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# Potential contribution of water treatment plant (WTP) sludge to river pollution

Potencial contribuição do lodo da estação de tratamento de água (ETA) para a poluição de rios

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This research aimed to characterize the Water Treatment Plant (WTP) sludge from a tropical coastal city in the Brazilian northeast. Parameters physical-chemical (color, pH, turbidity, total and sedimentable solids, chemical oxygen demand and total aluminum), microbiological (thermotolerant coliforms) and ecotoxicological analyses (lettuce seed - *Lactuca sativa*) and the marine microcrustacean (*Mysidopsis juniae*) were analyzed in samples from decanter sludge and washing water from the filters with sludge. As expected, the decanter sludge had a higher amount of solids than the filter, but despite that, the total solids concentration in the filter exceeded 86 times the limit for to be discharge into the water resources, according to Brazilian guidelines. Also, aluminum concentrations were above the guidelines to freshwater (910% - decanter sludge and 210% - filter sludge). Regarding ecotoxicity, sludge from both stages did not affect seed germination and even promoted the growth of 30 to 60% of radicles. On the other hand, the decanter sludge showed to be significative toxic to microcrustaceans in concentrations above than 15%. Considering that these sludges could be released directly into the river in estuarine zone, its potential for toxicity to the aquatic environment is highlighted, and the importance of properly treatment before the discharge in landfills.
Keywords: ecotoxicology, water treatment residual, Poxim river.

Esta pesquisa teve como objetivo caracterizar o lodo de uma Estação de Tratamento de Água (ETA) de uma cidade costeira e tropical do nordeste brasileiro. Os parâmetros físico-químicos (cor, pH, turbidez, sólidos totais e sedimentáveis, demanda química de oxigênio e alumínio total), análises microbiológicas (coliformes termotolerantes) e ecotoxicológicas (semente de alface - *Lactuca sativa*) e do microcrustáceo marinho (*Mysidopsis juniae*) foram analisados em amostras de lodo do decantador e da água de lavagem dos filtros. Como esperado, o lodo do decantador apresentou maior quantidade de sólidos do que o filtro, mas, apesar disso, a concentração total de sólidos no filtro ultrapassou 86 vezes o limite para ser despejado nos corpos hídricos, segundo as diretrizes brasileiras. Além disso, as concentrações de alumínio estavam acima das diretrizes para água doce (910% - lodo do decantador e 210% - lodo do filtro). Em relação à ecotoxicidade, o lodo de ambos os estágios não afetou a germinação das sementes e ainda promoveu o crescimento de 30 a 60% das radículas. Por outro lado, o lodo do decantador mostrou-se significativamente tóxico para microcrustáceos em concentrações acima de 15%. Considerando que esses lodos podem ser lançados diretamente no rio em zona estuarina, destaca-se seu potencial de toxicidade ao meio aquático e a importância do tratamento adequado antes de sua disposição final.
Palavra-chaves: ecotoxicologia, lodo de tratamento de água, rio Poxim.

## 1. INTRODUCTION

Water consumed by man should be free of substances harmful to health. Usually, this water undergoes treatment in order to act as an environmental control measure for the preservation of this health [1], and as one of the by-products of this treatment is generated the sludge [2]. However, most water treatment plants (WTP) deposit their residues in nearby bodies of water [3]. For example, the state of Pernambuco/Brazil has 75% of this sludge released into water bodies, 22% in the soil without any kind of treatment [4], which can compromise the environment with negative impacts.

Residues generated in WTP's are generally products of filter washing water and decanter discharges, containing particles present in the raw water and chemicals used in the treatment process. Then, sludge represents a quantitative and qualitative problem requiring adequate characterization, treatment and alternatives to its final disposal [3, 5], avoiding the discharge directly into water bodies due to the polluting potential. There are few studies on the toxic effects of sludge and the possible risks that improper disposal can cause to soil and water organisms present.

Most of the environmental problems associated with WTP sludge are related with chemicals used in the treatment of raw water, including aluminum sulfate, which is used as a coagulant in the process of flocculating water treatment [6]. According to Banet et al. (2020) [7], the accumulation of aluminum in soil and water compromises the development of aquatic plants since the presence of aluminum acts on the molecular structure of organisms, reducing the absorption of nutrients. The $Al^{3+}$ is the most toxic form of aluminum (Al) [8], causing inhibition of plant root growth [9], especially in tropical and acid soils [10], and is severely toxic [11]. Also, aquatic plants are susceptible to aluminum due to absorption through the roots and the growth rate is reduced in the presence of Al, with root growth being a good indicator of susceptibility for the environment [12].

