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Evaluation of the pH of solid residues generated in the cellulose industry adequate to the sludge hygienization for use in agriculture

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This paper looks into the possibility of using solid residues generated in the cellulose production process to hygienize sewage sludge for fertilization. During the cellulose fabrication process, dregs, grits and vegetable biomass ashes are generated. Seven experiments were conducted in the Hydraulic Research Institute´s laboratory at UFRGS, in order to determine the pH values of the three residues in analysis-ashes, dregs, grits and the combinations of sludge/ash, sludge/grits and sludge/dregs- at the beginning (IT) and at the end (after two hours-FT). The preparation of the dregs, grits and ash solutions consisted of the weighing of 20 g of each residue and its dissolution in 80 ml of water, thus obtaining the solutions of grits, dregs and ashes. The pH values of the samples were analyzed using bench pH-meter. The ashes, grits, the mixture sludge/ash and sludge/grits displayed an accentuated basic character due to their pH values which were ≥12. The dregs and the mixture sludge/dregs presented a less accentuated basic character than the ashes and the grits, with a pH equal to 9. The results show that the ashes and the grits can be used in the hygienization of sewage sludge.

Key words: Sludge, ashes, grits, dregs, hygienization, pH.

INTRODUCTION

In the paper and cellulose production process big quantities of residues are generated which need to be adequately disposed of. The main residues are dregs, grits, lime mud and biomass ashes. According to Almeida (2008), dregs are alkaline solid by-products composed of very small particles; their main constituents being carbonates, hydroxides and sulphides and, above all, Na and Ca. Grits are granular, yellow, odourless, alkaline solid residues that come from the lime and limestone mud calcinations process in the lime ovens (Nolasco et al., 2000). The biomass ashes are accumulated through the combustion of the vegetable biomass for the generation of heat and energy, containing a variety of macro and micronutrients resistant to incineration (Knapp

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and Insam, 2011). Among the various forms of final disposal of the sludge resulting from sewage treatment, the disposition on soil has been gaining popularity because of its richness in organic matter and nitrogen (Guerra et al., 2006). However, because the sludge comes from the sewers, it has some undesirable features such as the existence of pathogens, heavy metals and organic contaminants so it need to be stabilized/ hygienized before it is deposited on the soil (Prosab, 1999).

The destruction of pathogenic organisms is achieved through their exposure to conditions considered adverse to their survival, which include high temperatures, bactericidal effect of the Sun´s UV rays, aerobic conditions with high levels of O_2; reduction of the substrate, competition, predation by the zooplankton, high pH, presence of compounds toxic to certain bacteria or; combination of many of these factors (Andrade Neto, 1997).

Chemical stabilization consists of the addition of products that may inhibit biological activity or oxidise organic matter (Fernandes and Souza, 2001).

The most common chemical treatment is the alkaline in which a base, normally lime, is mixed with sludge, increasing its pH and destroying most of the pathogenic microorganisms. In order to achieve the intended goal, lime should be added to the sludge until its pH reaches 12 (Fernandes and Souza, 2001).

During the chemical stabilization, a series of reactions between sewage sludge and applied components occur. Thus, the chemical stabilization of sewage sludge is a process that converts sludge into a product appropriate for fertilization (Meurer, 2012). According to Von Sperling and Goncalves (2001), hygienization consists of the removal of pathogenic organisms.

The chemical features of wood ashes make them excellent material for the correction of the soil acidity, source of nutrients for tree plantations and, also constitutes a way of returning to nature what is collected as vegetable biomass, keeping or correcting the soil´s fertility and sustainability of the ecosystem (Bellote et al., 1994; Guerrini et al., 1994; Vance, 1996).

This experiment intends to evaluate the pH of three residues, namely, vegetable ash biomass, grit and dregs which are generated during the cellulose fabrication process, to hygienize sewage sludge for soil fertilization.

