

# Eliminating pathogens from biosolids by alkaline stabilization

## Eliminación de patógenos en biosólidos por estabilización alcalina

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### Abstract

The Cañaveralejo wastewater treatment plant (PTAR-C) based in Cali, Colombia, produces almost 100t day<sup>-1</sup> of biosolids. Although these do not have heavy metals restrictions, they are class B due to their high content of pathogenic microorganisms and parasites. Alkaline stabilization was evaluated with a 9% dose (w/w) of hydrated quicklime applied to different 0.5 ton piles of wet (66.5% humidity) and dry biosolids at ambient temperature (25 – 31°C) for 72 hours (50.1% humidity). The experiment had a completely randomized design, composed of 6 duplicated treatments. With the alkali stabilization treatment, the pH increased above 12 units for sufficient time to ensure pathogen and parasite reduction in order to achieve a class A material level. However, with dry biosolids lumps formed that reduced the homogenization of the substrate with the alkali material, an undesirable factor for pathogen reduction.

**Key-words:** Biosolids, effluents, alkali treatments.

### Resumen

La Planta de Tratamiento de Aguas Residuales de Cañaveralejo —PTAR-C de Cali— Colombia, produce alrededor de 100 t/día de biosólidos que, aunque no tienen restricción por metales pesados, son clase B por el nivel de microorganismos patógenos y parásitos. En un diseño completamente al azar, conformado por seis tratamientos con su respectivo duplicado, se evaluó la estabilización alcalina con dosis del 9% peso a peso de cal viva e hidratada, aplicada a pilas de 0.5 t de biosólidos húmedos (66.5%) y secos a temperatura ambiente (25 - 31°C) durante 72 h (humedad 50.1%). Con la estabilización alcalina el pH aumentó a valores superiores a 12 unidades durante el tiempo suficiente para garantizar la reducción de patógenos y parásitos, alcanzando un material clase A; sin embargo, el biosólido seco facilitó la formación de grumos que dificultaron las labores de homogenización del sustrato con los alcalinizantes, factor indeseable para la eficiente reducción de patógenos.

**Palabras clave:** Desechos sólidos, aguas residuales, procesamiento, tratamiento alcalino.

### Introduction

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The presence of pathogens, parasites, heavy metals and agrochemicals may limit the potential for agricultural use of the sludge generated in the treatment of municipal effluent water (Metcalf Eddy, 2003). The digestion of sludge stabilizes the organic material, and reduces the volume, the volatile solids (between 45-50%) and pathogenic agents (García, 2006; Mahamud et al., 1996a). Researchers recommend a sufficiently long observation of the biosolids characters to avoid environmental impact, and to contribute to the preservation and recuperation of the environment (Ferreira, 2000; Mahamud et al., 1996b).

In 1993 the Environmental Protection Agency of the USA (EPA) defined two classes of biosolids depending on the pathogenic content: Class **A** ( $<1 \times 10^3$  fecal coliforms,  $< \frac{3}{4}$  NMP/g de *Salmonella* sp.,  $< \frac{1}{4}$  helminthes eggs, and  $< \frac{1}{4}$  UFP viruses) which can be used without restriction in agricultural applications; and Class **B** ( $<2 \times 10^6$  fecal coliforms), the use of which is restricted (EPA, 2003).

The selection of stabilization methods to obtain biosolids of one of these classes (aerobic or anaerobic digestion, composting and alkaline stabilization) depends on the final use (Mahamud et al., 1996 a, b). In alkaline stabilization, the material hardens on being exposed to air, and heavy metals are fixed, phosphorus insolubilized, and nitrogen lost through volatilization (EPA, 1999; Andreoli et al., 2001).

To obtain material of class **A** it is necessary to maintain the pH above 12 units for 72 h, and a temperature of 52 °C for 12 h. To obtain a class **B** biosolids, the pH should be above 12 units for 2 h (EPA, 2003). More conservative researchers recommend that temperatures are maintained above 62 °C for 1 h, or above 50 °C for 1 day, or above 46 °C for 1 week (Strauch, 1998).