According to the Brazilian National Environmental Council [13], effluents from any polluting source can only be released into the receiving waterbodies after appropriate treatment, complying the physical-chemical quality standards, including testing ecotoxicological criteria for the evaluation of acute or chronic effects. In this way it is possible to evaluate the potentially harmful effects from that effluents and their constituents. In this view, this research aimed to characterize the physical-chemical, microbial and ecotoxicological aspects of the sludge from the decanters and filters from a WTP localized in a tropical city in the Brazilian northeast (Aracaju, Sergipe, Brazil).

## 2. MATERIAL AND METHODS

### 2.1 Study area

WTP is located in Sergipe/Brazil near to the Poxim River, which runs for approximately 15 km until the Atlantic Ocean. The treatment adopted at the WTP is the conventional. Conventional Water Treatment is a combination of coagulation, sedimentation and filtration and is the most widely applied water treatment technology in Brazil.

The coagulation stage involves adding aluminum sulphate, consequently the larger particles, or flakes, are heavy and quickly settle to the bottom of the water supply. This settling process is called sedimentation. Then, the water goes to the filters, where the solids not removed in the previous stage are retained. The Figure 1 illustrates the basic processes that occur during conventional treatment process in a WTP.

The WTP structure consists of four rectangular decanters with dimensions of 29.6 m length, 10.0 m wide and 4.3 m deep, where the particles sedimentation occurs expressively forming the sludge. The particles that did not have sufficient weight to decant are sent to the outlet of the decanter by the low water velocity, and toward the filters, which perform the rapid descending filtration. The six filters, each with 10.00 m length and 4.95 m, are composed of anthracite layer (upper layer), followed by sand (intermediary layer) and backing layer at the bottom. The decanters are washed twice a month while the filters are washed daily.

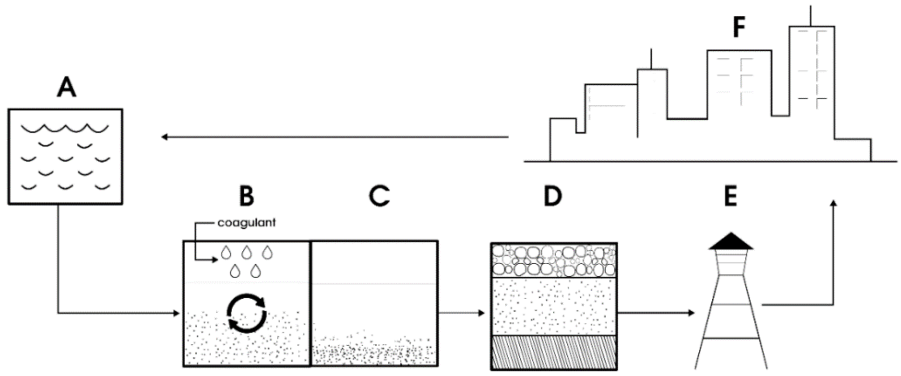


*Figure 1: Water Treatment Plant (Aracaju, Brazil): Raw water supply/Poxim river (A), Coagulation (B), Decanter (C), Filter (D), Water storage (E) and Distribution system (F).*

## 2.2 Sample preparation and characterization

Sludge samples from decanter (stage C) and filter (stage D) were collected manually during the cleaning process of the treatment system, and stored in polypropylene bottles (1L), further subdivided for chemical and microbiological evaluation (500 mL) and ecotoxicological evaluation (500 mL), and cooled at -10ºC. Five samplings were carried out between December 2017 and February 2018. The physical-chemical and microbiological analyses were performed according to the methods described in APHA (2012) [14] (Table 1).

*Table 1: Sludge parameter and methods of analyses according to APHA (2012) [14].*

| Parameter | Method |
|---|---|
| Sedimentable solids | 2540F |
| Total solid | 2540B |
| pH | 4500-H |
| Color | 2120 |
| Turbidity | 2130 |
| Total aluminum | 3111D |
| Chemical Oxygen Demand (COD) | 5220D |
| Thermotolerant coliforms | 9221B |

The samples were sent to the laboratory to be analyzed. The sludge from the decanter exceeded the detection limit of the analyzes and therefore had to be diluted in distilled water.

