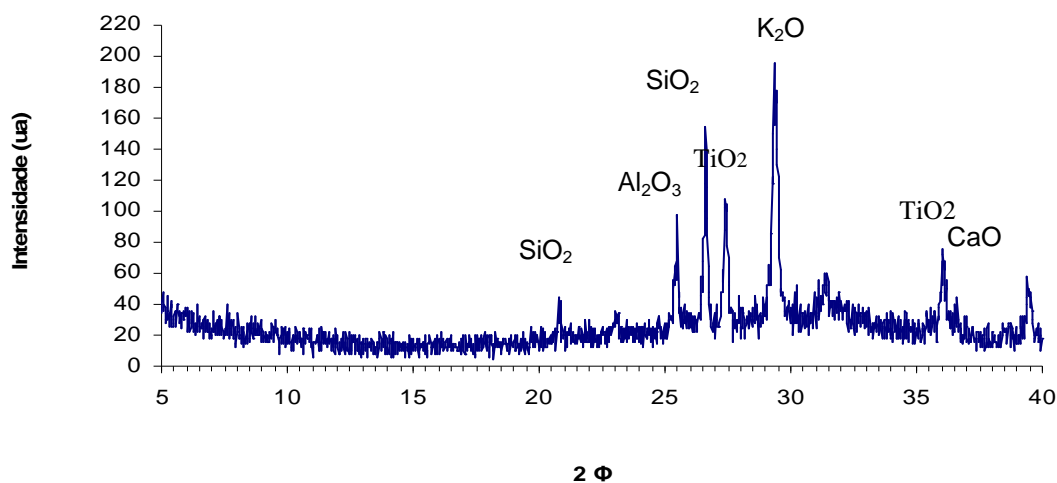


Figure 2. Diffraction spectrum of X-ray of the sludge.

The thermal analysis of the sludge can be seen in Figure 3 through thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC).

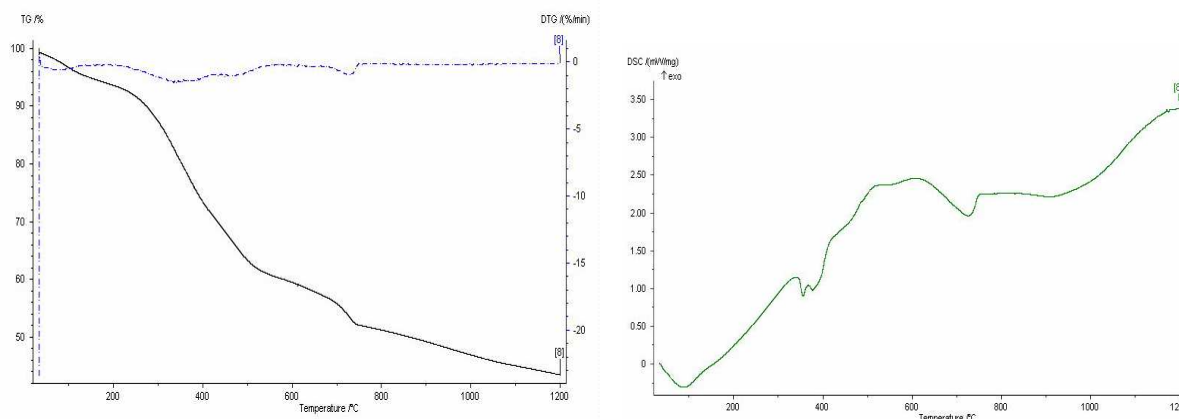


Figure 3. TG Sludge (A) and DSC sludge (B).

The TG curve observed in Figure 3 was treated by Netzsch Proteus ® software and shows three stages of mass loss. The first stage occurs at about 100 ° C and can be attributed to loss of absorbed water in the sludge, accounting for approximately 5% of the mass loss of sample. The second stage, which has the highest weight loss (about 32%) occurs in the temperature range (280-500 ° C), indicating high heat loss, which is probably associated with high content of organic matter contained in the sludge. The third stage occurs around 700 ° C, associated with loss of hydroxyl groups during the transformation of kaolinite into metakaolin. This reaction may be responsible for a mass loss of about 7%. At the end of the experiment, left 43% residue in the crucible, referring to the inorganic material that did not degrade in the temperature range studied (up to 1200 ° C) and can be attributed to the presence of oxides of alumina, silicon and titanium, identified at XRD of this project. Monteiro et. al. (2008) in the study of textile sludge of Goitaca fields also attributed the mass loss occurred between the temperatures 520 ° C and 600 ° C for the transformation of kaolinite into metakaolin. This event was also checked by Correa et. al. (2006), when studied clays in the region of Joinville, assigning the same transformation, the occurrence of an endothermic peak temperature of 600 ° C.