The main disadvantages of alkaline stabilization stem from the fact that the final material is mainly applicable to acid soils, and that the volume of biosolids is increased through the application of lime, generating higher transport and final treatment costs (Mahamud et al., 1996a; Barrios & Cabirol, 2002). After biosolid stabilization, the total alkalinity increases and the nitrogen / ammonium and the phosphorus content decreases (Andreoli et al., 2001; Williford, et al., 2007).

The present study was aimed at evaluating the pathogen reduction using alkaline stabilization with hydrated quick lime in biosolids from the Cañaveralejo wastewater treatment

plant (PTAR-C) based in Cali, Colombia with two levels of humidity: dehydrated in a filter press followed by 72 hours at ambient temperature; and filter press dehydration only.

## **Materials and methods**

The experimental phase was carried out in the maturation area of the Compost pilot plant at the Cañaveralejo PTAR, Cali, Colombia, 3° 28' 16.2" N & 76° 28' 39.3" O, 995 m.a.s.l. The PTAR treats 6.32 m<sup>3</sup>/s under the Advanced Primary Treatment (APT) mode; the sludge is weighed digested anaerobically, and dehydrated in a filter press, to produce approximately 100 t/day biosolids with 65% to 70% humidity (EMCALI, 2009).

To evaluate the alkaline stabilization, a completely randomized design was used with six treatments and two repetitions (Box 1). Two states of biosolids humidity were considered: 66.5% (normal humidity content) and 50.1% (additional dehydration at ambient temperature between 25 and 31°C for 72 h (dry biosolid). 9% of dehydrated lime and quicklime was applied to the biosolids piles of 0.5 t. The variables analyzed at the start and the end of the assay are presented in Box 2.

**Box 1.** Treatments applied in the experiment. Cañaveralejo waste water treatment plant (PTAR-C), Cali, Colombia.

Treat. (no.)	Substrate	Alkaline Material	Percent alkaline agent
1	Humid biosolid (500 kg/treatment)	Control	0%
2		Hydrated lime	9%
3		Quick lime	9%
4	dry biosolid (500 kg/treatment)	Control	0%
5		Hydrated lime	9%
6		Quick lime	9%

Due to the high number of experiences related to the contact time necessary for the reduction of biosolid pathogens (Boost & Poon 1998; Madera et al., 2002; Bina et al., 2004; Araque, 2006; Torres et al., 2008), observations of the process were made for 13 days of the variables in Box 3, forming compound samples through the quartering method. Day zero (0) was considered as the day of application of the alkaline treatment.

Descriptive statistical tools were employed for the observation variables, such as box plots for the pH, and the completely randomized model was applied for the response variables (fecal coliforms and helminthes eggs).

**Box 2.** Variable analyzed in the applied treatments at the start and the end of the experiment. PTAR-Cañaveralejo, Cali

Physical-chemical Variable	Unit	Measurement technique*
pH	Units	Potentiometer
Humidity	%	Gravimeter
Organic material	%	Digestion certification
Total Nitrogen	%	Digestion certification
Total phosphorus	mg/Kg	Digestion certification
Potassium	—	Spectrophotometer
Sodium -Na	meq/100mg	Spectrophotometer
Calcium -Ca	meq/100mg	Spectrophotometer
Magnesium -Mg	meq/100mg	Spectrophotometer
Iron -Fe	mg/kg	Spectrophotometer
Copper -Cu	mg/kg	Spectrophotometer
Manganese -Mn	mg/kg	Spectrophotometer
Zinc -Zn	mg/kg	Spectrophotometer
<b>Microbiological and parasitological</b>		
Fecal coliforms	UFC/g	Membrane filtration
<i>Salmonella</i> sp.	Presence	selective medium
Helminthes eggs	HH/g	Bailinger (1979) modified

a. Apha, 2005.

## Results and discussion

The humid and dry biosolids have potential agricultural application for their organic material and nutrient content. The ratio C/ N is within the range for biosolids from waste water treatment plants (ADEME, 2002), and are not restricted from the physical – chemical perspective, nor from heavy metal content, such as iron, copper, manganese and zinc, according to the EPA guide to pathogen and vector control in sludge and biosolids (EPA, 2003); However, due to their high microbe content, these biosolids are classified as Class B (Box 4).

