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Physico-Chemical and Microbiological Properties of the Sewage Sludge Produced by the Sewage Treatment Plant (STP) of Toledo - Paraná - Brazil

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ABSTRACT

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This work proposes to assess the microbiological and physico-chemical characteristics and to quantify the heavy metals of the biosolids produced by the sewage treatment plant - STE, of the city of Toledo - State of Paraná - Brazil. The conclusion of this analysis is that the sewage sludge produced by the city's Sewage Treatment Plant can be categorized as a Type A sludge because it has low rates of total and thermotolerant coliforms. The physico-chemical characteristics of the dry sludge and the levels of heavy metals found proved to be an excellent indicator for the use of this material in agricultural activities.

Introduction

Agricultural recycling has brought benefits to both man and nature. As such, recycling can transform a simple residue into an important agricultural input, which provides nutrients and organic matter to the soil. Major advantages have been observed with the use of sludge in agriculture, namely, a reduction in CO₂ emissions caused by incineration, less need for chemical fertilizers and an increase in the organic matter content of the soil

(Andraus *et al.*, 1997). In a larger-scale analysis, it was also found that the addition of sludge to the soil is in line with global policies for the preservation of the biosphere since it reduced the atmospheric emissions of CO₂ (Andrade and Sarno, 1990; Feitosa, 2009).

When the sludge is applied in agriculture, it benefits the physical properties of the soil,

such as the formation of aggregates of soil particles, which in turn increase the size of empty spaces, directly improving infiltration and water retention and also enabling a greater capacity of aeration (Lee, 2011).

The decomposition of sewage sludge (Figure 1), when applied to the soil, produces complexing agents that have the ability to solubilize unavailable forms of phosphorus present in the soil (Andreoli *et al.*, 1994), in addition to providing for the slow release of the compounds in the sludge.

For the recycling of sewage sludge to have a desired efficiency, it must comply with the appropriate planning and monitoring program, in which the following factors, among others, must be taken into account: required adaptations and environmental monitoring of the sewage treatment plants, sanitization alternatives, suitability of the areas of application and distribution operation, production estimates, and quality assessments. This work seeks to ensure that the sludge has a good quality to be used in agriculture (Bastos and Mara, 1993; Bettiol and Fernandes, 2004).

Sewage sludge is considered a waste of high agricultural value, but it has some pollutants in its composition that may contaminate the environment and be harmful to humans. In its decanting step, sewage treatment has the effect of concentrating heavy metals, complex organic compounds and pathogens. When the sludge is used in agriculture in a non-controlled fashion, this can contribute to the contamination of the soil, plants, groundwater and surface water through surface runoff, leaching and absorption processes (Fesp, 1991; Sanepar, 1997).

Excess nutrients can be mobilized chemically in the soil or even reach the surface waters and cause their eutrophication. The

uncontrolled application can reduce the productivity of the soil or impair the quality of agricultural products for human or animal use. The application of sewage sludge with low stabilization favors the release of odors, attracting vector insect to the product (Fiest *et al.*, 1998).

These potential impacts are usually generated by a lack of information, by the incorrect interpretation of many growers and consumers regarding the agricultural use of sludge and, mainly, by the prejudice that leads the consumer to not use the sludge. Such attitudes lead to the generation of environmental liabilities and the contamination of other locations (Gasi and Rossin, 1993; Bettiol and Fernandes, 2004).

The sludge generated in sewage treatment usually has a high concentration of organic matter, phosphorus, nitrogen and micronutrients. When this residue is treated with lime, it has the characteristic of correcting soil acidity, because when the sludge is subjected to the process of alkaline stabilization, its pH becomes basic. Nitrogen is the main component of sewage sludge. As such, one could say that this element is the reference to the limitation of its application rates (Bettiol and Camargo, 2000; Andreoli *et al.*, 2006).

In Japan and in some European countries, the leaching of nitrogen is monitored through the nitrate form found in the environment. In Paraná, for example, the volume of sludge tends to be controlled in direct relation to the element nitrogen when the capacity for nutrient assimilation is concerned (Imhoff and Imhoff, 1986; Miki *et al.*, 2006).

Heavy metals may have a higher concentration in the sludge than in the soil. It is therefore possible to see that the use of sludge as fertilizer should be controlled. The

management practices for sewage sludge in the soil must take into account the heavy metals concentrations in the residue, always monitoring the maximum cumulative levels allowed in the soil, the amount already accumulated, the regional conditions of the soil, the climatic conditions and the topography (Andreoli *et al.*, 1994; Andreoli, 1998; Andreoli, 1999; Berton, 2000; Van Haandel and Alem, 2006; Correia, 2009; Miranda, 2010).

The association of risks with heavy metals that are in the soil due to the application of biosolids depends on several original soil factors, such as: texture, type of clay, organic matter, pH, cation exchange capacity and weathering intensity (Berton, 2000; Correia, 2009; Miranda, 2010).