The DSC curve shows an endothermic peak at 100 ° C, according to Rodrigues (2002) refers to the process of dehydration of the material in the same region of water loss obtained by TGA. In about 340 ° C there is an exothermic peak, which may be linked to oxidation of organic matter. Moreira et. al. (2001), which evaluated the textile sludge for manufacturing building materials by DSC, observed two exothermic peaks at temperatures of 300 and 600 ° C and attributed to oxidation of organic matter contained in the sludge. The endothermic peak which occurred about 610 to 700 ° C, occurs at the same temperature range of the transformation of kaolinite into metakaolin, can be attributed to the same event. The peak at about 910 ° C may correspond to the beginning of the process of sintering the material, represented by the beginning of the formation of mullite. Correia et. al. (2006), when studied clays of the region of Joinville (SC), associated with the event occurring at a temperature of 980 ° C, as the beginning of the formation of mullite. Monteiro et. al. (2008) in the study of textile sludge of Goitaca fields also attributed the exothermic peak temperature of 932 ° C the dissociation of metakaolin, initiating the formation of mullite.

Thermal analysis of the sludge showed similarity in events in other studies carried out with sludge and clay. This condition reinforces what is technically possible to incorporation of the sludge in clay.

3.2. Toxicity tests of materials

Table 1 shows the results of find parameters of the solubilized products and to mode cropping prior to testing for toxicity.

Table 1. Parameters of solubilized product evaluated before the toxicity test.

<i>Solution</i>	<i>Temperature</i>	<i>OD</i>	<i>Conductivity</i>	<i>PH</i>
<i>Solubilized clay</i>	27,3 °C	4,5	34,11 mic / cm ²	5,2
<i>Solubilized sludge</i>	24,9°C	4,7	3,250 ms/cm ²	6,94
<i>Solub. composed of 20%</i>	25,8 °C	5,2	80 mic / cm ²	6,4
<i>Mode Cropping</i>	24,3 °C	5,6	200 mic / cm ²	7,2

Table 1 showed that there is great variation in the conductivity of each solution. This condition must directly affect the outcome of the analysis. The temperature, dissolved oxygen (DO) and pH had little variation.

Table 2 shows the results of parameters found of the product solubilized of clay in nature, sludge in nature and compost with 20% of silt incorporated in clay after the test of toxicity.

Table 2. Parameters of product evaluated after the solubilized toxicity test.

<i>Solution</i>	<i>Dissolves</i>	<i>Temperature</i>	<i>OD</i>	<i>PH</i>
<i>Solubilized clay</i>	10 %	24,9°C	4,2	5,8
<i>Solubilized sludge</i>	10 %	24,9°C	4,4	6,09
<i>Solub. composed of 20%</i>	10 %	26,3 °C	4,4	5,8
<i>Solubilized clay</i>	100 %	25,3°C	4	6,15
<i>Solubilized sludge</i>	100 %	25,3°C	1,7	6,76
<i>Solub. composed of 20%</i>	100 %	26,3 °C	4,3	6,15

According to the data of Table 2 shows that there was a significant reduction in dissolved oxygen (DO) in the treatment using 100% of the solubilized sludge. This condition is very detrimental to survival of microorganisms.

Table 3 shows the results of the count of microorganism surviving of treatment of the solubilized product from the clay in nature.

Table 3. Count of microorganisms surviving the 0.0 / 0.01 / 0.1 / 1 / 10 / 100% and with four duplicates of the solubilized of clay.

<i>Solubilized of clay</i>	<i>0 %</i>	<i>0,01 %</i>	<i>0,10 %</i>	<i>1,00 %</i>	<i>10 %</i>	<i>100 %</i>
<i>Population</i>	5	5	5	5	5	5
<i>Survivors after 48 hours</i>	4	5	4	4	5	4
<i>Survivors after 48 hours</i>	5	4	5	5	4	4
<i>Survivors after 48 hours</i>	3	4	4	5	5	5
<i>Survivors after 48 hours</i>	4	4	4	5	5	4

The results shown in Table 3 show that the material solubilized from the clay does not have toxic characteristics. Even when placed microorganisms directly on the solution from solubilized of the clay, it was found that the mortality rate has no significant difference compared to what happened to the solution of the mode cultivation.

