

TREATMENT OF BOTTOM SLUDGE FROM FACULTATIVE LAGOONS WITH STABILIZATION IN LABORATORY CONDITIONS

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ABSTRACT

In this paper we show the results obtained from the chemical stabilization process of pond sludge by adding common hydrated lime. Doses of lime at 5%, 7%, 9%, 10%, 11%, 12%, and 15% were added in controlled laboratory conditions to a sample of sludge taken from the bottom of the primary facultative pond of the wastewater treatment system in Montería in Colombia. The sludge's dangerousness (corrosiveness, flammability, and reactivity) and concentrations of sulfides, heavy metals (As, Ba, Cd, Cr, Hg, Ag, Pb, Se), coliforms, *Salmonella*, mesophilic bacteria, *Ascaris lumbricoides*, and other helminths were analyzed in the sludge sample before and after application of an optimal 10% dose. The reactivity due to sulfides and cyanides in the sludge sample was controlled with this dose, but coliforms and mesophilic bacteria concentrations did not decrease. Some heavy metals (Cd, Cr, Hg, Ag, Pb) increased after application of the hydrated lime dose, although the concentrations found do not pose environmental danger according to existing environmental regulations.

KEYWORDS: Sludges; Hydrated lime; pH; Heavy metal; Reactivity; Coliforms.

TRATAMIENTO DE LODOS DE FONDO DE LAGUNAS FACULTATIVAS CON ESTABILIZACIÓN EN CONDICIONES DE LABORATORIO

RESUMEN

En este artículo se muestran los resultados obtenidos en el proceso de estabilización química de lodos de lagunas de estabilización, mediante la adición de cal hidratada común. Se adicionaron en condiciones controladas de laboratorio, dosis de cal al 5 %, 7 %, 9 %, 10 %, 11 %, 12 % y 15 % a una muestra de lodos de fondo de la laguna primaria del sistema de tratamiento de aguas residuales de la ciudad de Montería, Colombia. Se analizaron la peligrosidad del lodo (corrosividad, inflamabilidad y reactividad) y las concentraciones de sulfuros, metales pesados (As, Ba, Cd, Cr, Hg, Ag, Pb, Se), coliformes, *salmonella*, bacterias mesófilas, áscaris y otros helmintos, antes y después de aplicar una dosis óptima al 10%. Con esta dosis se logró controlar la reactividad por sulfuros y cianuros, pero no se

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redujeron las concentraciones de coliformes y de bacterias mesófilas. Algunos metales pesados (Cd, Cr, Hg, Ag, Pb) aumentaron luego de la aplicación de la dosis de cal hidratada, aunque las concentraciones halladas no constituyen peligro ambiental, de acuerdo a la normatividad ambiental vigente.

PALABRAS CLAVE: lodos; cal hidratada; pH; metales pesados; reactividad; coliformes.

TRATAMENTO DE LODO DE FUNDO DE LAGOAS FACULTATIVAS COM ESTABILIZAÇÃO EM CONDIÇÕES DE LABORATÓRIO.

RESUMO

Neste artigo apresentam-se os resultados obtidos no processo de estabilização química de lodos de lagoas de estabilização, mediante a adição de cal hidratada comum. Adicionaram-se em condições controladas de laboratório, dose de cal ao 5%, 7%, 9%, 10%, 11%, 12% e 15% a uma mostra de lodos de fundo da lagoa primária do sistema de tratamento de águas residuais da cidade de Montería, Colômbia. Analisaram-se a periculosidade do lodo (corrosividade, inflamabilidade e reatividade) e as concentrações de sulfeto, metais pesados (As, Ba, Cd, Cr, Hg, Ag, Pb, Se), coliformes, salmonela, bactérias mesófila, áscaris e outros helmintos, antes e depois de aplicar uma dose ótima ao 10%. Com este dose conseguiu-se controlar a reatividade por sulfetos e cianuretos, mas não se reduziram as concentrações de coliformes e de bactérias mesófilas. Alguns metais pesados (Cd, Cr, Hg, Ag, Pb) aumentaram logo da aplicação da dose de cal hidratada, embora as concentrações achadas não constituem perigo ambiental, de acordo com a normatividad ambiental vigente.

