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Characterization of the Primary Sludge from Pharmaceutical Industry Effluents and Final Disposition

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Abstract: The generation of large volumes of waste by industrial processes has become an object of study because of the necessity to characterize the composition of residues in order to suggest appropriate treatments and to minimize adverse environmental impacts. We performed analyses of total fixed and volatile solids, moisture, and chemical oxygen demand (COD). We found high organic matter content. We also measured physicochemical characteristics, including corrosivity, reactivity, and toxicity. Sewage sludge showed levels of chloride and sodium above the maximum allowed limits. These data suggest the potential for anaerobic digestion as a treatment option for sewage sludge and for its use as a biofertilizer.

Keywords: sewage sludge; characterization; toxicity; biofuel; biofertilizer

1. Introduction

In an effluent treatment station (ETS), wastewater is usually treated by physical routes such as flotation and sedimentation, chemical pathways such as flocculation, and by biological processes such as microbial treatment [1]. According to Ji et al. [2], anaerobic biological treatment is a preferred option for the treatment of wastewater with pharmaceutical products, considering its advantages, including withstanding high organic loads, lower sludge production, and lower operating costs than conventional activated sludge process.

After effluent treatment, the sludge obtained and organic residue decanted both during and at the end of the process should be treated. Generally, three types of sludge are produced: primary, secondary, and tertiary. Sludge can undergo processes of densification, stabilization, conditioning, dehydration or dewatering, purification, and final disposal. Generally, sludge is destined for landfills; however, there are several studies reporting the use of sludge as a potential source of biofertilizer for agricultural systems as well as biofuel production. This is a recurring concern of industries, with respect to achieving environmental sustainability. Liu et al. [3], for example, optimized biogas production through the anaerobic co-digestion of sludge with low organic content and food residues.

Sludge produced in an ETS contains nutrients, especially nitrogen, phosphorus, and carbon, and, in smaller amounts, potassium, calcium, magnesium, organic matter, heavy metals, pathogens, and other elements from tributaries. These may pose health risks and environment hazards if they are not properly monitored and controlled [4]. The concentration of substances from pharmaceutical residues varies among regions and their properties significantly affect treatment performance [5].

The need for characterization prior to treatment has been increasingly carried by ETSs. Laboreaux, in the city of Itabira, Minas Gerais, Brazil, showed that pressurized filtered sludge, if subjected to drying to improve heat output, can be used as a fuel to supply consumers in the region, replacing coal or firewood [6]. Another study characterized the physicochemical properties of oily residues from sanitation, and only from this study was it proposed that the use of these residues may serve as a substrate for the production of biofuels [7].

It is imperative to carry out studies on the composition and characterization of solid waste in order to propose appropriate treatment, minimizing environmental impacts, and to optimize the resources in ETS equipment. The characteristics of sludge directly affect the processes of treatment and final disposal; therefore, understanding of its characteristics is fundamental to defining residue management [8].

Knowledge of the effluent generated by an industry is essential to the determination of treatment processes performed by the ETS that aims to accord with legislation, in order to dispose of it safely into the environment and ecosystem, as well as to reuse it, guaranteeing environmental sustainability.

The objective of this work was to characterize the physicochemical properties of primary sludge of a pharmaceutical industry ETS, classifying it in terms of its potential risks according to current legislation, as well as its final disposal.

2. Experiment Part

The ETS of the pharmaceutical industry operates an activated sludge system with prolonged aeration for the treatment of human and industrial sewage. Sludge from human sewage, after decantation, flows from the system to dehydrate in a drying bed. Subsequently, it is collected and sent to landfills legally licensed for this purpose. Industrial sewage is sent directly to final stabilization treatment and is not subjected to initial treatment.

2.1. Collection of Primary Sludge Samples

ETS sludge samples were collected in December, January, February, and March, in a drying bed that constantly received fresh sludge (Figure 1A). Every month, small samples were collected, including drying; after collection, the sludge was homogenized (Figure 1B). The samples were stored in plastic buckets, individually capped, and analyses were conducted immediately after collection. Standard collection and analysis techniques were maintained every month (December, January, February, and March).

Figure 1. (**A**) Drying bed with dry primary sewage sludge. (**B**) Drying bed with primary sewage sludge after discharge.