Sludge characteristics

The sludge under analysis is the digested anaerobic, from the sewage treatment at Serraria Treatment Facility located on the southern region of Porto Alegre, Ipanema, which is operated by the Municipal Water and Sewage Department. (DMAE in Portuguese). The Sewage Treatment Facility, (ETE in Portuguese), uses centrifuges to dehydrate the sludge and, to facilitate the separation between solids and liquids, it uses the cationic polymer, polyacrimalide, (PAM). The samples were collected from the containers, after dehydration with 80% humidity between June and November 2017. ETE generates around 50 m^3 of sludge which is deposited at a sanitary landfill (DMAE, 2014).

Location of the factory generating the residues

The three residues under analysis come from cellulose production facility at Veracel Company, operating in both forest and industrial areas, which is in Bahia, in the southern region of Brazil. Its plantations cover 5 municipalities, namely, Eunápolis, Canavieiros, Belmonte, Porto Seguro and Santa Cruz Cabrália. In the forestry area, the company´s cycle goes through the following stages: use of Technologies to produce eucalyptus clones, seedlings´ production, soil preparation, planting, fertilization, harvest and transportation to the factory. The processing begins with the reception of certified timber, harvested from the eucalyptus forests. After physical and chemical processes, the timber is transformed into cellulose.

Cellulose production method

Veracel uses the Kraft method in its cellulose production process. The choice of this method is due to its efficiency and the fact that it is the most common in the cellulose industry. According to Amaral (2008), one of the main features of the Kraft method is the retrieval of chemical products and its stages are: cleaning, in order to achieve complete separation of mass from leach with the least dissolution possible; the evaporation of water from the leach until it reaches a combustible concentration; burning of the leach followed by the dissolution of the merged products; caustification- conversion of sodium carbonate into hydroxide.

According to the company, the facility generates about 1.960 tons/month of dregs/grits and 270 tons of ash. It is important to mention that, at Veracel, grits and dregs are mixed and considered a single type of residue. The process comprises the following stages.

Timber preparation (debarking)

The timber comes to the mill in the form of logs from whence it is sent to the mechanic debarkers, if it is not debarked yet. The bark reduces the cellulose yields besides increasing the amount of impurities.

Figure 1 to 3 present simplified diagrams of a timber preparation yard, formation and treatment of wood chips and liquors´ retrieval, respectively.

Chipping and chip classification

After debarking, the logs are sent to the chippers where

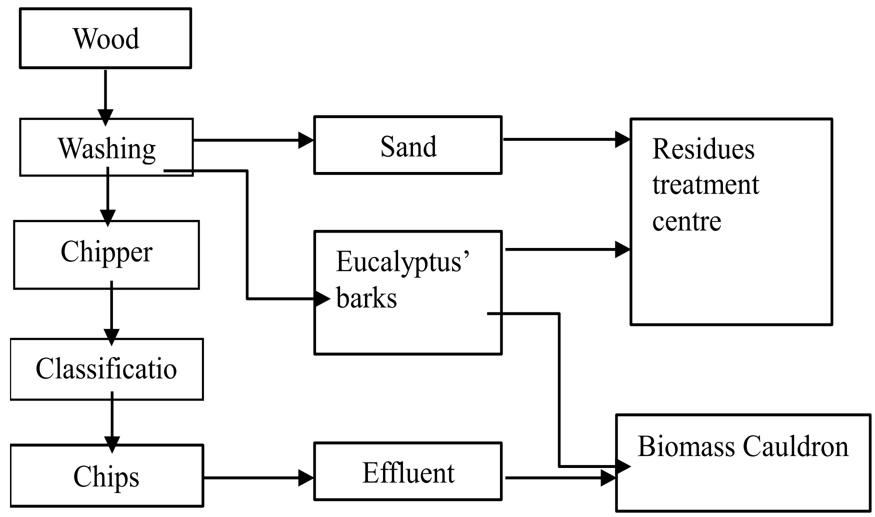


Figure 1. Simplified flow chart of the timber yard.

they are transformed into chips with appropriate dimensions for pulping (Guerra et al., 2006). This stage is meant to transform the logs into fragments small enough to allow the penetration of the pulping liquor used in the chemical processes. Figure 2 presents the chips´ formation and treatment process.