Table 4 shows the results of the count of microorganism surviving treatment of the product solubilized of sludge in nature.

Table 4. Count of microorganisms surviving the 0.0 / 0.01 / 0.1 / 1 / 10 / 100% and with four duplicates of the solubilized of sludge.

<i>Solubilized of sludge</i>	<i>0 %</i>	<i>0,01 %</i>	<i>0,10 %</i>	<i>1,00 %</i>	<i>10 %</i>	<i>100 %</i>
<i>Population</i>	5	5	5	5	5	5
<i>Survivors after 48 hours</i>	4	5	4	4	3	0
<i>Survivors after 48 hours</i>	5	4	5	5	4	0
<i>Survivors after 48 hours</i>	3	4	4	5	2	0
<i>Survivors after 48 hours</i>	4	4	4	5	3	0

The results shown in Table 4 show that the solubilized material of the sludge has toxic characteristics. Microorganisms when placed directly into the solution from the solubilized sludge, ie, without dilution with mode cultivation, it was found that the mortality rate was 100%, indicating a significant difference compared to what happened to the solution of the mode cultivation. For dilution, using 10% of the sludge solubilized in the solution of mode cultivation, there was still significant differences between the compositions, but can not be considered a toxic material.

With the objective to evaluate the maximum percentage of dilution of solubilized of the sludge in the solution of mode cultivation, was performed a battery of new tests using dilutions of 0, 10, 12, 15 and 20% according to table 5.

Table 5. Count of microorganisms surviving the 0.0 / 10 / 12 / 15 / 20 / e 50 % and with four duplicates of the solubilized of sludge.

<i>Solubilized of sludge</i>	<i>0 %</i>	<i>10 %</i>	<i>12 %</i>	<i>15 %</i>	<i>20 %</i>	<i>50 %</i>
<i>Population</i>	5	5	5	5	5	5
<i>Survivors after 48 hours</i>	5	3	2	0	0	0
<i>Survivors after 48 hours</i>	4	4	1	1	0	0
<i>Survivors after 48 hours</i>	4	2	2	0	0	0
<i>Survivors after 48 hours</i>	5	3	1	0	0	0

The results in Table 5 showed that the maximum percentage for the dilution of the solubilized of the sludge is 10%, when measured as the dilution of 12% and had a very high mortality rate, indicating toxicity of the composition .

Table 6 shows the results of the microorganism count surviving of treatment solubilized the product of the compound with 20% sludge incorporated into in clayey mass, after the processing of it, burned at 800 ° C.

Table 6. Count of microorganisms surviving the 0.0 / 0.01 / 0.1 / 1 / 10 / e 100 % and with four duplicates of the solubilized of sludge.

<i>Solubilized compost</i>	<i>0 %</i>	<i>0,01 %</i>	<i>0,10 %</i>	<i>1,00 %</i>	<i>10 %</i>	<i>100 %</i>
<i>Population</i>	5	5	5	5	5	5
<i>Survivors after 48 hours</i>	4	5	5	4	4	4
<i>Survivors after 48 hours</i>	5	4	5	4	4	3
<i>Survivors after 48 hours</i>	5	5	4	5	5	4
<i>Survivors after 48 hours</i>	4	4	4	5	4	3

The results shown in Table 6 show that the material solubilized compound with 20% of sludge does not have toxic characteristics. Even when placed the microorganisms directly in solution from solubilized of the clay, it was found that even with a significant difference compared to what happened to the solution of the mode cultivation, the mortality rate does not characterize a toxic waste.

4. CONCLUSIONS

Through these results it is concluded that:

- The chemical composition of the sludge has similarity with the chemical composition of clay;
- The sludge has a greater amount of organic waste, when compared with the clay and this fact directly affects the mass loss due to the action of the firing temperature of the material;
- The sludge generated in the process of decantation and filter press of treatment station effluent from a textile industry, show in the results of toxicity that has toxic characteristics when analyzing the sludge in nature.
- Can verify that the incorporation of ceramic sludge did not change the classification of ceramic compound. Thus, through this analysis of toxicity, it can be stated that the incorporation of sludge in clay in the proportions indicated in this project does not alter the environmental characteristics of red ceramic.
- Through the characterization of raw materials, it was found that it is possible to incorporation of textile sludge in clay for the manufacture of tiles, blocks and solid bricks, when used in the procedures and amounts shown in this work.
- The incorporation of sludge from a sewage treatment plant effluent textile can be a viable approach to mitigate the environmental problems caused by industrial solid waste.

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