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Evaluation of coagulation sludge from raw water treated with Moringa oleifera for agricultural use

Evaluación de lodos de coagulación de agua cruda tratada con Moringa oleífera para uso agrícola

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ABSTRACT

Coagulation-flocculation is a physical-chemical process responsible for producing the largest amount of sludge in the purification of natural raw water. Conventionally, aluminum sulfate or alum has been used as a coagulant. However, disposal of the sludge produced has been problematic for the environment due to excess aluminum. Currently, the convenience of using natural coagulants such as seed extracts from *Moringa oleifera* (MO) is being studied, although, the properties of sewage sludge produced and its possible reuse are unknown. In this paper the physical-chemical, nutritional and dangerous characteristics from MO sludge were evaluated by using standard methods to verify its potential use in agricultural soils. Results indicated that pH, electrical conductivity, ion exchange capacity, organic matter and micronutrients from sludge were suitable for application to soils with agricultural potential; but deficiency of macronutrients and presence of fecal coliforms limits it to be used as soil improver and not as fertilizer. Sludge stabilization with hydrated lime at doses greater than or equal to 3 % was effective to ensure the elimination of pathogenic microorganisms and to obtain a Class A sludge, unrestricted for agricultural use and suitable for acid soils.

Keywords: Agricultural soil, *Moringa oleifera*, reuse, sludge.

RESUMEN

La coagulación-floculación es el proceso fisicoquímico responsable de la mayor producción de lodos residuales en la potabilización de aguas crudas naturales. Convencionalmente se ha utilizado como coagulante el sulfato de aluminio o alumbre. Sin embargo, la disposición de los lodos que produce ha resultado problemática para el ambiente debido al exceso de aluminio. Actualmente se estudia la conveniencia de utilizar coagulantes naturales como extractos de semilla de *Moringa oleífera* (MO), aunque se desconocen las propiedades del lodo residual que produce y su posible reutilización. En este trabajo se evaluaron las características físicas, químicas y nutricionales de los lodos de MO, siguiendo métodos estandar, para verificar su posible utilización en suelos agrícolas. El pH, la conductividad eléctrica, la capacidad de intercambio iónico, la materia orgánica y los micronutrientes del lodo de MO resultaron apropiados para ser aplicados en suelos con vocación agrícola; pero la deficiencia de macronutrientes y la presencia de coliformes fecales lo restringe a ser utilizado como mejorador de suelos y no como abono. La estabilización del lodo con cal hidratada, en dosis iguales o superiores a 3 %, resultó efectiva para garantizar la eliminación de microorganismos patógenos y para la obtención de un biosólido de clase A, sin restricciones para uso agrícola y conveniente para suelos ácidos.

Palabras clave: Lodos, *Moringa oleífera*, reuso, suelos agrícolas.

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Introduction

Raw water purification includes physical and chemical processes, such as sedimentation, coagulation, flocculation, filtration and disinfection. The coagulation and flocculation are the main pre-treatment practices employed by the water industry worldwide and, conventionally, aluminum and iron salts have been used for this purpose for many years. Therefore, sludge from aluminum sulfate or alum are the by-products most widely generated by this industry, and its characteristics are highly dependent on the quality of source

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water, the quality and purity of alum and other chemicals used in the treatment (Elangovan and Subramanianet, 2011; Zhao *et al*., 2011; Maiden *et al.,* 2015).

Generally, production of sludge generated in the water treatment process has been estimated between 1 % and 3 % of the total volume of treated raw water (Blakemore *et al.,* 1998). Nonetheless, they are considered as an industrial waste product and, as such, there is fear regarding the use of this material for disposal in the environment (Ippolito *et al.,* 2011). The disposal of alum sludge is a concern in various parts of the world, mainly because of its toxicity to fish, freshwater, algae, protozoa and marine bacteria (Georges *et al.,* 1995; Gutiérrez *et al.,* 1998.). In addition, these wastes have a wide variety of undesirable features that can have adverse effects on the environment when used as soil improvers in agricultural land (Kupper *et al*., 2014; Alvarenga *et al.,* 2015).