PALAVRAS-CHAVE: Lodos; Cal hidratada; PH; Metais pesados; Reatividade; Coliformes.

1. INTRODUCTION

Stabilization ponds are an excellent alternative for domestic wastewater treatment for Colombian municipalities because of their low degree of complexity and their simple operation. Organic material is removed through sedimentation and aerobic and anaerobic biological processes that generate bottom sludge over time, gradually reducing the pond's depth. Bottom sludge accumulation rates of 0.052 m³/person/year (Gonçalves et al., 2002) and 0.050 m³/person/year (Peña et al., 2000) have been reported in Brazil and Colombia, respectively.

Sludge accumulation is greater in primary lagoons and can affect their performance due to the reduction of effective volume and changes in the bottom structure, which alter the reservoir's hydraulics (Peña et al., 2000). Therefore, the periodical elimination of sludge tends to be unavoidable, and the long-term sustainability of these systems depends on effective management of this process. It is necessary to evacuate sludge every 5 to 10 years during the first stage of the lagoon's useful life (Bouza & Salas, 2013).

Appropriate disposal of bottom sludge is a real environmental problem since it is generally reactive due to cyanide and sulfide content, which is a result of the oxidation of organic matter in anaerobic conditions. This process usually involves reactions with reduction of organic sulfates due to the action of *Desulfovibrio* bacteria (Metcalf & Eddy, 1995). This sludge can also contain pathogenic organisms and contaminants, a wide range of heavy metals, and high concentrations of soluble salts which can negatively affect soil properties (Samaras et al., 2008). They are therefore considered dangerous and require treatment.

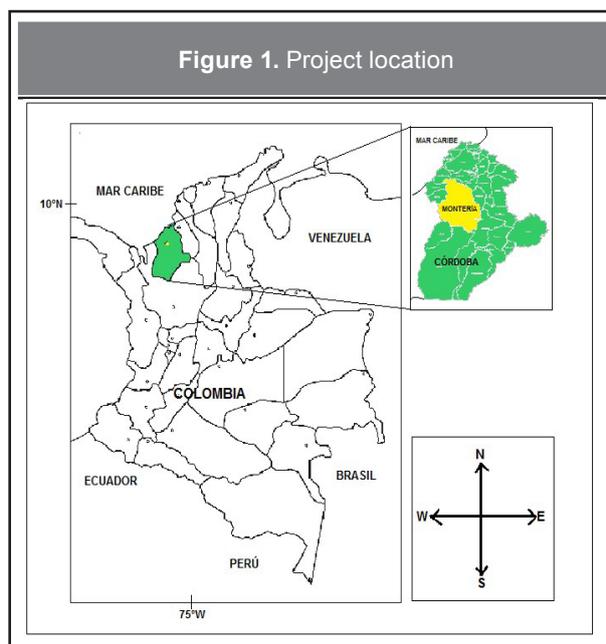
One of the most economical and simple techniques for avoiding bacterial activity in the sludge is chemical stabilization through the addition of lime to elevate pH to above 11.0 (Wong et al., 2000; Nakasaki et al., 1985). The reaction that takes place between the water and the lime raises pH and reduces the pathogen content (Diocaretz et al., 2010) due to the creation of an environment that does not favor the survival of microorganisms (Metcalf & Eddy, 1998). Several studies have ratified the necessity of a stable pH at 12.0 for a period of 20 to 60 days for effective elimination of