The standard applied by the State of Paraná is based on those the Environmental Protection Agency of the United States uses to control heavy metals and ensure both environmental and human safety. They establish a maximum limit of 50 dry tons per hectare over a time period of 10 years. Obviously, this system requires a constant monitoring of the accumulation of these metals in the soil (Sanepar, 1997).

Studies carried out with the sludge from the Belém sewage treatment station showed that even when doubling the dosage, i.e. 100 (t/ha), there was no significant increase in the concentrations of elements in the soil resulting from the application of the sewage sludge. Experiments performed with higher doses in a percolation column showed that the heavy metals remain on the surface of the soil, penetrating until approximately 2.5 cm of depth, with zinc being the only element to achieve the maximum percolation depth of 10cm when considering soils with pH 4 (Kamogawa *et al.*, 1997, Fiest *et al.*, 1998).

The objective of the monitoring is to demonstrate and evaluate the environmental compliance of the entire operational process. To this end, the establishment of criteria, methods and strategies for the evaluation of the contamination generated by the application of sewage sludge in agriculture, are required. Law no. 12,493 of 22/01/1999 states that every company that generates residue should be liable for the problems caused both to the environment and health by the generated waste (Roque, 1997).

In general, the processes that cover recycling should be monitored regularly in order to minimize operational failures. The effects of the sewage sludge on the soil and the quality of the biosolid produced are two parameters of great importance that should be evaluated constantly (Sanepar, 1997; Van Haandel and Alem, 2006).

The operating permit must be obtained from the environmental agency, i.e. the sanitation company concerned should submit the documentation of its treatment plant, the operating license and the distribution plan. The registration documentation should also be presented with the following requirements: registry information, characterization of the sewage treatment station, characterization of the disinfection system, sludge management area, characterization of the sludge, general description of the area of application, characterization of the soil capacity, technical description of the final disposal process in the agricultural soil (Simon and Tedesco, 1993; Simoneti, 2006).

This work proposes to assess the microbiological and physico-chemical characteristics and to quantify the heavy metals of the biosolids produced by the sewage treatment plant - STE, of the city of Toledo – Paraná – Brasil, is that this work is carried out.

Materials and Methods

The sludge used in this study was collected from the Sewage Treatment Plant (STE) of the City of Toledo, in the western region of the State of Paraná, Brazil, located on the geographic coordinates: 24° 43' 53" , South and 53° 45' 55" , West and at an altitude of 576 meters (Figure 2).

The pH was determined by weighing 10g of the sample and passing it to a 100mL beaker, adding distilled and sterilized water, maintaining it in rest for four hours to make the determination in a potentiometer with a properly calibrated electrode.

In the microbiological analyses of total and thermotolerant coliforms (*Escherichia coli*), the multiple tubes technique according to the Standard Methods was used. The presence of *Salmonella* was determined according to the procedure described in the manual of methods for microbiological and parasitological analysis in the agricultural recycling of sewage sludge (Bonnet, 1998) and according to the Cestesb L5 218 standard (1993).

The evaluated parameters were: C (g.kg^{-1}); C:N ratio (g.kg^{-1}); N-NH₄ (mg.kg^{-1}); N-NO₃ (mg.kg^{-1}); total P (%); total S (%); P (%); K (%) and Mg (%). All these parameters were determined through the methods recommended by the APHA, AWWA, WPCF (1998).

The following heavy metals were determined: cadmium, copper, nickel, lead and zinc. All metals were determined through atomic absorption spectrometry with an air-acetylene flame, after dissolution in a microwave oven under pressure, using the EPA-3051 method (USEPA 1985, 1994): Dissolution of 0.5 g of the sample in 10 mL of concentrated HNO₃ for 10 minutes at 175 °C and 200 Psig.

Results and Discussion

The results obtained for the characterization of the sludge from the sewage treatment plant of the city of Toledo - Paraná - Brazil, are summarized in Tables 1, 2 and 3.

By evaluating table 1, one can see that the pH of the sludge assessed is in the range of 7.56 ± 1.06 , with this being considered normal for this type of material. The content of organic C in the evaluated matter stayed at $152 \pm 15.89 \text{ g.kg}^{-1}$ of matter under study. These results are in agreement with those obtained by The Ros *et al.*, (1993).

Nitrogen is present in sewage in a variety of forms because of its various states of oxidation, and because it changes quickly from one state to another depending on the physical and biochemical conditions. Ammonia may be present as molecular ammonia, NH₃, or as ammonium ion, NH₄. The balance between these two forms in water is strongly dependent on the pH and temperature (Bettiol *et al.*, 2006). In this work, $1,546 \pm 245.06 \text{ mg.kg}^{-1}$ in the form of ammonium ion, NH₄, and $536 \pm 123.98 \text{ mg.kg}^{-1}$ in the form of NO₃, were detected.