**Box 3.** Variables evaluated and frequency of measurement. PTAR-Cañaveralejo, Cali.

Variable	Frequency
<b>Field measurements</b>	
Temperature (°C)	Day 0: eight measurements daily, every hour for the first 8 h Day 1 to 6: three measurements daily. Day 7 - 13: one measurement daily.
Humidity (%)	Measurement on days 0, 3, 5, 8, 11 & 13
pH (units)	Day 0: four measurements daily every 2 h for the first 8 h. Day 1 al 3: two measurements daily. Day 3 - 13: one measurement daily.
<b>Microbiological and parasitological</b>	
Fecal coliforms (UFC/g)	Measurement on Day 0, 3 & 13.
<i>Salmonella</i> sp. (presence)	Measurement on Day 0, 3 & 13.
Helminthes eggs (HH/g)	Measurement on Day 0, 3 & 13.

In the dry biosolids, an additional reduction in humidity of 16.4% was achieved before mixing with the alkaline agent. The physical-chemical, microbiological and parasitological characteristics were not altered, although lumps formed in the sludge making it difficult to mix the lime with the biosolid, and thus affecting the ability to reduce pathogen levels (Andreoli et al., 2001). This phenomenon was also observed by Torres et al. (2007) in biosolid compost from PTAR-C.

**Box 4.** Initial biosolid characterization. PTAR-Cañaveralejo, Cali.

Variable	Humid biosolid*	Dry biosolid**	Reference values
<b>Physical-chemical</b>			
pH	7.21	6.96	6.5 – 7.5 (1)
Humidity (%)	66.5	50.1	—
Organic material (%)	29.58	25.88	—
Total nitrogen(%)	2.42	2.25	1.6-3.0 (1)
Ratio C/N	7.10	6.7	—
Total phosphorus (mg/kg)	304.03	296.73	15000 – 40000 <sup>a</sup>
Potassium (meq/100g)	0.05	0.04	0-3.0 (1)
Sodium (meq/100g)	0.02	0.02	—
Calcium (meq/100g)	0.70	0.68	—
Magnesium (meq/100g)	0.06	0.07	—
CIC (meq/100g)	104.49	125.85	—
Iron-Fe (mg/kg)	<1.00***	<1.00***	3.0-8.0 <sup>a</sup>
Copper- Cu (mg/kg)	<0.10***	<0.10***	1500 <sup>b</sup>
Manganese- Mn (mg/kg)	11.28	13.40	—
Zinc- Zn (mg/kg)	2.71	2.71	2800 <sup>b</sup>
<b>Microbiological and Parasitological</b>			
Fecal coliforms (UFC/g)	6.30 x10 <sup>5</sup>	7.90 x 10 <sup>5</sup>	Class <b>A</b> : <1X10 <sup>3</sup> <sup>b</sup> Class <b>B</b> : <2X10 <sup>6</sup> <sup>b</sup>
<i>Salmonella</i> sp.	Absence	Absence	< 3NMP/4gr
Helminthes eggs (HH/g)	5	5	Class A: < 1HH/4 gr <sup>b</sup>

\* Derived from anaerobic digestion and dehydration in a filter press.

\*\* Humid biosolid submitted to natural dehydration at ambient temperature (25 - 31 °C) for 72 h.

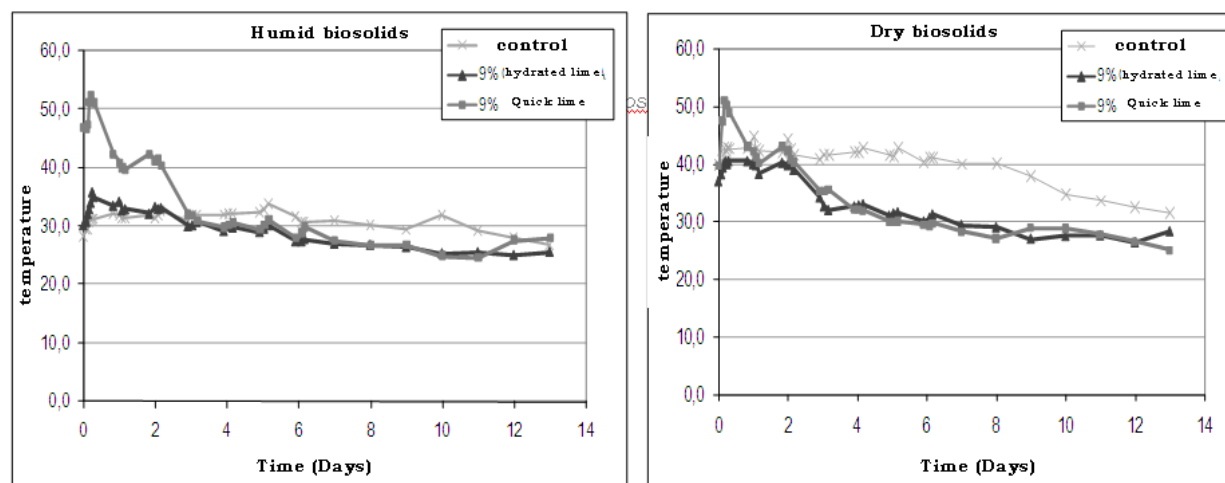
\*\*\* Limit of detection of the method.