2.2. Physicochemical Characterization of Samples

The analyses of total solids (TS), total fixed solids (TFS), total volatile solids (TVS), and determination of moisture and chemical oxygen demand (COD) were carried out at the Anaerobic Biodigestion Laboratory of the Institute of Agrarian Sciences of the Federal University of Minas Gerais (ICA/UFMG), Montes Claros Regional Campus. We measured the percentage value of total solids (TS), total fixed solids (TFS) and total volatile solids (TVS), moisture determination, and chemical oxygen demand (COD) in December, January, February, and March. After data collection, arithmetic mean and descriptive statistical analysis of the values were performed to characterize these criteria, related to the sludge studied. For analysis of TS, TFS, and TVS, the gravimetric method was used (2540 solids) [9]; for COD analysis, the colorimetric method was used, according to the Standard Methods for Examination of Water and Wastewater, 20th ed. [9]. Moisture content was determined by subtraction of the total solids, by Equation (1).

$$Moisture(100\ \%) = 100 - total\ solids\ content\ (1) \tag{1}$$

For toxicity determinations, the following parameters were analyzed: Leaching toxicity classification—inorganic parameters; leaching toxicity classification—organic parameters (pesticides); leaching toxicity classification—organic parameters (other organic); classification of toxicity by solubilization—inorganic parameters (chromatography); classification of toxicity by solubilization—organic parameters (chromatography) and classification of macro- and micronutrients. For these analyses, a single sample was homogenized, starting from samples collected in the months of December, January, February, and March. The analyses were performed according to the standards described by the United States Environmental Protection Agency—USEPA [10] and Standard Methods for Examination of Water and Wastewater, 20th [9].

3. Results and Discussion

After descriptive analysis of the data, we obtained mean values of TS (25.05%), TFS (7.52%), and TVS (17.40%). The largest fraction of TS was represented by TVS, indicating a high biodegradable fraction of this residue. This result was compatible with that of Oliveira et al. [7], who performed chemical physical characterization of oily residues from sanitation waste and observed that the largest fraction of total solids was represented by volatile solids. Cucina et al. [11], when evaluating the risks and benefits associated with the agricultural use of three different organic fertilizers derived from a pharmaceutical manufacturing effluent, found values of 14.30% and 8.80% for total solids and volatile solids, respectively.

In relation to the moisture content of the sludge, a value of 74.95% was found. In a study carried out by Silva [12], when evaluating the characteristics of sludge produced at the Brasília Norte Sewage Treatment Plant (Brasília - DF), there was a moisture content of 83%. Moisture variation correlated with environmental factors. In December, February, and March, this occurred on the day of collection and over the subsequent days, as opposed to the meteorological situation in January when there was no precipitation [13].

Regarding COD, the mean value obtained was 267.333 mg L^{-1}. This value was higher when compared to COD measurements reported in the literature. According to some studies, COD values from effluents of the pharmaceutical industry vary between 4.410 and 40.000 mg L^{-1} [14,15]. The high value observed for sewage sludge can be explained by the high concentration of organic material that is dumped in the effluent of the pharmaceutical industry, requiring a large amount of oxygen to oxidize and stabilize this organic material.

Tables 1–3 display the results of the leaching toxicity analyses, including the amounts of heavy metals and pesticides in the waste that is indispensable for evaluation for agricultural use, in addition to the evaluation of the risk heavy metals pose to the environment.

Table 1. Classification of leached residue toxicity for inorganic parameters according to the Standard Methods for Examination of Water and Wastewater, 20th ed. [9], including maximum permitted value, limit of detection and the concentration of leached residue in the leached residue.

Parameter	Maximum Permitted Value (mg L^{-1})	Limit of Detection (mg L^{-1})	Concentration in Leached Residue (mg L^{-1})
Arsenic	1.0	0.01	<0.01
Barium	70.0	0.02	<0.02
Cadmium	0.5	0.001	<0.001
Lead	1.0	0.01	<0.01
Total chromium	5.0	0.01	<0.01
Fluorides	150.0	0.10	<0.10
Mercury	0.1	0.0031	<0.002
Silver	5.0	0.01	<0.01
Selenium	1.0	0.01	<0.01

Table 2. Classification of leached residues for organic parameters (pesticide), according to the United States Environmental Protection Agency—USEPA [10]; maximum permitted value, limit of detection, and the concentration in the leached residue.