## 2.3 Ecotoxicity assessments

In order to evaluate the toxicity potential to terrestrial and aquatic systems in the case of disposal this effluent directly in the soil or in the adjacent river (estuarine zone), phytotoxicity analyses were performed on lettuce seeds (*Lactuca sativa*), with samples of the collection 2, 3, 4 and 5 (collection 1 was eliminated for this test, because sample quality was affected due to non-standard storage), and the survival analysis of the marine microcrustacean *Mysidopsis juniae* for collection 4 and 5.

### *2.3.1 Phytotoxicity*

*Lactuca sativa* seeds were bought in agricultural products store. The evaluation of germination seed (%) and radicle growth (mm) after 120 h of exposure were performed according to the method described by USEPA (1996) [15]. Before the exposure, the seeds were placed in distilled water for 20 minutes to break dormancy and facilitate contact with the effluent. Samples from the decanter and the filter were tested without dilution (100% raw sample). Polyethylene Petri plate (90 mm) were coated on filter paper (25 μm pores) and moistened with 2 mL of the sample (in duplicate), and then 10 seeds were placed equidistant among them (n=20). After this, the plates were closed and wrapped in foil to avoid light exposure, and then incubated at 25 °C for 120 h. At the end of this period, the number of germinated seeds (radicles > 5 mm) was measured, the radicle was measured with the aid of the digital caliper. Only seeds considered germinated (radicles> 5 mm) were considered for radicle length analysis. The criterion of acceptability for the assay is the germination of at least 65% of the seeds exposed in the control, which was distilled water [15].

*2.3.2 Survival of microcrustacean*

Mysids (*M. juniae*) used in the experiment were obtained from Laboratory of Ecotoxicological Studies and all procedures for the culture and for the acute toxicity test followed the method described in NBR 15308 [16]. The animals were kept in aquaria (6 L) with artificial seawater prepared from the addition of sea salt (Red Sea®) to the distilled water and adjusted to the salinity 35 ± 2. Daily, the aquaria were cleaned and the organisms fed, and once a month the adult organisms were counted and the pairing maintained (1 male: 3 females), with a total of 60 individuals per aquaria. The temperature was maintained at 25 ± 2°C, with constant aeration and a photoperiod of 12h light/12 h dark. The feeding was made with nauplii of *Artemia* sp. (48 h), and for adults, the nauplii were enriched with cod liver oil and fish oil.

Considering the possibility of disposal the WTP effluent in the Poxim river near to the estuarine area, the sludge samples were salinized (35 ± 2) with the same salt used in the artificial seawater. In a pilot test performed with samples from the collection 3, it was observed that there was no need to dilute the filter sludge sample (100% raw sample). Nevertheless, due to the high toxicity of the raw samples, decanter samples were diluted in artificial seawater (20%, 15%, 10%, 5%, and 2.5%), also used as control (0%). Each concentration was tested in triplicate, and 10 mysids per replicate (1-4 days-old) were added in beakers with 300 mL of each solution, for a period of 96h [16]. The beakers were kept in an incubator with temperature (25 ± 2ºC) and photoperiod (12 h light/12 h dark) controlled, being fed every 24 h with *Artemia* sp. nauplii (48 h), after the counting of the alive individuals. After 96h, the test was finished and the final survival was registered. The physical-chemical parameters pH, dissolved oxygen (OD), salinity and temperature were analyzed at each concentration at the beginning and at the end of the exposure.

### 2.4. Data analysis

The physical-chemical and microbiological analyses were performed from a sub-sample for each collection, and at the end was analyzed from the mean and standard deviation (n = 5). In the phytotoxicity assay data were presented from the mean of the two replicates for each of the four collection analyzed (n = 8 for each concentration). In the acute toxicity tests with mysids, the non-observed effect concentration (NOEC) and lowest observed effect concentration (LOEC) was calculated by comparison between the control and concentrations by means of one-way variance analysis (ANOVA), using Kruskal-Wallis test followed by Dunn's was performed in Graphpad Prism version 5.

## 3. RESULTS AND DISCUSSION

Due to the high solids concentration and viscosity of the decanter sludge (Figure 2), the samples remained homogeneous throughout the test, hence not forming phase between the supernatant and the decanted one. Because of this, the sedimentable solids from the decanter sludge was visibly above the limit. Therefore, it is thoroughly inadvisable to release it in the waterbody without previous treatment since the resolution CONAMA nº430 [13] limits as to a maximum value of sedimentable solids in effluent at 1 mL/L, in the Imhoff cone test.