a. Metcalf & Eddy (2003). b = EPA (2003).

## Alkaline stabilization

**Temperature.** The addition of lime increased the temperature on the first day (40 °C & 52 °C), with quick lime being responsible for the greatest increase. Although the elevated temperatures did not continue for the minimum time recommended by the EPA (2003) to achieve a Class **A** biosolid (Figure 1), the experiences of alkaline stabilization of Araque (2006) and Torres et al. (2009) showed that the biosolids derived from the waste water treatment plants that do not fulfill this requirement may also achieve Class A status. In the dry biosolid, the temperature difference between quick lime and hydrated lime was less, showing the potential effect of

humidity, giving a lesser liberation of heat in the reaction, and consequently lower increase in the same.



**Figure 1.** Temperature behavior through time in the treatments.  
PTAR-Cañaveralejo, Cali.

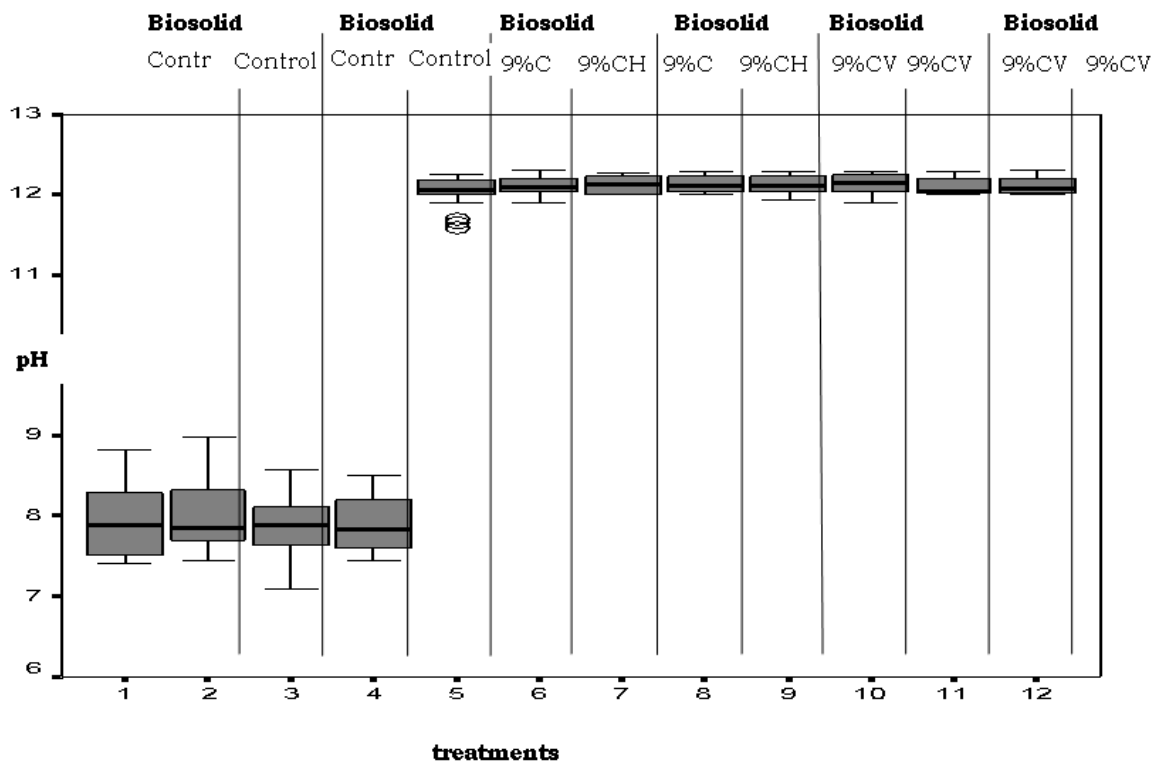
**Humidity.** The final humidity content varied between 28% and 31% for humid biosolids, and between 18% and 32% for the dry material. The natural dehydration of the humid biosolid after 72 hours did not significantly affect the final humidity levels, with the exception of the treatment with 9% hydrated lime that presented the lowest level (Box 5), fulfilling the requirement of Icontec (2003) of levels lower than 20% for agricultural products, and material used as fertilizers and conditioners. These results show that in order to fulfill the requirements, it would probably be necessary to prolong the contact time of the alkaline agent.