Parameter	Maximum Permitted Value (mg L^{-1})	Limit of Detection (mg L^{-1})	Concentration in Leached Residue (mg L^{-1})
Aldrin and dieldrin	0.003	0.001	<0.001
Chlordane (all isomers)	0.02	0.001	<0.001
Dichlorodiphenyltrichloroethane (all isomers)	0.2	0.001	<0.001
Pentachlorophenol	0.9	0.01	<0.01
2,4- dichlorophenoxyacetic acid	3.0	0.01	<0.01
Endrin	0.06	0.001	<0.001
Heptachloro and Heptachloro Epoxide	0.003	0.001	<0.001
Lindane	0.2	0.001	<0.001
Methoxychlor	2.0	0.001	<0.001
Toxaphene	0.5	0.002	<0.002
2,4,5- Trichlorophenol	0.2	0.002	<0.002
2,4,5-trichlorophenoxypropionic acid	1.0	0.01	<0.01

Table 3. Classification of leached residues—organic parameters (other organics) according to the United States Environmental Protection Agency—USEPA [10], maximum permitted value, limit of detection, and the concentration value in the sewage sludge analyzed.

Parameter	Maximum Permitted Value (mg L^{-1})	Limit of Detection (mg L^{-1})	Concentration in Leached Residue (mg L^{-1})
Benzene	0.5	0.04	<0.04
Benzo(a)pyrene	0.07	0.002	<0.002
Vinyl chloride	0.5	0.04	<0.04
Chlorobenzene	100	0.01	<0.01
Chloroform	6.0	0.04	<0.04
o-Cresol	200	0.01	<0.01
m-Cresol	200	0.01	<0.01
p-Cresol	200	0.01	<0.01
1,4-Dichlorobenzene	7.5	0.04	<0.04
1,2-Dichloroethane	1.0	0.04	<0.04
1,2-Dichloroethylene	3.0	0.04	<0.04
2,4-Dinitrotoluene	0.13	0.01	<0.01
Hexachlorobenzene	0.1	0.001	<0.001
Hexachlorobutadiene	0.5	0.04	<0.04
Hexachloroethane	3.0	0.01	<0.01
Methyl ethyl ketone	200	0.5	<0.5
Nitrobenzene	2.0	0.01	<0.01
Pyridine	5.0	0.01	<0.01
Carbon tetrachloride	0.2	0.04	<0.04
Tetrachlorethylene	4.0	0.04	<0.04
Trichlorethylene	7.0	0.04	<0.04
2,4,5-Trichlorophenol	400	0.01	<0.01
2,4,6-Trichlorophenol	20	0.01	<0.01

According to the results presented in Table 1, the concentration of inorganic compounds in the leached residue agrees with the established limits. Therefore, the primary sludge from pharmaceutical industry effluents cannot be considered toxic in terms of this parameter.

Table 2 shows that the concentration of organic compounds (pesticides) agree with the established limits. Therefore, primary sludge from pharmaceutical industry effluents can not be considered toxic with respect to this parameter.

According to the results presented in Table 3, the concentration of organic compounds (other organic) also agrees with the established limits. Therefore, the primary sludge from pharmaceutical industry effluents cannot be considered toxic with respect to this parameter.

According to Turki et al. [16], cited by Rigobello et al. [17], the organic compounds that are present in the residues can leach into groundwater and surface water and, because of the difficulty of their removal by conventional methods such as coagulation, sedimentation, flocculation, and filtration, they can be found in water intended for human consumption. These same authors, Turki et al. [16], studying landfill leachate samples before and after treatment with Fenton reagent, identified aromatic acids (1,2-diphenylcyclobutane, 4-phenyl cyclohexane, and 1,2-benzenedicarboxylic acid), phenolic compounds (2,4-bisphenol, 4-methyl phenol), aliphatic acid and ester (bis(2-methoxyethyl)ester), phthalate ester - alcohols (2-chlorocyclohexanol and cholestanol), and polyaromatic hydrocarbons (anthracene and naphthalene). All of the aforementioned compounds are considered mutagenic or carcinogenic [18], except for cholestanol, a natural sterol that may be associated with natural sources of organic matter of terrigenous or marine origin [19].

Although the evaluation of organic pollutants in sludge from wastewater treatment plants is a practice already established in some countries [20], there have been no studies related to the identification of these compounds in primary sludge of effluents from the pharmaceutical industry in Brazil.

Tables 4 and 5 display the results of the solubility toxicity analyses, including heavy metals and pesticides in waste, the understanding of which is indispensable for agricultural purposes, in addition to the fact that heavy metals pose a risk to the environment.

Table 4. Classification of solubility toxicity—inorganic parameters, measured by chromatography: maximum permitted value, limit of detection, and concentration of solubility in the sample, according to the Standard Methods for Examination of Water and Wastewater, 20th ed. [9].