It is possible to find numerous research studies on the use of sludge generated in wastewater treatment, but there are limited review articles on the use of alum sludge (Dassanayake *et al.,* 2015). However, this sludge is considered as one of the best adsorbents for the removal of contaminants from wastewater, in particular, for a wide range of heavy metals (Gibbons *et al.,* 2009), phosphorus (Yang *et al.,* 2006; Mohammed and Rashid, 2012), perchlorates (Makris *et al.,* 2006), fluorides (Sujana *et al.,* 1998) and textile dyes (Chu, 2000) by adsorption or by chemical precipitation. They have also been used as a substrate in artificial wetlands for tertiary treatment (Zhao *et al*., 2011; Bai *et al*., 2014) and as a coagulant to remove oil, grease, chemical oxygen demand and suspended solids from wastewater (Jangkorn *et al.,* 2011). Additionally, alum sludge can be used as a partial replacement of clay during the manufacturing process of bricks (Elangovan and Subramanian, 2011), although they are of low tensile strength (Carvalho and Antas, 2005). The use of alum sludge in cement manufacturing has also been reported (Pan *et al.,* 2004), as well as a raw material in the manufacture of ceramic products (Vincenzi *et al.,* 2005).

The use of alum sludge as a soil improver has been questioned by several investigators due to the high concentrations of aluminum ions it provides, and its toxicity in the soil (Babatunde and Zhao, 2007) due to the different features of the sludge according to the processes of each water treatment plant and the climate changes that alter the availability of contaminants in the ground (Zhao *et al*., 2011; Maiden *et al*., 2015).

Currently, the use of alternative coagulants different to inorganic salts is being extensively studied in the world. Technical literature has reported a large number of coagulants-flocculants from plant, animal or microorganism sources used in the treatment of natural waters and sewage (Betatache *et al.,* 2014). Among the natural coagulants, the seed extract from *Moringa oleifera* tree is undoubtedly the most studied by the scientific community because its coagulant properties have been widely recognized (Yin, 2010). Several studies have shown that coagulant activity from moringa seeds is comparable with that obtained by using aluminum sulfate or alum (Arnoldson *et al*., 2008; ; Ridwan *et al*., 2011; Sandoval *et al*., 2013; Shahzad *et al.,* 2014; Feria *et al.,* 2014; Rodiño *et al*., 2015).

Moringa oleifera is a tropical plant originally from northwest India (Ledo *et al.,* 2009) belonging to the Moringaceae family. The coagulant is avowedly active, safe and cheap (Ndabigengesere *et al.,* 1995; Pritchard *et al.,* 2009), making its widespread use possible in water treatment, bringing economic benefits to producing countries, besides being an environmentally correct alternative. Ndabigengesere *et al*. (1995) verified that the sludge generated by using the seed of *Moringa oleifera* as a coagulant is not toxic and has a substantially smaller volume than the sludge produced when aluminum sulfate is used as a coagulant. However, currently there are no studies on the physicochemical quality of this sludge, nor its potential use, particularly for agricultural use.

The purpose of this research is to conduct a physicochemical characterization of the sludge obtained from the purification of raw water from a surface stream, using *Moringa oleifera* seeds as a coagulant; and identify their potential use in agricultural soils vocation.