**pH.** With the lime dose used, the pH increased to a value of 12 units (Figure 2), meeting the recommendations of the EPA (2003) for pathogen reduction.

**Box 5.** Humidity behavior (%) through time. PTAR-Cañaveralejo, Cali.

No.	Treatment	Alkaline Material	Percent (%) alkaline agent	Day							
				0*	1	2	3	5	8	11	13
1	Humid Biosolid	Control	0	66.5	63.5	59.9	55.9	49.2	37.1	32.8	30.7
2		Hydrated lime	9	60.3	57.7	48.4	48.6	41.1	34.0	30.5	28.1
3		Quick lime	9	55.0	52.1	50.9	49.4	36.6	34.4	34.1	29.3
4	Dry biosolid	Control	0	50.1	45.3	45.2	43.0	42.6	39.8	35.6	31.6
5		Hydrated lime	9	49.0	44.0	43.8	41.5	36.1	22.4	20.5	18.5
6		Quick lime	9	47.2	45.0	43.3	39.8	37.4	29.7	28.1	26.6

\* Sample taken at the moment of mixing the biosolid with the alkaline agent.



**Figure 2.** Box-plot for pH in treated biosolids. PTAR Cañaveralejo, Cali.  
CH = Hydrated lime. CV = Quick lime. D = duplicate. Atypical value.

### Microbiological and parasitological

Although in this study an experimental design was applied, it was not possible to undertake analysis of variance, as the results of the response variables (fecal coliforms and helminthes eggs) at the end of the assay were zero (0) for the lime treatments (Box 6).

The hydrated and quick lime at the 9% dose resulted in a total reduction of fecal coliforms and helminthes eggs, achieving a Class A biosolid after 13 days. Additionally, the assay did not detect the presence of *salmonella* sp. These results confirm that if the pH is maintained at a high level for more than 72 h, even though the temperature requirement is not met, it is sufficient to obtain a biosolid that does not generate risks of pathogenic microorganisms (Araque, 2006; Torres et al., 2009 ).

### Physical – chemical characteristics

On the 13<sup>th</sup> day, the control treatments presented similar final values (Box 7), indicating that there is not a significant effect of reducing the humidity content as applied to the dry biosolid. All of the treatments showed a potential use for agriculture given their high nutrient content. Additionally, they did not present restrictions due to content of manganese, zinc, copper, and iron, in agreement with the recommendations of the EPA (2003).

**Box 6.** Results for fecal coliforms and helminth eggs on days 0, 3 & 13. PTAR-Cañaveralejo, Cali.

Biosolid	Treatment	Day					
		0		3		13	
		CF	HH	CF	HH	CF	HH
Humid Biosolid	Control	3.87 x 10 <sup>6</sup>	6	2.76 x 10 <sup>6</sup>	3	3.32 x 10 <sup>3</sup>	3
Dry Biosolid	Control	1.36 x 10 <sup>6</sup>	3	8.00 x 10 <sup>4</sup>	3	3.02 x 10 <sup>4</sup>	3
Humid Biosolid	9% hydrated lime	0	0	0	0	0	0
Dry Biosolid	9% hydrated lime	0	0	0	0	0	0
Humid Biosolid	9% quick lime	0	0	0	0	0	0
Dry Biosolid	9% quick lime	15	0	0	0	0	0