Parameter	Maximum Permitted Value (mg L^{-1})	Limit of Detection (mg L^{-1})	Concentration in the Residual Solubilized (mg L^{-1})
Arsenic (mg As L^{-1})	0.01	0.01	<0.01
Barium (mg Ba L^{-1})	0.7	0.02	<0.02
Cadmium (mg Cd L^{-1})	0.005	0.001	<0.001
Lead (mg Pb L^{-1})	0.01	0.01	<0.01
Cyanide (mg CN L^{-1})	0.07	0.01	0.03
Total chromium (mg Cr L^{-1})	0.05	0.01	<0.01
Fluorides (mg F L^{-1})	1.5	0.10	<0.10
Mercury (mg Hg L^{-1})	0.001	0.0002	<0.0002
Nitrate (mg N L^{-1})	10.0	0.05	0.49
Silver (mg Ag L^{-1})	0.05	0.01	<0.01
Selenium (mg Al L^{-1})	0.01	0.01	<0.01
Aluminum (mg Al L^{-1})	0.2	0.05	<0.05
Chloride (mg Cl L^{-1})	250	2.0	437
Copper (mg Cu L^{-1})	2.0	0.009	<0.009
Iron (mg Fe L^{-1})	0.3	0.1	0.12
Manganese (mg M L^{-1})	0.1	0.05	<0.05
Sodium (mg Na L^{-1})	200	0.8	444
Sulfate (mg $SO_4{}^{-2}$ L^{-1})	250	1.00	62.0
Zinc (mg Zn L^{-1})	5.0	0.1	<0.10
Surfactants (mg MBAS L^{-1})	0.5	0.10	0.18
Total Phenols (mg C_2H_5OH L^{-1})	0.01	0.001	<0.001

Table 5. Classification of solubility toxicity—organic parameters, measured by chromatography: maximum permitted value, limit of detection, and concentration of the solubility in the sample, according to the United States Environmental Protection Agency—USEPA [10].

Parameter	Maximum Permitted Value (mg L^{-1})	Limit of Detection (mg L^{-1})	Concentration in the Residual Solubilized Sample (mg L^{-1})
Aldrin and dieldrin	3.0×10^{-5}	2.0×10^{-5}	$<2.0 \times 10^{-5}$
Chlordane (all isomers)	2.0×10^{-4}	2.0×10^{-4}	<0.0002
2,4–D	0.03	1.0×10^{-2}	<0.01
DDT (all isomers)	2.0×10^{-3}	1.0×10^{-3}	<0.001
Endrin	6.0×10^{-4}	2.0×10^{-4}	<0.0002
Heptachlor and its epoxide	3.0×10^{-3}	2.0×10^{-5}	$<2.0 \times 10^{-5}$
Hexachlorobenzene	1.0×10^{-3}	1.0×10^{-3}	<0.001
Lindane	2.0×10^{-3}	1.0×10^{-3}	<0.001
Methoxychlor	0.02	1.0×10^{-3}	<0.001
Toxaphene	5.0×10^{-3}	2.0×10^{-3}	<0.002
2,4,5-T	2.0×10^{-3}	2.0×10^{-3}	<0.002
2,4,5-TP	0.03	1.0×10^{-2}	<0.01

Within the classification of solubility toxicity for inorganic parameters (Table 4), the primary sludge of effluents from the pharmaceutical industry presented levels of chloride and sodium above the maximum limits allowed.

According to Lakhdar et al. [21], organic matter controls the effects of salt on soils. Therefore, high levels of chloride and sodium (Table 4) in the sludge would be controlled by the high content of organic matter, indicated by the high volatile solids content and the COD that are indirect measures of the organic matter content of the substrate.

According to Daliakopoulos et al. [22], cited by Cucina et al. [11], the content thereof should be highlighted when organic fertilizers of pharmaceutical origin are applied to soils to avoid salination and/or negative effects, for example, the structure of the soil, colloidal dispersion, or inhibition of plant growth.

According to the results presented in Table 5, within the classification of toxicity by solubilization for organic parameters, the primary sludge of pharmaceutical industry effluents presented levels within the maximum allowed limits.

Table 6 shows the results for the analysis of macro- and micronutrients that are indispensable for disposal of soil residues.

Table 6. Classification of macro- and micronutrients of effluent treatment station (ETS) primary sewage sludge: method of analysis, according to the Standard Methods for Examination of Water and Wastewater, 20th ed. [9] and maximum permitted concentration in Brazil [23].