Dark liquor retrieval

The dark liquor's retrieval cycle and the alkaline´s energy processes reduce the loss of chemicals and minimize the production costs. The dark liquor´s heat generates enough energy to power the cellulose production plant. The main stages of the chemical retrieval process are: Evaporation of the dark liquor, incineration of the dark liquor in the retrieval cauldron, caustification and lime regeneration.

Chemical composition of the residues

As mentioned previously, in the cellulose and paper production process, large quantities of residues are generated which need to be adequately disposed of. The main residues are dregs, grits, lime mud and biomass. In this study, the pH of those residues was evaluated, except for lime mud. The residues are described in detail as follows.

Dregs

The limestone oxides originating from the process, organic matter, sulphur compounds, sodium and magnesium are the dregs' components. The metals present in the dregs may originate from wearing out of the equipment used in the process and the raw materials used for cellulose extraction (Maeda et al., 2010).

Grits

Calcium oxides, magnesium and potassium synthesized from lime mud are the main components of grits. Metallic content is greater than that of dregs. The next table presents the composition of grits.

Ashes

The ashes from the vegetable biomass are referred to as fertilizers due their significant basic cations content (Norstrom et al., 2012). However, they are primarily considered an acidity corrective material due to their high levels of oxides, hydroxides and calcium carbonates, although the magnesium hydroxides and carbonates, potassium and phosphorus are equally important (Haraldsen et al., 2011). Thus, the vegetable biomass ashes can be used as an acidity corrector, as well as sources of calcium, magnesium and potassium. Ferreiro et al. (2011) and Arshad et al. (2012) reported that soil acidity was the major limitation to food production worldwide and advocated the use of forest ashes as a corrective in acid soils. The main concern in relation to the use of forest biomass ashes as a fertilizer is the high concentration of potentially toxic elements, including heavy metals which may increase their concentration in the soil.

The composition of the ashes varies, according to the material used in and the intensity of the burning process. In the case of the forest biomass, the typical composition

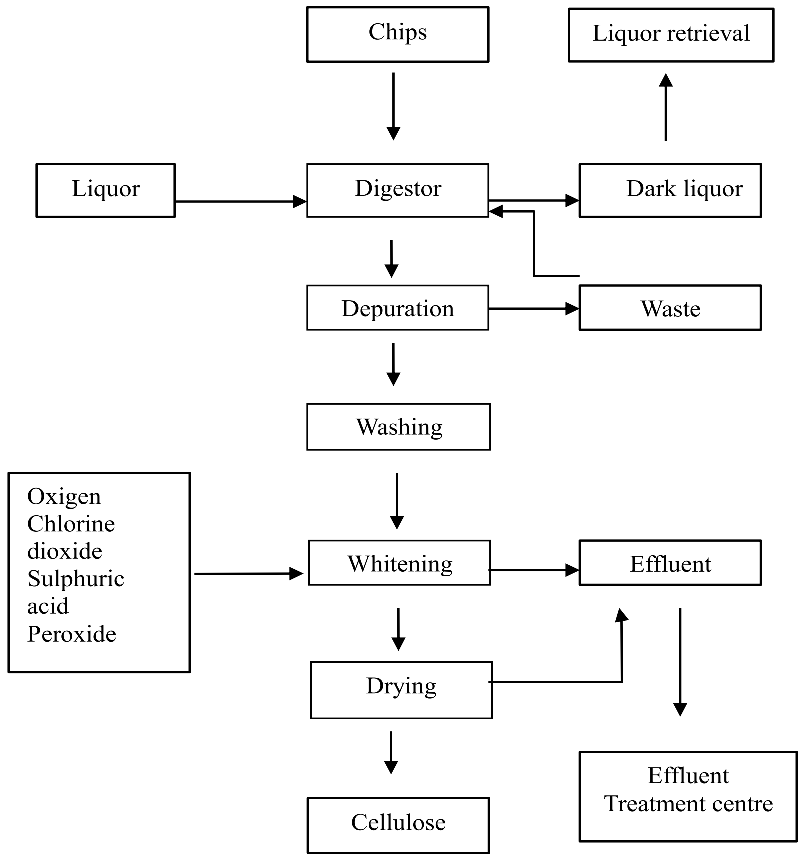


Figure 2. Chips´ formation and treatment flow chart.