Nitrogen is an essential element for plant growth and the living beings in the soil. The proper use of the sludge should be aimed at the efficient use of nitrogen, with a minimum loss through percolation, volatilization, denitrification and surface erosion. With the decomposition of sludge added to the soil, the organic nitrogen is converted into ammonium or nitrate. Soil colloids may retain the ammonium, but nitrate will usually be leached out of the root zone because the capacity of soils to retain it is low.

In reducing conditions, on the other hand, denitrification may occur, a process by which the nitrogen from the nitrate is transformed

into gaseous nitrogen. Another fundamental issue is the balance of this nitrogen (Lira *et al.*, 2008).

The organic matter of the sludge applied to the soil undergoes mineralization, releasing nitrogen in the ammoniacal and nitrate form, which are added to those existing before the application. As such, the amount of sludge applied must be such that the amount of nitrate or ammonium present does not exceed the amount the plant will use, since the excess would be easily-leachable material that could reach and contaminate underground bodies of water (Melo and Marques, 2000). This is perhaps one of the most important elements for the monitoring of areas where sewage sludge is used. The levels of total P (%); total S (%); P (%); K (%) and Mg (%), were 8 ± 1.22 , 1.23 ± 0.45 , 1.34 ± 0.23 , 165 ± 32.78 and 5.21 ± 1.26 , respectively.

Excess phosphorus, sulfur and potassium pose practically no risk to the plants because toxicities of these element are hardly ever detected, and, additionally, our phosphorous deficient soils retain it with great energy. The contamination of groundwater by this element is therefore very difficult. Precautions must be taken, however, because the surface erosion of the solid material may drag the retained phosphorus with it. In certain situations, it may then be released in the bodies of surface water to which the material flowed (Berton, 2000).

Brazilian legislation adopts limits for heavy metals and also provides for the need to respect the limits of heavy metal accumulation through the control of metal concentrations in the soil. The levels of heavy metals detected in sewage sludge from the STE in the city of Toledo can be observed in Table 2. The values detected are within acceptable levels according to Brazilian legislation and also according to the levels

recommended by the Usepa (1985) and Usepa (1994).

The heavy metals that are present in the sewage sludge have been the object of many studies because of the impact these elements have on both human and animal health, in addition to the quality of the food, which must not be forgotten (Viel, 1994).

Heavy metals can accumulate in the soil for a long time. The sludge has the following metals in its composition: Cd, Co, Cr, Cu, Fe, Hg, Mn, Mo, Pb, Sn and Zn. Some among these are considered essential for plants and animals, such as Cu, Mo and Zn, while others are toxic, Cd, Hg and Pb (Sanepar, 1997; Pires and Andrade, 2006).

The sludge generated in the treatment of urban sewage generally has a low concentration of heavy metals. If industrial sewage enters the same sewage treatment, along with rainwater, then there is a significant increase of these elements (Andreoli, 1999).

The absorption of nutrients by plants and the toxicity of heavy metals are greatly dependent on the chemical species present. According to Roque, (1997) the ionic species is the one with the highest rate of absorption. This can be explained by the fact that only the free metals, such as Cd^{2+} , have the ability to cross the plasmalema and enter the cytoplasm of the cells. Complexed metal ions with inorganic ligands or chelated with organic ligands in the soil solution, on the other hand, cannot be absorbed directly, but need to be broken from the ligands by an exchange process (Soccol and Paulino, 2000).

The plants absorb the free cations in solution because the root cells have a negative potential along the cell membrane, favoring the absorption of cationic species. Complexed

cations (Silva *et al.*, 2000), on the other hand, have smaller positive charges or even negative charges. Another factor that inhibits the absorption of complex cations is the increase in the volume of the species.

The sludge contains the most varied pathogenic micro-organisms. However, the mere presence of the infectious agent in the sludge used in agriculture doesn't necessarily mean the immediate transmission of diseases. It only characterizes a potential risk. The levels of pathogenic micro-organisms detected in the sewage sludge from the STE in the city of Toledo - Paraná - Brazil, can be observed in Table 3.

The real risk of an individual being infected depends, in fact, on the combination of a series of factors, such as: the resistance of the pathogens to the sewage treatment and environmental conditions; the infective dose; the pathogenicity; the susceptibility and degree of host immunity; the degree of human exposure to the transmission foci. Before a micro-organism present in an effluent used in agriculture can cause a disease, therefore, it would first have to resist the sewage treatment

processes employed and survive in the environment in sufficient number to infect a susceptible individual (Bastos, 1993).