Experimental development

Raw water samples

Raw water samples were taken from the right bank of the Sinu River in the Mocari neighborhood, in the city of Monteria, Cordoba. Simple sampling between March and October 2015 was made, at the end of the dry period and the beginning of the rainy season in the region**.**

Preparation of coagulant extract

M. oleifera seed was obtained from the dried pods that were manually removed from the shell and then dried at room temperature for one day (Pritchard *et al.,* 2010). The dried seeds were ground in a Corona brand hand grinder, obtaining a fine powder that was sieved using a Grain Test brand mesh of 0,6 mm (Number 30 according to series of Tyler ATSM E-11/2004). Then, 10,0 grams of seed were taken and dissolved up to 1,0 liter with 1,0 % (w/v) saline solution. The solutions were initially mixed for one hour with a Schott E & Q AMPC-1 magnetic stirrer, then centrifuged at 3,500 rpm during 10 minutes in a K Gemmy brand centrifuge, model PLC-05, and finally filtered under reduced pressure with a GAST-Mod-DUAp104-AA vacuum using a cellulose filter paper. The filtrate was labeled as coagulant saline extract (SCE 10,000 mg L^{-1}) and kept refrigerated at 4°C (Rodiño *et al*., 2015).

Jar Test

For treatability tests, 16 different turbidities ranging from 125 to 380 NTU were taken, depending on the samples randomly performed. Extract from *Moringa oleifera* seeds was applied as a coagulant in a single dose of 25,0 mg L^{-1} to each of the samples obtained in the monitoring (Feria *et al.,* 2014). A jar test EyQ F6-300-T brand equipment with six rotating paddles and equal number of 1000 mL beakers was used. Rapid mixing was done at 200 rpm for one minute (shear rate of 170 s^{-1}), followed by slow mixing of 40 rpm for 20 minutes (shear rate 22 s^{-1}), and a settling time of 30 minutes (Muyibi *et al.,* 2003; Feria *et al.,* 2014).

Physicochemical parameters of Sludge

Physicochemical, microbiological, and hazardousness parameters in the overall sample of sludge were analyzed. The pH, conductivity, cation exchange capacity, moisture content, organic matter, heavy metals (As, Cd, Cr, Hg, Pb), salmonella, helminth eggs, total and fecal coliforms analyses were done according to standard methods (APHA, 2005). For the analysis of flammability, reactivity, reactive cyanide, and reactive sulfide in sludge, the methods proposed by the US Environmental Protection Agency were followed (EPA, 1999). Finally, a comparison between the samples of the sludges was performed.

Chemical stabilization of sludge

Out of the total sample collected in the trials of jar test, 500 grams were taken to analyze the physical and chemical characteristics of the sludge, and 500 grams were taken for the stabilization process with hydrated lime. Afterwards, 5 subsamples of 100 grams each were prepared, mixed with doses of hydrated lime at 1 %, 2 %, 3 %, 4 %, 5 %, i.e. in weight to weight ratio of dry sludge and hydrated lime (Samaras *et al.,* 2008; Madera *et al.,* 2011). The pH of the sub-samples was measured with three repetitions, every 60 minutes during the first 12 hours and then every 12 hours until completing monitoring of 72 continuous hours (EPA, 1993). The lowest concentration capable of guaranteeing sludge to a stable pH above 12 units and absence of pathogens, was determined as the optimal dosage of hydrated lime for the stabilizing of the sludge (EPA, 1993; Feria and Martinez, 2014).

Data were analyzed using the statistical package Statgraphics Centurion, version XVII, with a personal computer DELL. A one-way ANOVA (P-value <0,05) was used to compare the main concentration differences from the samples and coefficients of variation, to determine the degree of homogeneity or heterogeneity from the pH depending on the dose of hydrated lime applied to the sludge samples.

Result

Table 1 shows the results of the physicochemical analysis of the sludge sample of *Moringa oleifera*.

Table 1. Physicochemical characteristic of *Moringa oleifera* sludge compared with alum sludge, worm compost and compost.