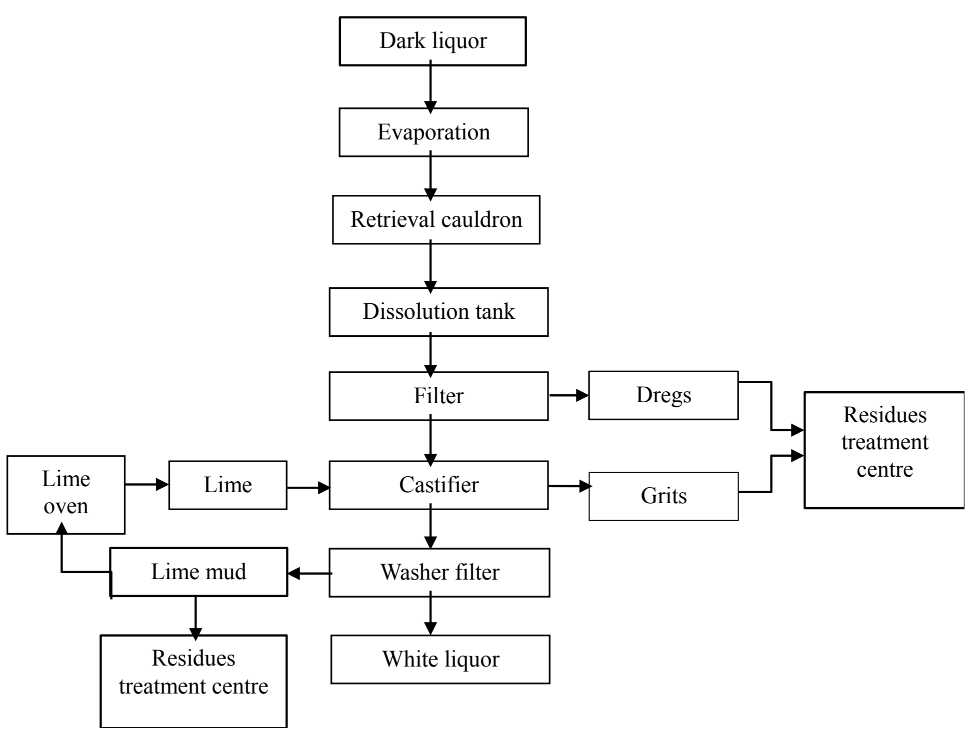


Figure 3. Simplified liquor retrieval flow chart.

is as shown in Table 3.

MATERIALS AND METHODS

Location of the study

The study was conducted in Porto Alegre, Brazil. The Municipality of Porto Alegre comprises 496.7 km^2 and a population of 1.476.867 inhabitants (IBGE, 2015). It is located at an altitude of 22 m; Latitude: 30° 1' 40" South; Longitude: 51° 13' 43" West.

The climate is subtropical humid; there are four seasons, though, owing to its being located along a transitional zone, weather tends to vary frequently.

The annual average temperature is 19.5°C; between 10 and 25°C in Autumn (March-June); Between 2 and 20ºC in winter (June to September); between 15 and 30°C in Spring (September to December) and varies between 25 and 35°C in Summer (December-March). (Porto Alegre Municipal Government-Tourism, available online through www2.portoalegre.rs.gov.br/turismo/default.php?p_ secao=260). Seven experiments were conducted at the Institute of Hydraulic.