Salmonella in wastewater sludge (Amer, 1997; Reimers et al., 1998). Lime stabilization is therefore considered to be a treatment that requires a relatively long period of time (Arthurson, 2008). However, Strauch (1999) reported elimination of *Salmonella* in a period of 24 hours with a stable pH of 10. The author concluded that the elimination of these pathogens depends on the pH obtained, the lime's activity period, and the sludge's drainage (Strauch, 1999). In agreement with the previous findings, Bina et al. (2004) showed that the microbiological quality of a treatment system's sludge fulfilled the requirements for classification in category B in the first 2 hours of a test with a pH of 12, while in order to qualify for classification in category A, the inactivation of *Salmonella* and fecal coliforms was achieved after 2 and 24 hours, respectively, with the same pH (Bina et al., 2004).

The reactions involved in this process range from the neutralization of acids to the precipitation of undesired elements in the form of soluble salts (Gonçalves, 1999). The process also produces ammonia, which favors the reduction of viable helminth eggs (Méndez et al., 2002). The determining factors in the efficiency of the stabilization process when using virgin lime are the increase in pH and temperature (Thomaz-Soccol, 1998), while the effectiveness of hydrated lime depends only on the increase in pH (Andreoli, 1997).

An additional benefit of lime stabilization is that the high pH favors the precipitation of the majority of the metals present in the sludge, thereby reducing their solubility and mobility. The free calcium ions provided by the hydrated lime form complexes with odorous kinds of sulfides, such as hydrogen sulfide and organic mercaptans, resulting in sludge with less odor. Lime also causes an increase in solids content, making handling and storage of the sludge easier (Oates, 1998). This process has been proposed as an advanced treatment for reuse and proper disposal of wastewater sludge in important work documents in the European Union (Spinosa, 2004).

The goal of this study is to show the results of bottom sludge stabilization in the facultative lagoon in the city of Monteria, Colombia and the sludge's physiochemical characteristics before and after the chemical stabilization process using hydrated lime.

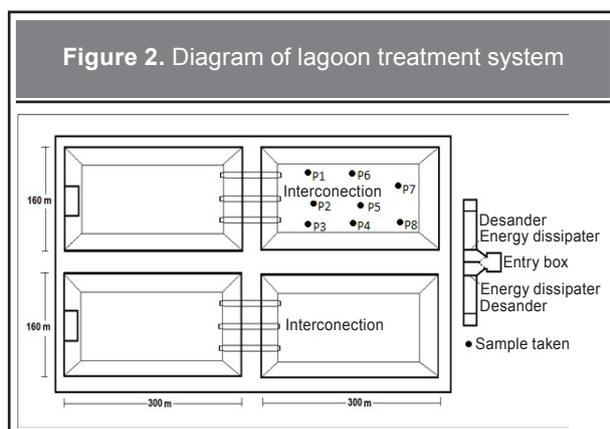


2. MATERIALS AND METHODS

The samples were collected from the wastewater treatment system in the city of Monteria in the north of Colombia. The treatment system receives the wastewater of approximately 300,000 inhabitants with an average inflow of 240 L/s. There are two parallel treatment trains, each with a facultative lagoon, followed by a maturation lagoon. 8 samples were taken from the facultative lagoon of the older treatment train in order to test the sludge's corrosiveness, flammability, and reactivity, as well as its concentration of sulfur, heavy metals (As, Ba, Cd, Cr, Hg, Ag, Pb, Se), bacterial strength, coliforms, *Salmonella sp.*, *Ascaris lumbricoides*, and other helminths. This analysis was completed in accordance with the methods established by the American Public Health Association (APHA, 1995) and by the Environmental Protection Agency (EPA, 1982).

Figure 1 shows treatment system's location, and **Figure 2** shows a diagram of the existing lagoons and the simple location.

The samples were collected, stored, and transported to the laboratory at the Universidad Pontificia Bolivariana in Monteria following the protocol described by the U.S. Environmental Protection Agency (EPA, 1993).