Parameter	Moringa oleifera sludge	Range alum sludge*	Worm compost**	Compost**
pH	6,27	5,12 - 8,0	6	7,42
E. Conductivity (dS m^{-1})	0,0126	0,36 - 1,66	1,5***	2***
Cation exchange capacity (cmol kg^{-1})	24,74	13,6 - 56,5	27***	30***
Organic matter (g kg^{-1})	18,50	63 – 144	20***	25***
Total N (g kg^{-1})	0,4187	4,0 - 4,8	22,40	22
Total P (g kg^{-1})	0,002	3,13 - 3,50	1,20	1,40
Total Al (g kg^{-1})	9,53	27 - 153	250***	250***
Total Ca (g kg^{-1})	0,98	2,2 - 11,70	13,30	9,50
Total Mg (g kg^{-1})	0,52	2,40 - 7,90	12,10	8,40
Total Na (mg kg^{-1})	99	175	1200	2600
Total K (mg kg^{-1})	82	148	7900	2200
Total Mn (g kg^{-1})	0,40	0,80 - 2,99	0,196	0,213
Total Zn (mg kg^{-1})	153,40	53,3 - 160	91	86
Total Cu (mg kg^{-1})	77,40	35 – 624	38	41
Total Fe (g kg^{-1})	1,59	4,87 – 37	0,357	0,367

*** Adapted from Munévar, 2004.
** Adapted from Olivares *et al.,* 2012.
* Adapted from Dassanayake *et al.,* 2015.

The pH of the slough resulting from the coagulation of raw water with *Moringa oleifera* (MO) was 6,27, a value that is in the typical range for proper plant growth (Bohn *et al.,* 1985). The (EC) electrical conductivity of MO sludge (0,0126 dS m^{-1}) is well below the typical range of the EC of alum sludge (0,36-1,66 dS m^{-1}) and the critical value recommended for agricultural soils (4,0 dS m^{-1}), which gives it better characteristics for application to crops sensitive to salinity (Brady and Weil, 2002). The cation exchange capacity (CEC) of sludge from MO was 24,74 cmol kg^{-1}

which, like the Ion Capacity Exchange from alum sludge (13,6-56,5 cmol kg^{-1}), is comparable to the typical range of ICE for agricultural soils from 3,5 to 35,6 cmol kg^{-1} (Dayton *et al.,* 2001). High values of CEC in MO sludge allow the supply of cationic nutrients to the soil and facilitates growth and development of plants (Dayton *et al.,* 2001).

The content of organic matter in the MO sludge is lower than that reported in the literature for alum sludge, due to characteristics of raw water from the source. However, the organic matter content presents significant amounts that can improve physicochemical properties of agricultural soils. Macronutrients such as N, P, Ca, Mg, Na, and K present in MO sludge are in concentrations well below the concentrations reported by Olivares *et al*. (2012) for vermicompost and compost samples, limiting its use as fertilizer. However, micronutrients or trace elements such as Mn, Zn, Cu and Fe show concentrations higher than those reported for vermicompost and compost samples, favoring its use as a soil amendment. The main constituent of sludge alum is aluminum; therefore, its concentration is higher compared to the Al concentration in MO sludge. This feature of MO sludge flavors the soil, as excess aluminum can alter the symbiotic relationships between plants and microbes very close to the rhizosphere (Zhao *et al.,* 2011).

The toxicity due to heavy metals from sludge is shown in Table 2. For this classification, the criteria established in Colombian law for the use of biosolids generated in wastewater treatment plants were taken (Ministerio de Vivienda, Ciudad y Territorio de Colombia, 2014). These guidelines are widely used to differentiate municipal and industrial solid waste, as hazardous or not.

Table 2. Toxicity due to heavy metals in dry sludge from *Moringa oleifera.*

Parameter	Moringa oleifera sludge	Ceiling concentration limits**	
		Biosolids class A	Biosolids class B
Arsenic mg kg^{-1}	0,6	20	40
Cadmium mg kg^{-1}	0,6	8	40
Chromium mg kg^{-1}	105,8	1000	1500
Mercury mg kg^{-1}	0,238	10	20
Lead mg kg^{-1}	6,4	300	400

*Adapted from Dassanayake *et al.,* 2015.
**Decree 1287/2014 (Colombia).