Research, Federal University of Rio Grande do Sul, in order to determine the pH values of the three residues under analysis - ashes, dregs, grits and the mixtures sludge/ash, sludge/grits and sludge/dregs, at the beginning, (IT) and at the end (after two hours-FT) and the results are shown in Table 4. For the solutions of pure ash, dregs and grits, readings were taken only once, since their pH does not change over time. However, the readings of the combinations, were taken twice, at the beginning, (IT) and at the end, (FT), with a time lapse of 2 h.

Preparation of the dregs, grits and ashes solutions consists of the weighing of 20 g of each followed by its dissolution in 80 ml of water. Each experiment was conducted using sludge from a different batch; however, the three residues used for the pH monitoring came from the same batch.

After the preparation of the solutions, the following step consisted in the determination of the pH values for each one of them. For the mixtures of sludge/ash, sludge/grits and sludge/dregs, 150 g of sludge were weighed with 80% humidity, the equivalent to 30 g of dry sludge and the initial pH was determined. Afterwards, the 150 g of sludge with 80% humidity were mixed with100 ml of each previously prepared residue solutions, separately. Finally, the pH of each combination was determined.

Using a bench pH-meter, the pH values of the samples of digested sludge, ashes, dregs, grits and the combinations sludge/ash, sludge/dregs and sludge/grits, were determined.

RESULTS AND DISCUSSION

Table 4 presents the pH values obtained from the three residues and the three combinations, in all the 7 experiments undertaken. Based on the pH results presented in Table 4, a second Table 5, was designed indicating the maximum, medium and minimum values for each parameter resulting from the seven experiments undertaken.

The graph bellow showing just the medium/average pH values is based on the results displayed in Table 5. From the data presented by Graph 1, the following can be noted: In relation to the digested sludge, (LD), the minimum, maxim and medium pH values show that it has a neutral pH and that no products with basic (chemical) properties were added during the dewatering process. These readings are considered excellent and typical for anaerobicaly digested sludge. A study by Fernandes and Silva (1999), that analyzed the efficiency of ETE Belém's sludge disinfection processes for agricultural use, came up with digested sludge with 7.0 pH.

In relation to the ashes, (C), the results displayed in Table 5, show that they have an accentuated basic character because their pH readings are ≥ 12. According to Table 3 data, the ashes have a high potassium content if compared to the grits and dregs. While analyzing the quality of vegetable ash for use as fertilizers in the Curitiba metropolitan area, Darolt and Osaki (1991) concluded that vegetable ashes, which were rarely used as soil fertilizer, contained calcium, magnesium, phosphorus and other elements. Diniz and Beig (1987) used ash in red/yelow latosoil, sandy phase, in doses oscillating between 1 and 4 t/ha, and obtained excellent results Maeda and Bognola (2013), while studying the chemical properties of soil treated with residues from the cellulose and paper industry, concluded that pH and Ca, Mg and P contents increased as the tested doses also increased while the AL content and its saturation decreased with application of all the tested materials, mainly wood ash. According to Erich and Ohno (1992), Etiegni and Campbell (1991) and Dahl et al. (2009), the forest ashes' ability to neutralize the soil's acidity depends mainly on the quantity of oxides, hydroxides and magnesium carbonates, potassium and limestone available.

Grits (G) - still based on the results of Table 5; it is noticeable that grits' pH values are similar to those of the ashes, which are ≥ 12. Such high pH values are due to their chemical composition, that is, the existence of compounds with accentuated alkaline behaviour, according to the data in Table 2. Chemically, the grits are 53% calcium oxide, which is an important ingredient for soil stabilization (Machado et al., 2003). Destefani et al. (2010) characterized and evaluated the grits form the cellulose industry and obtained the following composition. 96.80% CaO; 1.49% SO2; 1.37% K_2O; 0.22% SrO and 0.11% Fe_2O_3.