Before application in areas of agricultural use, the sludge must be properly treated in order to ensure the reduction of pathogens. Sludge treated through methods approved by the environmental agencies and showing densities of thermotolerant coliforms below 10^3 MPN/g TS (Most Probable Number per gram of Total Solids) are considered to be a class "A" sludge (Usepa, 1985; Usepa, 1994).

A class "A" sludge is characterized by having thermotolerant coliforms <1000 MPN/g dry solids, or less than three *Salmonella* sp. per four grams of solids. The authors don't recommend the use of the *Salmonella* test instead of the coliform test. On the one hand, it is less accurate, , and, on the other, the chance of finding three *Salmonella* bacteria in four grams of sludge is much smaller than that of finding 1000 coliforms in a gram. Considering this classification, the sewage sludge generated by the STE of the city of Toledo - Paraná, is classified as type "A" (Melo and Marques, 2000).

Table.1 Physical-chemical characteristics of the sewage sludge (dried basis) coming from the treatment plant in Toledo - PR - including the macronutrient content. Means followed by the standard deviation

Analyzed Parameters	Mean Concentrations
pH	7,56±1,06
C (g.Kg ⁻¹)	152±15,89
C:N ratio (g.Kg ⁻¹)	10:2
N-NH ₄ (mg.Kg ⁻¹)	1,546±245,06
N-NO ₃ (mg.Kg ⁻¹)	536±123,98
Total P (%)	8±1,22
Total S (%)	1.23±0,45
K (%)	1.34±0,23
Ca (%)	165±32,78
Mg (%)	5.21±1,26

Table.2 Heavy metal content in the sewage sludge (dried basis) from the treatment plant in Toledo - PR. Means followed by the standard deviation

Elements	Mean obtained*	Acceptable levels*
Cd	5.05±1,07	85
Cr	68.78±12,54	3000
Cu	123.45±23,21	4300
Ni	89.76±10,67	420
Pb	143.05±24,39	840
Zn	1256.09±200,89	7500

Source: (USEPA, 1995) - In mg of pollutant per kg of sludge (dried basis).

Table.3 Determination of total and thermotolerant coliforms (*Escherichia coli*) and Salmonella in the samples used in the experiment

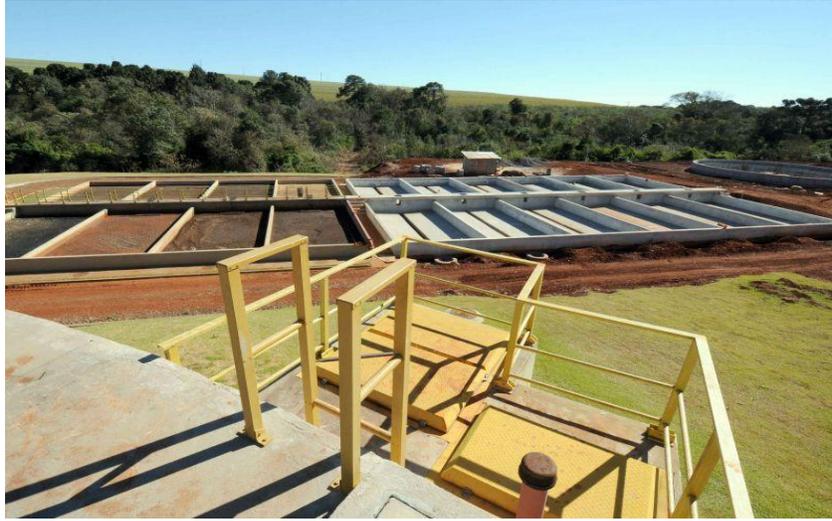
Micro-organisms	Indices obtained*
Total Coliforms	$6,3 \times 10^3$
Thermotolerant Coliforms	$3,2 \times 10^2$
Salmonella	1.2×10

* Data provided in MPN.mg⁻¹

Figure.1 Drying bed of the sewage sludge generated in the sewage treatment station



Figure.2 Location of the Sewage Treatment Plant (STE) of the City of Toledo - Paraná – Brazil



From the perspective of the agricultural recycling of sewage sludge, a purely microbiological criterion does not seem to satisfy a broader analysis of the potential process risks for human or environmental contamination. A question that remains open to discussion is linked to the maximum levels of survival that could be tolerated in the sludge without putting public health at risk, considering all environmental aspects.

After performing this study within the aforementioned conditions, the conclusion can be drawn that the sewage sludge produced by the Sewage Treatment Plant of the city of Toledo, State of Paraná, Brazil, can be categorized as a Type A sludge because it has low rates of total and thermotolerant coliforms.

The physico-chemical characteristics of the sludge on a dried basis and the levels of heavy metals found can be an excellent indicator for agricultural recycling, but more data is needed on the dynamics of these components in the applied soil.

Conflicts of interest

The authors declare there are no ethical, publishing of financial conflicts of interest regarding the data of this study.

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