Concentrations of heavy metals in MO sludge were significantly lower than those maximum concentrations established in Colombian law. Therefore, it can be considered as non-toxic solid waste and classified as a biosolid class A (i.e., unrestricted heavy metal for agricultural use). Also, these sludges have few environmental risks compared to those produced in the wastewater treatment plants because they are cleaner and have a low content of heavy metals and other hazardous organic compounds (Dassanayake *et al.,* 2015).

The reactivity of a hazardous waste depends largely on the concentration of cyanides and sulfides present. However, Colombian regulations for hazardous waste do not quantify the minimum allowable value to consider a hazardous waste by reactivity from concentrations of sulfides and cyanides. The Mexican standard (NOM-052-SEMARNAT-1993) was used as a reference, which establishes that when exposed to pH conditions between 2,0 and 12,5 units, a sludge can generate gases, vapors or toxic fumes, and being reactive with concentrations higher than 250 mgCN^- kg^{-1} of waste for cyanides or 500 mgS^{-2} kg^{-1} of waste for sulfides (Feria and Martinez, 2014). Table 3 shows the results of the dangerousness of MO sludge according to their flammability and reactivity.

Table 3. Hazards from *Moringa oleifera* sludge by reactivity and flammability.

Parameter	Moringa oleifera sludge	Ceiling concentration Limits*
Inflammability	Negative	-
Reactivity	Negative	-
Reactive Cyanide mg CN^- kg^{-1}	<8,0	250 mg CN^- kg^{-1}
Reactive Sulfide mg S^2 kg^{-1}	21,2	500 mg S^{-2} kg^{-1}

* NOM-052-SEMARNAT-1993 (México).

Flammability, reactivity, reactivity due to cyanides and sulfides were negative or less than the criterion for dangerousness adopted, classifying MO sludge as nonhazardous to the environment.

Table 4 shows the results of the characterization of MO sludges by pathogenic microorganisms. A waste with infectious characteristics is considered dangerous because it can cause disease in humans, animals, and plants.

Table 4. Coliforms, salmonella and helminth eggs concentrations in MO slough.

Parameter	Moringa oleifera sludge	Ceiling concentration limits	
		Biosolids class A	Biosolids class B
Total Coliforms, CFU per g. biosolids (dry weight).	4000	-	-
Fecal Coliforms, CFU per g. biosolids (dry weight).	<3000	<1000	<2000
Salmonella sp, CFU per 25 g. biosolids (dry weight).	Absence/25g	Absence	<1000
Viable helminth ova, Number viable helminth ova per 4 g. biosolids (dry weight).	0	<1	<10

* Decree 4741/2005 (Colombia).

Concentrations of fecal coliforms in MO sludge showed that it cannot be classified as Class B or Class A material, since it contains concentrations of up to 3000 CFU g^{-1}, indicating that they require additional disinfection for agricultural use. However, the levels of harmful pathogens in the MO sludge are significantly lower compared to typical concentrations in sludge produced in the wastewater treatment plants (Elliot and Dempsey, 1991). For salmonella and helminth eggs, MO sludge meets the maximum allowed concentrations in Colombian law.

To achieve viability of MO sludge for agricultural use without pathogenic restrictions, hydrated lime was applied (alkaline stabilization) in order to increase the pH above 12 units and keeping it for at least 72 hours to achieve a significant reduction of pathogens. With these pH values, tolerance limits for the growth and survival of resistant organisms such as helminth eggs (EPA, 1993) is exceeded. Table 5 shows pH results according to the applied dosage shown and the time elapsed in the chemical stabilization assay.

Table 5. Performance of pH from hydrated lime doses applied to slough subsamples.