15 kilograms were taken from the most representative sample according to the results of a statistical analysis, and 7 subsamples of 1.0 kilograms each were prepared. These were mixed with doses of hydrated lime at 5%, 7%, 9%, 10%, 11%, 12%, and 15 %, in a weight-to-weight ratio of dry sludge and hydrated lime (Lim et al., 2002; Samaras et al., 2008, Madera et al., 2011). The subsamples' pH was measured with three repetitions every 30 minutes during the first 2 hours and then each hour for 22 contiguous hours. To reduce the attraction of vectors in sludge, the EPA recommends adding lime in sufficient quantities to achieve, exceed, and maintain pH at 12.0 units for a period of at least 2 hours and maintain a pH of at least 11.5 without adding more lime during an additional 22 hours (EPA, 1992; Andreoli, 2001). The sludge's pH was measured with a Metrohm brand 827 model potentiometer in a proportion of 1:2.5 (w/v, sludge weight: volume of distilled water) (Wong et al., 2000; Page et al., 1982; APHA, 1995). Corrosiveness, reactivity (due to sulfides and cyanides), and flammability, as well as concentrations of heavy metals (As, Ba, Cd, Cr, Hg, Ag, Pb, Se), coliforms, *Salmonella*, *Ascaris lumbricoides*, bacterial strength, mesophilic bacteria, and other helminths were also analyzed before and after application of the minimum dose in the sludge subsample, which allowed a pH greater than 11.5 the study period.

The data was analyzed using the statistical software Statgraphics Centurion, version XVII, with a Dell PC. One-way ANOVA (P-value < 0.05) was used to compare the main concentration differences between the samples taken from the lagoon and the variation coefficients in order to determine the degree

of homogeneity or heterogeneity of pH in terms of the dose of hydrated lime applied to the sludge.

3. RESULTS AND DISCUSSION

The results of the analyses performed on the sludge samples are presented in **Table 1**. None of the samples have corrosive or flammable properties, but all are reactive due to sulfide content with high concentrations in the facultative lagoon's inflow area. *Salmonella* shows a non-uniform presence in the area at the bottom of the lagoon, but no *Ascaris lumbricoides* eggs or other helminths were detected. Except for barium and mercury, the concentrations of heavy metals in the sludge samples were below the detectable limit for the analysis method used, which indicates the low presence of these contaminants in the lagoon bottom sludge.

The statistical results show that any sample taken is valid for obtaining the subsamples to which the different doses of hydrated lime will be applied, given that the verification and variance tests support the null hypothesis that the standard deviations of all 8 samples is the same. The P-value is less than 0.05, that is, there is no statistically significant difference between the standard deviations with a reliability level of 95.0%. The sample from point 3 was chosen to obtain the subsamples required for the test.

Table 2 shows the results of pH behavior with different doses of hydrated lime applied to the sludge subsamples in laboratory conditions. The 5% and 7% doses lowered pH levels below 12.0 units during the first 3 hours of the test and were therefore discarded. The 10% dose of hydrated lime maintained the pH of the sludge leachates with an average value of 12.0 \pm 0.15 with a maximum of 12.42 units pH and a minimum value of 11.84 units at the end of the test, thereby meeting the minimum conditions for being considered as a possible optimal dose (minimum pH of 11.5 units).

The 11% to 15% doses maintained pH above 12.0 units without reaching 12.5 units and presented a very small variation coefficient throughout the test. Given that the optimal dose will be the minimum dose of hydrated lime applied to the sludge able to maintain the pH of its leachates above 11.5 pH units, the 10% dose is the optimal dose for stabilization of the sludge from the facultative lagoon studied.