*Figure 2: Filter sludge (left) and Decanter sludge (right) of the Poxim Water Treatment Plant (Aracaju, Sergipe), during sedimentable solids analysis (after one hour of testing).*

Santos et al. [17] observed that the variation in the amount of total suspended solids in the raw water influences the variation in stake production in the ETA. Souza and Almeida Filho (2017) [18] found values between 32 and 53 mL/L in two WTP analyzed, what means that, in general, WTP effluent has high concentrations of solids. The amount of solids in the sludge is closely linked to the quality water collected from reservoir or river, which may vary seasonally, also dependent of the products types and quantities used in the treatment process, and finally by the interval of filters and decanters cleaning. Considering that the water treatment flow follows from the decanters to the filters, it was possible to observe that the decanter is responsible for the retention of most of the particles, reinforcing the importance of this stage to reduce solids in the water supply.

The concentration of these solids influences the appearance of the sludge as well as its solubility in the water body that will receive this residue. According to Richter (2001) [19], concentrations lower than 5% of solids confer to the sludge with liquid appearance. Nevertheless, the appearance of the sludge of the decanter of Poxim WTP presented dense and viscous appearance. Nevertheless, the values obtained in the filter samples ranged from 0.02 to 0.09%, being quite similar to the raw water from the Poxim river that had 86 to 500 mg/L of total solids that corresponding to up to 0.5% solids [19].

The pH was the variable that had the least variation between the both sludge type (Table 2). According to CONAMA Resolution nº 430 [13], these effluents are within the acceptable range (5 to 9), this means that even though the water received a dosage of disinfection product before passing through the filter, it has not provided change on the pH in this water and consequently in the sludge. Souza and Almeida-Filho (2017) [18], in the study developed with WTP filter water, also obtained pH values within the range cited by the resolution, ranging from 7.07 to 7.85.

As expected in the color variable, the decanter sludge had higher values (98% up) when compared to the filter sludge. As mentioned before, the data variability between the collections was possibly due to the temporal variations of the raw water quality. Michelan et al. (2019) [21] report that rainfall regime may influence the water quality, since the runoff may carry higher organic matter content to the river and consequently more sludge is generated in WTP process. Regarding color and turbidity, the Brazilian legislation does not address limits, however, the values obtained are largely above those presented by Souza and Almeida- Filho (2017) [18] that registered 165 to 3690 uH for the filter sludge and 3560 to 33000 uH for decanter sludge, and for turbidity the results were 285 to 445 uT (filter) and 410 to 2080 uT (decanter) and by Nóbrega et al. (2011) [1] that registered 17 to 469 uH for the filter sludge and for turbidity the results were 4 to 297 uT (filter). At WTP Poxim, the turbidity reduction was observed in the settling process, remaining about 0.6% of the turbidity of the filter sludge.

In Brazil, there is no concentration limit for discharge of effluent with aluminum residue, but the limit of concentration for freshwater is 0.1 mg/L [22]. This indicates that for this residue to be discarded in water bodies, the dilution factor must be 920 and 210, coming from the decanter and filter, respectively.

The total aluminum registered to Poxim WTP sludges was 92 mg/L (decanter) and 21 mg/L (filter) (Table 2). Approximately 23% of the aluminum present in the decanter sludge remained to the filters, emphasizing the substance stayed dissolved in the water and aggregated in the sedimentable particles that were retained in the filter. The coagulant (aluminum sulfate) is dosed according to the quality of the raw water [23].

The concentration of aluminum found in the decanter of Poxim WTP is far above the values obtained in the literature, as demonstrated by Lopes and Serra (2016) [24] who obtained 0.8 mg/L. The presence of aluminum in the sludge was expected given that an aluminum-based compound is used as a chemical coagulant. Although the concentration of aluminum in the water supply has not been quantified, it is important to note that the human individual's exposure to aluminum can cause neurological and physiological damage and possibly associate with the presence of aluminum in the onset of Alzheimer's disease [25].