Time (contact hours)	Dosage of hydrated lime (%)				
	1	2	3	4	5
0	11,77	12,03	12,40	12,44	12,46
1	11,45	11,94	12,23	12,25	12,45
2	11,56	12,11	12,03	12,50	12,49
3	11,36	11,97	12,08	12,31	12,58
4	11,61	12,26	12,32	12,43	12,63
5	11,76	12,24	12,28	12,43	12,55
6	11,82	12,15	12,38	12,41	12,51
7	11,91	12,02	12,50	12,62	12,61
8	11,70	12,10	12,50	12,45	12,56
9	11,90	12,40	12,64	12,68	12,69
10	11,77	12,14	12,47	12,60	12,65
11	11,58	12,25	12,48	12,52	12,59
12	11,91	12,29	12,53	12,63	12,68
24	11,60	12,02	12,34	12,35	12,77
36	11,71	11,96	12,20	12,30	12,60
48	11,72	11,98	12,14	12,26	12,64
60	11,68	12,01	12,25	12,37	12,69
72	11,54	12,00	12,30	12,27	12,72
Mean	11,69	12,10	12,34	12,43	12,6
Standard deviation	0,151	0,131	0,161	0,131	0,087
Coeff, of variation	1 %	1 %	1 %	1 %	1 %

The coefficient of variation among pH data for each applied dosage was 1 %, which indicates homogeneity through the time period evaluated. However, when applying one-way ANOVA to the results of Table 5, it was found that the "P" value is less than 0,05, meaning that there is a statistically significant difference, with a confidence level of 95 %, when different dosages of lime are applied to the sludge.

The dosages of hydrated lime, which kept the pH above 12 units during the test time, were those with concentration equal to or greater than 3 % (w/w). The alkalized MO sludge, besides being a material containing organic matter and usable nutrients for agricultural use, which means a reduction of production costs by decreasing acquisition of fertilizer, has a high reactive power on the soil due to the remaining alkalizing effect. For this reason, its use would be focused mainly on soils having acid pH (Torres *et al.,* 2008).

Table 6 shows the concentrations of pathogenic organisms in the subsamples of sludge, stabilized with hydrated lime after applying different dosages.

Table 6. Concentration of total and fecal coliforms after applying dosages of lime to the sludge subsamples.

Parameter Pathogenic organisms	Dosage of hydrated lime (%)				
	1	2	3	4	5
Total Coliforms, CF per g. biosolids	470	250	0	0	0
Fecal Coliforms, CFU per g. biosolids	130	84	0	0	0

In the samples treated with hydrated lime with dosages equal to or greater than 3 % there was no presence of pathogenic microorganisms in the MO sludge. This condition is similar to that found in other studies (Torres *et al*., 2008; Plancha *et al.,* 2008). The results showed that stabilization with hydrated lime is an effective method for sludge disinfection treatment containing pathogenic bacteria. The above confirms the recommendation of the US Environmental Protection Agency to keep a pH greater than 12 units for more than 3 days, even if the measured temperature was lower than 52°C (Madera *et al.,* 2011).

Conclusions

According to the physicochemical characteristics found in MO sludge, its application for crop soils is possible. However, because of poor levels of macronutrients and high levels of micronutrients, it cannot be classified as a fertilizer but as a soil improver; particularly in soils that have deficiencies of trace elements such as Mn, Zn, Cu and Fe.

Alkaline stabilization of MO sludge with hydrated lime in proportion equal to or greater than 3 %, allowed raising and maintaining the pH to 12 units for 72 hours. This ensured an efficient reduction of pathogenic microorganisms and obtaining a class A sludge, that is, without any restrictions for use on soils suitable for agriculture, and especially recommended for acid soils.

The use of saline extracts of MO for purification of raw water not only represents an efficient and safe coagulant for the process, but also the by-products of the treatment, i.e. the sludge is useful as soil improvers and its disposal is environmentally sound, economical, and helps the improvement of soils that are acid and poor in micronutrients.

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