Table 1. Physiochemical and biological characteristics of the sludge

Analysis	P1	P2	P3	P4	P5	P6	P7	P8	Reference Level
Corrosiveness	-	-	-	-	-	-	-	-	-
Flammability	-	-	-	-	-	-	-	-	-
Reactivity	+	+	+	+	+	+	+	+	-
Residual Sulfur (mg/L)	6572,1	7572,1	5045.6	3929,3	1415,3	2698,8	2291,7	4322,8	500 ppm
Bacterial Strength (UCF/g)	14x10 ⁵	39x10 ³	11x10 ⁴	98x10 ³	54x10 ³	11x10 ⁵	11x10 ⁶	77x10 ⁴	-
<i>Salmonella</i> sp (NMP. <i>Salmo.g</i>)	0,02	0,0	5,61	0,22	0,76	5,56	0	6,50	-
Fecal Coliforms (NMP de C/g)	16x10 ⁴	20x10 ⁴	42x10 ⁴	28x10 ⁴	58x10 ⁴	42x10 ⁴	60x10 ⁴	50x10 ⁴	3, 2x10 ⁶
Áscaris lumbricoides (eggs/gr of base dry sludge)	0	0	0	0	0	0	0	0	1
Other helminths	No obs.	-							
Arsenic (mg/L)	<0,552	< 0,552	<0,552	<0,552	<0,552	<0,552	<0,552	<0,552	5,00
Barium (mg/L)	<0,031	<0,031	<0,031	1,43	1,59	1,85	1,27	1,95	100,0
Cadmium (mg/L)	<0,006	<0,006	<0,006	<0,006	<0,006	<0,006	<0,006	<0,006	1,00
Chromium (mg/L)	<0,088	<0,088	<0,088	<0,088	<0,088	<0,088	<0,088	<0,088	5,00
Mercury (µg/L)	<0,488	<0,488	<0,488	<0,488	4,59	<0,488	<0,488	<0,488	0,20
Silver (mg/L)	<0,0032	<0,0032	<0,0032	<0,0032	<0,0032	<0,0032	<0,0032	<0,0032	5,00
Lead (mg/L)	<0,031	<0,031	<0,031	<0,031	<0,031	<0,031	<0,031	<0,031	5,00
Selenium (mg/L)	<0,230	<0,230	<0,230	<0,230	<0,230	<0,230	<0,230	<0,230	1,00

Table 3 shows the behavior of the criteria tests to define the dangerousness of the sludge (corrosiveness, reactivity, flammability) before and after application of the optimal dose.

In order to consider the sludge dangerous based on its corrosiveness characteristics, the waste must

be aqueous and have a pH lower than or equal to 2.0 or higher than or equal to 12.5 pH units according to the information established in Annex 3 by Decree 4741 in 2005. As show in **Table 3**, the samples do not show corrosiveness, and the pH remained within the range specified.

The reactivity of dangerous waste depends greatly on the concentration of sulfides and cyanides present in it. Given that Colombian law regarding dangerous waste does not specify the minimum permissible value for considering waste to be dangerous based on sulfur and cyanide concentrations, the Mexican regulation (NOM-052-SEMARNAT-1993) was used as a reference. This regulation establishes that sludge can generate toxic gases, vapors, or smoke when exposed to pH conditions between 2.0 and 12.5 and be reactive with concentrations above 250 mgCN-/kg waste due to cyanides or 500 mgS-2/kg waste due to sulfides. The fresh sludge sample showed reactive properties with cyanide and sulfide concentrations above the reference limit values, but after application of the optimal dose, it did not show reactivity due to sulfides, though it did for cyanides if compared with the Mexican reference regulation. Flammability properties were negative for the samples.

Table 4 shows the results of heavy metal properties in the sludge leachates. The concentration of some heavy metals such as cadmium, chromium, mercury, silver, and lead increased in the leachates after application of the optimal dose. This is explained by the fact that these elements can easily move from the solid phase to the liquid phase in aquatic systems and vice versa, due to variations in both biotic and abiotic components, mainly because of pH variations (Alloway, 1995). The sediment's pH has an effect on the bioavailability of the majority of the heavy metals because it affects the balance between metallic speciation, solubility, adsorption, and ion exchange, making the metals soluble in the leachate (Reichman, 2002). In acid sediments, competition is caused between H⁺ ions and the metallic cations for exchange sites. At high pH levels in the sludge, desorption of the heavy metals toward the aqueous medium is caused, increasing their concentration and bioavailability in the leachate, while at low pH levels, the opposite effect occurs (Alloway, 1995).