Regarding Chemical Oxygen Demand (COD), a significant reduction was observed in the filter sludge (Table 2). Again, the values obtained for Poxim WTP were above the values registered by other WTPs as shown by Lopes and Serra (2016) [24] that obtained 5280 mg/L. Compared with COD values from domestic sewage, which in the 450 to 800 mg/L range is classified as a strong sewage [26] and around 292.2 mg/L as a weak sewage [27], it can be stated that the COD of the filters sludge resembles the weak sewage, while the COD of the decanters sludge significantly exceeds the COD range for strong domestic sewage.

In the water treatment process, the last step is the disinfection and removal of microbiological agents, it occurs after the passage to the filter. As shown in Table 2, there is a trend of a higher concentration of thermotolerant coliforms in the decanter sludge (314 ± 128 MPN/100 mL), being practically absent in the filter. The CONAMA Resolution nº 357 [22] limits the presence of 1000 MPN/100 mL for thermotolerant coliforms in freshwater systems (class 2), so, it is unlikely that the sludge from Poxim WTP could contribute to microbiological contamination in the region, including the estuarine area. In studies developed by Di Bernardo et al. (2011) [28] it was observed that the concentration of *Escherichia coli* (*E. coli*) in samples of decanters in the cleaning phase is 16.600 MPN/100 mL, while in the water samples of the filters the lowest concentration of *E. coli* determined was 3 MPN/100 mL.

Concerning the toxicity tests, the mean germination rate in the control was 90 ± 0%, following the criterion of acceptability for the assay [14]. The germination rate for the decanter sludge was 90 ± 13.1% (CV = 14.2%) and 82.1 ± 11.7% (CV = 14,1%) for the filter sludge, indicating that there was no inhibition on germination. The seeds exposed to the control had growth of 26.0 ± 0.6 cm, being observed a growth stimulus in the radicles in the decanter sludge (41.9 ± 5.5 cm, CV = 13.0%) and to the filter sludge (40.7 ± 8.3 cm, CV = 20.5%) exposure. This growth stimulus could be associated to the high levels of organic matter and humic substances in the WTP, evidencing the agricultural potential of this type of effluent [29] or they may have caused symptoms of nutritional deficiency [30]. However, it is necessary to consider the significant levels of aluminum present in the sludge (Table 2) that may compromise the plant development such as stem, leaves, and fruits [31]. Still, it is possible that this sludge can be disposed of in a controlled way in the soil [32], but it is important to study the toxic potential interaction with other metals in soil to all phases of plants development.

*Table 2: Physical-chemical and microbiological variables of sludge samples from the decanter and the filter of the Poxim river water treatment plant (Aracaju, Sergipe, Brazil).*

| Variables | Collection | Decanter | Filter | Standards of launch effluents* |
|---|---|---|---|---|
| **Sedimentable solids (mL/L)** | 1 | n.d. | 102.0 | |
| | 2 | n.d. | 90.0 | |
| | 3 | n.d. | 115.0 | 1.0 |
| | 4 | n.d. | 90.0 | |
| | 5 | n.d. | 40.0 | |
| | **mean ± SD** | **-** | **87.4 ±28.4** | |
| **Total solid (%)** | 1 | 2.48 | 0.03 | |
| | 2 | 10.5 | 0.04 | |
| | 3 | 7.13 | 0.09 | - |
| | 4 | 4.02 | 0.02 | |
| | 5 | 4.85 | 0.06 | |
| | **mean ± SD** | **5.80 ± 3.12** | **0.05 ± 0.03** | |
| **pH** | 1 | 7.60 | 7.12 | |
| | 2 | 8.18 | 7.11 | |
| | 3 | 7.72 | 7.68 | 5 - 9 |
| | 4 | 7.14 | 6.45 | |
| | 5 | 8.05 | 7.73 | |
| | **mean ± SD** | **7.74 ±0.41** | **7.22 ±0.52** | |
| **Color (uH)** | 1 | 142400 | 3360 | |
| | 2 | 232000 | 3950 | |
| | 3 | 123500 | 4120 | - |
| | 4 | 163000 | 2840 | |
| | 5 | 94400 | 1990 | |
| | **mean ± SD** | **151060 ±51813** | **3252 ± 868** | |
| **Turbidity (uT)** | 1 | 12400 | 98 | |
| | 2 | 166000 | 400 | |
| | 3 | 11000 | 250 | - |
| | 4 | 17500 | 396 | |
| | 5 | 11120 | 150 | |
| | **mean ± SD** | **43604 ± 68472** | **258 ± 138** | |
| **Aluminum (mg/L)** | 1 | 83.0 | 20.2 | |
| | 2 | 126.6 | 26.5 | |
| | 3 | 67.3 | 0.2 | - |
| | 4 | 97.4 | 33.3 | |
| | 5 | 85.3 | 24.0 | |
| | **mean ± SD** | **91.9 ± 22.1** | **20.8 ± 12.5** | |
| **COD (mg/L)** | 1 | 19195 | 219 | |
| | 2 | 20595 | 243 | |
| | 3 | 18180 | 219 | 120 |
| | 4 | 16973 | 226 | |
| | 5 | 13592 | 183 | |
| | **mean ± SD** | **17707 ± 2657** | **218.0 ±21.9** | |
| **Thermotolerant coliforms (MPN/100mL)** | 1 | 200 | < 1.8 | |
| | 2 | 520 | < 1.8 | |
| | 3 | 220 | < 1.8 | - |
| | 4 | 340 | < 1.8 | |
| | 5 | 290 | < 1.8 | |
| | **mean ± SD** | **314 ±128** | **< 1.8** | |