Assay	Results	Maximum Concentration Permitted in Brazil [23]
Calcium	4.10 mg kg^{-1}	-
Total organic carbon	25.20%	-
Copper	252.30 mg kg^{-1}	1500 mg kg^{-1}
Total sulfur	5.22%	-
Total phosphorus	10.20 mg kg^{-1}	-
Magnesium	1091.60 mg kg^{-1}	-
Molybdenum	0.281 mg kg^{-1}	50 mg kg^{-1}
Nickel	47.50 mg kg^{-1}	420 mg kg^{-1}
Nitrite	1.02 mg kg^{-1}	-
Ammoniacal nitrogen	78.88 mg kg^{-1}	-
Kjeldahl nitrogen	0.84%	-
Potassium	1384.8 mg kg^{-1}	-
Selenium	<0.002 mg kg^{-1}	100 mg kg^{-1}

Sewage sludge is a source of important nutrients. According to Teixeira et al. [24], cited by Botero et al. [25], in the flocculation and decantation stage, a residue rich in organic matter, micro- and macronutrients is generated. Its use can reduce the environmental impact associated with inadequate disposal of such waste.

According to Correia [26], the percentage of carbon present in the dry mass of sewage sludge ranges from 18% to 50%. The value of 25.2% (Table 6) found for the sludge accords with already recorded data. In addition, we observed that the concentrations of copper, molybdenum, nickel, and selenium were below the maximum allowed values [23]. On the other hand, the concentrations found for other compounds (macro- and micronutrients) disagreed with established values, and there should be no absence of these elements [23].

The use of sewage sludge for agricultural use is regulated by law as prescribed by resolution number 375/2006 of the National Council for the Environment in CONAMA [23], establishing criteria and procedures for agricultural use of sewage sludge generated in sewage treatment plants. The Technical Report on Solid Waste Classification of the Brazilian Association of Technical Standards ABNT NBR 10004 establishes a classification for solid wastes [27], divided into two classes that can be classified as Class I, considered to be hazardous, and Class II, considered to be non-hazardous. The second classification is further divided into inert (B) and non-inert (A).

ETS sludge from the pharmaceutical industry, according to the ABNT NBR 10004 [27] Solid Waste Classification Technical Report, is classified as Class II A (non-hazardous, non-inert). It is also neither corrosive nor reactive [27]. The toxicity evaluated by the parameters analyzed, namely, leaching toxicity classification—inorganic parameters, leaching toxicity classification—organic parameters (pesticides), leaching toxicity classification—organic parameters (other organic), classification of toxicity by solubilization—inorganic parameters (chromatography), and classification of toxicity by solubilization—organic parameters (chromatography), presented results according to specifications [27].

According to Cieslik et al. [8], one of the methods of stabilization of sewage sludge is anaerobic biodigestion with biogas production. The products of this process can be used in agriculture and in the generation of energy, contributing to the sustainable development of small and large facilities. Anaerobic biodigestion (degradation of biodegradable organic matter by microorganisms in the absence of oxygen gas) is a common effective method for the stabilization of sludge, concomitant with the production of biogas. The various methods of performing anaerobic biodigestion should be challenged to optimize the achievement of the desired product [28]. The generation of energy from biogas must take into account certain criteria to reach maximum efficiency, including the raw materials and energy demands (input) [29].

Matteo et al. [30] achieved satisfactory results, studying environmentally sustainable solutions, including biogas production and energy conversion. Based on the results presented here, it can be inferred that the treatment of sewage sludge is a promising alternative for the production of biofertilizer and biofuels.

4. Conclusions

Given the current environmental problems generated by the high volume of solid waste, this study contributes data to better understanding the constitution of sewage sludge from the pharmaceutical industry, in order to implement viable alternative measures. Primary sludge from the pharmaceutical industry presents levels of inorganic and organic compounds that are below the specified maximum levels, except for those of chloride and sodium, in addition to containing a high content of organic matter that contributes to the control of salt levels in soil. According to the Technical Report on Solid Waste Classification ABNT NBR 10004: 2004, the primary sludge is classified within Class II A (non-hazardous—non-inert). It also contains important micro- and macronutrients, contributing to the production of biofertilizer from the residues in question, in addition presenting itself as an attractive alternative for the production of biofuels, through anaerobic biodigestion. This is an economically viable and environmentally sustainable proposal for the reuse of solid waste and alternative energy production.

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