Table 2. pH behavior with different doses of hydrated lime

Time (Hours)	Concentration of Hydrated Lime Dose (%)							
	0	5	7	9	10	11	12	15
1	6,88	11,83	11,96	12,39	12,41	12,43	12,44	12,46
2	7,22	11,82	11,93	12,35	12,42	12,43	12,41	12,45
3	7,17	11,48	11,60	11,93	12,33	12,40	12,42	12,44
4	7,09	-	-	12,13	12,18	12,27	12,31	12,43
5	8,01	-	-	12,02	12,23	12,30	12,31	12,41
6	7,28	-	-	11,90	12,24	12,27	12,31	12,43
7	7,14	-	-	11,95	12,19	12,29	12,30	12,42
8	6,98	-	-	12,00	12,12	12,25	12,31	12,41
9	7,01	-	-	12,00	12,20	12,27	12,31	12,42
10	7,11	-	-	12,03	12,20	12,27	12,32	12,43
11	8,03	-	-	11,99	12,19	12,25	12,33	12,42
12	7,12	-	-	12,00	12,20	12,27	12,33	12,40
16	7,05	-	-	11,80	12,00	12,22	12,32	12,40
24	6,98	-	-	11,49	11,84	12,17	12,31	12,42
Average pH	7,22	11,71	11,83	12,00	12,20	12,29	12,34	12,42
Standard Dev.	0,35	0,20	0,20	0,22	0,15	0,08	0,05	0,02
Coeff. Variation	0,05	0,02	0,02	0,02	0,01	0,01	0,00	0,00

Table 3. Sludge dangerousness due to corrosiveness, reactivity, and flammability

Parameters	Before Optimal Dose	After Applying Optimal Dose	Reference Limit Level
Corrosiveness	Negative	Negative	-
pH-residue pH Units	7.637	12.184	pH<2 y pH>12.5*
Reactivity	Positive	Negative	-
Reactive Cyanide mg CN ⁻ /Kg _(BS)	3373.50	385.55	250 mg C/kg**
Reactive Sulfur mg S ⁻² /Kg _(BS)	1211.14	9.0	500 mg S ⁻² /Kg**
Flammability	Negative	Negative	-

* Decreto 4741/2005 (Colombia) ** NOM-052-SEMARNAT-1993 (México)

Table 4. Heavy metals in sludge leachates

Parameters	Before Optimal Dose	After Applying Optimal Dose	Maximum Level Allowed in Leachate *
Arsenic-Leachate mg As/L	<0,552	<0,552	5.00
Barium-Leachate mg Ba/L	1.911	0.552	100.00
Cadmium-Leachate mg Cd/L	<0,006	0.014	1.00
Chromium-Leachate mg Cr/L	<0,088	0.151	5.00
Murcury-Leachate µg Hg/L	<0,488	13.242	200
Silver-Leachate mg Ag/L	<0,0032	0.0260	5.00
Lead-Leachate mg Pb/L	<0,031	1.351	5.00
Selenium-Leachate mg Se/L	<0,230	<0,230	1.00
% Solids (%)	74.08	87.19	N/A

* Dec. 4741/2005 (Colombia)

With the exception of Mo, Se, and As, bioavailability of the heavy metals in the sludge decreased as the sludge's pH increased due to their precipitation as insoluble hydroxides, carbonates, and organic complexes, but their concentration increased in the leachates (Silviera et al., 2003). This explains what occurred during the test stages.