1. Dregs (D) - behaved differently from the ashes and the grits in respect to the minimum, medium and maximum pH values. Based on the results in Table, the dregs' medium pH value was 9.82. From this reading, it is evident that the dregs are residues with a basic behaviour, although less accentuated if compared to the ashes' and the grits'. The pH values' difference between dregs, dregs and ashes stems from their chemical compositions. As seen in Table 1, the dregs' calcium oxide content is 35%, less than the grits 53%. Medeiros (2008) analyzing the possibility of correcting the acidity of humid, aluminic cambisol soil, obtained the following chemical composition of dregs: Mg = 9 g/kg; Ca =354 g/kg; Na = 10 g/kg; pH= 10.7; neutralization capacity80%.

Table 1. Presents the chemical properties of the dregs

Parameter	Value	Parameter	Value
CaO-%	35.7	Pb- mg.kg^{-1}	50
MgO-%	3.62	Cu- mg.kg^{-1}	100
SO_3^{-2}-%	1.6	As- mg.kg^{-1}	3
N-NH_3-%	<0.00	Fe- mg.kg^{-1}	4.800
N-total-%	<0.01	Mg- mg.kg^{-1}	16.000
C(fixo a 105°C)-%	20.8	Mn- mg.kg^{-1}	5.800
Compost. de sódio-%	4.7	Ni- mg.kg^{-1}	100
Metals-%	3.2	Ti- mg.kg^{-1}	500
Silicatos-%	30.4	Zn- mg.kg^{-1}	40
Sb- mg.kg^{-1}	20	Al- mg.kg^{-1}	4.800
Cd- mg.kg^{-1}	5	PN-%	72

Table 2. Chemical Properties of grits

Parameter	Value	Parameter	Value
CaO -%	53	Al- mg.kg^{-1}	3100
MgO -%	1.83	Sb- mg.kg^{-1}	20
K_2O-%	1.2	Cd- mg.kg^{-1}	50
$SO_3^{-2-\%}$	0.7	Pb- mg.kg^{-1}	50
OH^{-n}-%	0.4	Cu-mg.kg^{-1}	20
Sílica Solúvel-%	0.4	As- mg.kg^{-1}	2
N-NH_3 %	<0.005	Fe- mg.kg^{-1}	1600
N-total	<0.01	Mg- mg.kg^{-1}	2800
Compost. de sódio %	0.13	Mn- mg.kg^{-1}	200
Metais-%	0.84	Ni- mg.kg^{-1}	40
Silicatos (balanço)-%	41.5	Ti- mg.kg^{-1}	500
PN-%	100	Zn- mg.kg^{-1}	10

Table 3. Chemical properties of forest biomass ashes

Parameter	Value	Parameter	Value
SiO_2-%	18-25	Mn-%	0.5
CaO-%	25-35	K_2O-%	10-15
MgO-%	6-7		
Fe-%	3-5		
Al_2O_3-%	2-4	Densidade-kg.m^{-3}	193
P_2O_3-%	1.6-3.4	PRNT-%	25
Na_2O-%	0.5-0.9		

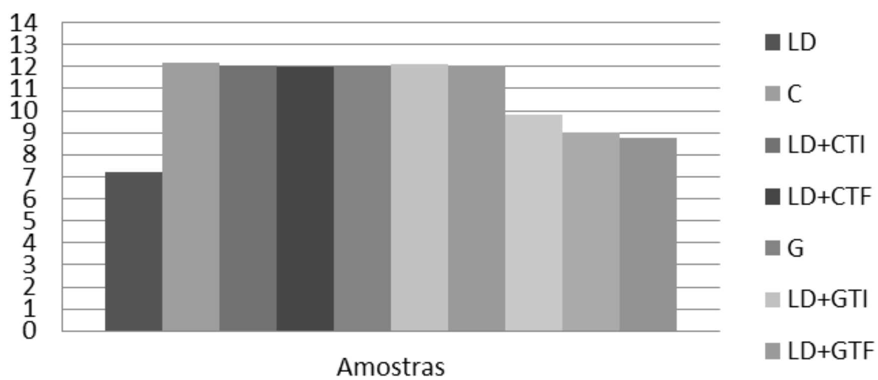
2. Sludge/ashes: This mixture's pH medium values were 12.05 at the beginning, (IT) and 11.98 at the end, (FT) as shown in Table 5. These readings showed that the mixture can obtain an excellent pH value for the sludge hygienization and stabilization process. According to Pinto (2001) chemical sludge hygienization mechanism uses an alkalinizing product to increase the sludge's pH to ≥ 12 values for, at least, 2 h.