n.d.: not determined; SD: standard deviation; MPN: most probable number; <1.8 MPN/100 mL/L: detectable minimum limit.
* Brazilian standards of launch effluents defined by the CONAMA [13].

In the tests carried out to evaluate the toxic potential of WTP sludge to Poxim river adjacent to the estuarine area, it was observed that the filter sludge did not cause microcrustaceans mortality. On the other hand, the decanter sludge showed to be highly toxic, with LOEC of 20% and 15% in collections 4 and 5, respectively (Figure 3).

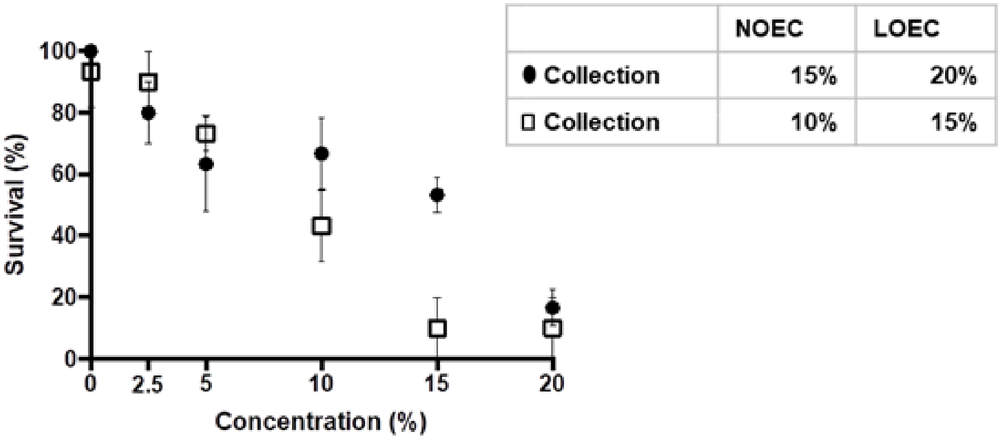


*Figure 3: Survival (%) of the microcrustacean* Mysidopsis juniae *(n = 30) exposed to the decanter sludge from the Water Treatment Plant (Aracaju, Sergipe, Brazil). NOEC is the non-observed effect concentration and LOEC is the lowest observed effect concentration.*

Studies in the Poxim estuarine region have already identified low environmental quality for both water and sediment samples [33], and WTP Poxim effluents may be contributing to the environmental degradation of this estuary.

## 4. CONCLUSION

The results obtained in this study call attention to the potential impact caused by the dispose of the sludge from the Poxim WTP, mainly to decanter sludge, which has high concentrations of pollutant chemicals that can compromise the quality of the water sources in the neighborhood.

It was observed that the residue from WTP has elevated concentration of aluminum, however in Brazil there is no legislation regulating the disposal of aluminum in water or soil. However, it was also registered that the sludge does not seem to cause harmful effects on germination and primary growth of the plant, but further evaluations are needed regarding the effect of high aluminum content on plant organisms. On the other hand, the sludge from the decanter can cause a severe decrease in the survival of marine microcrustaceans. The chemical and physical characterization of samples indicated that it cannot be released in an aquatic environment without previous treatment.

## 5. ACKNOWLEDGMENTS

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