However, if the reference values established in Annex 3 from Decree 4741 of 2005 are considered, all the concentrations found in the samples analyzed under the different test conditions stayed within the maximum permissible limits in the leachate such that

the toxicity risk due to heavy metals present in the leachates is low for the environment.

Waste with infectious properties is considered dangerous when it contains pathogenic agents. Pathogenic agents are microorganisms (such as bacteria, parasites, viruses, *Rickettsia*, and mold) and other agents with sufficient virulence and concentration to cause illnesses in humans or in animals. However, Colombian regulations do not establish permissible limits, and therefore the absence of these pathogenic agents will be considered the maximum permissible level. French legislation considers sludge to be sterilized if it contains less than 3 viable helminth eggs per

10.0 grams of sludge after treatment (Journal Officiel Français, 1998), while the EPA proposes reducing *Salmonella*, enteric viruses, and viable helminth eggs to below detectable limits as a goal for Class A sludge. In the case of helminth eggs, the detectable limit is defined as less than 1 viable helminth egg per 4.0 grams of total biosolids (EPA, 1992). **Table 5** shows the results obtained for biorisk properties of the sludge.

The results obtained before and after application of the optimal dose of hydrated lime to the sludge samples in laboratory conditions indicate that there are no changes in the biological parameters' concentrations. The concentrations of mesophilic bacteria and fecal and total coliforms found do not indicate that the dose applied had any effect whatsoever on total or partial elimination of these microorganisms. These results contrast with those found in recent studies, which claim control and inactivation of *Salmonella* and fecal coliforms with doses that guarantee a pH of 12.0 units for 24 hours (Bina et al., 2004). Studies on stabilization of primary sludge from the Cañaveralejo Wastewater Treatment Plant in Cali, Colombia, report control of coliforms with pH levels close to 12 units during a period of 3 days after a 15% dose of hydrated lime (Madera et al., 2011), which suggests that the 10% dose applied to the sludge studied does not guarantee complete sterilization, though it does guarantee a reaction of vector attraction in the sludge. However, it is possible to consider that the absence of

Salmonella and *Ascaris lumbricoides* eggs in the sludge studied indicates a low biological risk.

4. CONCLUSIONS

Applications of hydrated lime above 10% in relation to the quantity of solids present in the sludge raise pH to 12 units during the first two (2) hours of application and above 11.5 units during the remaining 22 hours. However, this dose does not guarantee total elimination of pathogenic agents such as mesophilic bacteria and fecal and total coliforms present in the sludge sample. In contrast, the dose applied efficiently reduced the sludge's reactivity, mainly caused by sulfurs (more than 99% efficiency), and to a lesser degree, that caused by cyanides (88.6% efficiency). According to the dangerousness, toxicity, and biological risk properties obtained from the sludge after application of the optimal dose of hydrated lime, it is possible to affirm that the treated sludge does not pose an environmental risk at the moment of its final disposal, whether in a sanitary landfill or on non-agricultural land.

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Table 5. Concentrations of coliforms, *Salmonella*, *Ascaris*, mesophilic bacteria, and other helminths in sludge

Parameters	Before Optimal Dose	After Applying Optimal Dose	Maximum Level Allowed in Leachate*
Total Coliforms, NMP/100 mL	1600	1600	Absent
Fecal Coliforms, NMP/100 mL.	1600	1600	Absent
Fecal Coliforms, NMP fec colif/g	18x10 ⁴	18x10 ⁴	Absent
<i>Salmonella</i> , NMP salmo/100 MI.	0	0	Absent
<i>Salmonella</i> , NMP salmo/g	0	0	Absent
<i>Ascaris lumbricoide</i> huevos/gr. Base seca.	0	0	Absent
Bacterial Strength, UFC/gr	47x10 ⁴	47x10 ⁴	Absent
Mesophilic Bacteria, UFC/ml	54	54	Absent
Other Helminths	Not Observed	Not Observed	Absent

* Dec. 4741/2005 (Colombia)

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