3. Sludge/grits: Similarly to the sludge/ashes mixture, the sludge/grits mixture also presented excellent pH values, that is, ≥ 12. When the sewage sludge's pH increases, the cellular protoplasm's colloidal nature changes, resulting in an uninhabitable environment, lethal to the pathogens. Microbial activity takes place in environments

Table 4. pH values obtained during the experiments.

Sample	Digested sludge	Ashes	Grit	Dreg	Sludge/ashes		Sludge/grit		Sludge/dreg	
					TI	TF	TI	TF	TI	TF
E1	7.22	12.10	12.01	9.90	12.05	11.85	12.04	12.06	8.84	8.43
E2	7.27	12.07	11.97	9.62	12.01	11.99	12.20	11.98	8.30	8.22
E3	7.20	12.17	11.99	9.69	12.01	11.99	12.00	11.98	8.44	8.21
E4	7.23	12.21	11.92	9.75	11.95	11.85	12.14	12.08	9.55	9.42
E5	7.32	12.20	12.04	9.83	12.02	11.98	12.09	12.00	9.40	9.20
E6	7.18	12.52	12.13	10.23	12.25	12.22	12.08	12.04	9.76	9.35
E7	7.10	12.06	12.07	9.70	12.03	11.98	12.09	12.07	8.90	8.73

E-Experiment; IT-Initial time; FT-Final time.



Graph 1. Medium pH values. LD, digested sludge; C, ashes; G-grits, D-dregs; CTI, ashes initial time; CTF, ashes final time; GTI, grits initial time; GTF, grits final time; DTI, dregs initial time; DTF, dregs final time.

Table 5. Minimum, maximum and medium pH values from the 7 experiments.

Simple	pH value					
	Minimum		Maximum		Medium	
Digested sludge	7.10		7.32		7.22	
ashes	12.06		12.52		12.19	
Grits	11.92		12.13		12.02	
Dregs	9.62		10.23		9.82	
Mixtures	**TI**	**TF**	**TI**	**TF**	**TI**	**TF**
sludge/ashes	11.95	11.85	12.25	12.22	12.05	11.98
sludge/*grits*	12.00	11.98	12.20	12.08	12.09	12.03
sludge/*dregs*	8.30	8.21	9.76	9.76	9.03	8.79

between 6.5 and 9.0 pH, so pH is the primary issue in reducing microorganisms in sludge (Pinto, 2001). High pH does not just eliminate bacteria it also prevents the movement of heavy metals in the soil (Junior et al., 2001).

4. Sludge/dregs: This mixture's medium pH was 8.79, a lower figure if compared to the readings obtained from sludge/ash and sludge/grits mixtures. The dregs' low pH value might be because of the fact that in dregs, most of the cations are bound to the carbonate group.

Conclusion

The solid residues generated in the cellulose industry– ashes, grits and dregs – are predominantly basic in their chemical composition. The ashes and grits can be used to hygienize sewage sludge for use on agricultural soils. These two residues, ashes and grits, have a pH level equal or superior to 12 and when mixed with sludge they maintain this figure for two hours. Thus, the hygienization of sludge using vegetable biomass ashes is a viable alternative as, besides eliminating pathogens, there is an increase in potassium levels, which does not occur when sludge is hygienized using lime. The product resulting from the combination of sewage sludge, ashes and grits can be considered a biosolid as it contains the necessary nutrients to improve soil fertility.

CONFLICT OF INTERESTS

The authors, Saidelamine Abibe Mahadal and Gino Roberto Gehling declare that there will be no conflict of interest within the scope of this article because it is their own and that all sources consulted were cited within the article.

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