CF: Fecal coliforms (UFC/g), H.H: helminthes eggs (HH/g)

**Box 7.** Final physical-chemical results for the experimental biosolids. PTAR-Cañaveralejo, Cali.

Variable/substrate	Humid	Dry	Humid	Dry	Humid	Dry	Reference value
	Biosolid * (control)	biosolid ** (control)	Biosolid * (9% c.h. <sup>a</sup> )	biosolid ** (9% c.v. <sup>b</sup> )	Biosolid * (9% c.h.)	biosolid ** (9% c.v.)	
pH	7.24	7.20	11.37	12.66	12.65	12.68	5.5 – 8.0 <sup>c</sup>
Humidity %	30.7	31.6	28.1	18.5	29.3	26.6	30 – 60 <sup>a</sup>
O.M. %	28.31	28.52	26.35	25.02	23.30	23.77	—
Total nitrogen %	2.21	2.14	1.47	1.30	1.49	1.04	3.0 <sup>d</sup> )
Ratio C/N	7.43	7.73	10.39	11.16	9.07	13.25	—
Total P mg/kg	284.38	294.42	3.03	1.83	0.47	1.63	1.5 – 4 <sup>d</sup>
K meq/100g	0.06	0.05	0.03	0.03	0.03	0.03	—
Na meq/100g	0.03	0.02	0.02	0.02	0.01	0.01	—
Ca meq/100g	1.57	1.51	2.82	4.97	4.99	5.04	—
Mg meq/100g	0.10	0.09	0.04	0.21	0.22	0.22	—
CIC meq/100g	97.90	103.22	68.47	67.81	65.66	59.55	—
Fe mg/kg	1.26	1.00	1.00	1.00	1.00	1.00	—
Cu mg/kg	0.45	0.42	14.18	19.32	18.35	17.32	1500 <sup>e</sup>
Mn mg/kg	38.09	22.30	0.25	0.25	0.25	0.25	—
Zn mg/kg	56.42	20.59	0.30	0.15	0.15	0.15	2800 <sup>e</sup>

\* Derived from anaerobic digestion and dehydration.

\*\* Submitted to natural dehydration at ambient temperature (25 - 31 °C) for 72 h.

\*\*\* Limit of detection of the method.

a = hydrated lime. b = quick lime. c. Metcalf & Eddy (2003). d = Fernández & de Souza (2001). e = EPA (2003).

The final pH values were high, and so it is recommended to perform a neutralization of the solids before their use in agriculture or application to soils with acidity problems. A reduction in organic material, nitrogen and phosphorus was seen in the treatments as a consequence of the volatilization of carbon and nitrogen due to the increase in temperature and the pH during the alkaline stabilization process, which is reflected in the ration C / N. The low phosphorus content seen (Box 8) was probably due to the immobilization reaction of this element, forming precipitates of phosphate and calcium (Fernandes & Souza, 2001; Méndez et al., 2002; Andreoli et al., 2001; Williford et al., 2007). Zinc and manganese are immobilized at high pH values, being converted to insoluble elements, a condition that is reflected in the low final values of these elements obtained in this study. .

The increase in copper in the lime treatments shows that this element may be present in traces in the lime, a condition associated with the type and the source of lime. Once the lime and the biosolids have been mixed, the copper may convert to a more soluble form, which is



reflected in the increased levels at the end of the experiment. Jamali et al. (2008) found that the metal content in the sludge diminished after application of lime, as its availability is reduced at high pH, with the exception of cadmium and copper, which increased their mobility by 4% or 5% and thus their availability for plants, corroborating the increase in copper in the treatments and the reduction in iron, manganese and zinc.

## **Conclusions**

The results obtained in this study allow the following conclusions to be made:

- The additional drying step at ambient temperature for 72 h promoted the formation of lumps, which hindered the homogenization of the biosolids with the alkaline agents.
- In the piles of 0.5 t, with 9% of quick lime and hydrated lime, the pH increased to values greater than 12 units, sufficient time for a reduction in pathogens and parasites, achieving a Class A biosolid, without meeting the requirements of recommended temperature.
- The physical-chemical quality of the biosolids sanitized with lime, showed their potential for application without any restrictions except that of pH.

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