

1 **EVALUATION OF CONTAMINANTS IN AGRICULTURAL SOILS IN AN**
2 **IRRIGATION DISTRICT IN COLOMBIA**

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17

18 **Abstract**

19 This study evaluated the concentration and distribution of heavy metals (HM) (Cr, Ni, Pb,
20 Cd, Hg, and Zn) and pesticides (organochlorine and organophosphorus) and the relationship
21 of these pollutants with the physicochemical properties of agricultural soils in an Irrigation
22 District (ID) in Colombia. Soils samples were analyzed for pH, humidity, organic matter, P
23 total, N total, electric conductivity (EC), cation exchange capacity, and texture (% sand, clay
24 and silt). Canonical correlation was used to determined relationship between soil properties
25 and HM. Soil pollution were evaluated with geoaccumulation index (Igeo), contamination
26 factor (CF), degree of contamination (Cdeg) and pollution load index (PLI). The results
27 indicated that, in general, the soils had adequate physicochemical conditions for the
28 establishment and development of crops. The presence of pesticides in the soils was not
29 reported. However, concentrations HM was detected (Zn>Cr>Ni>Pb>Hg>Cd). The soil
30 characteristics (silt, clay, pH and EC) contributed to explain HM concentrations. The Igeo
31 indicated that the soils are heavily contaminated with Hg (3<Igeo<4). The CF was very high
32 for Hg (>6). The Cdeg presented moderate to considerable variations (>6Cdeg<24). The PLI

33 indicated that the soils are contaminated (1.308). The presence of HM may be associated
34 with the agricultural and quarries activities carried out near the ID. The impact caused by
35 high concentrations of HM can lead environmental, economic and social impacts in the study
36 zone.

37 **Key words:** heavy metals, pesticides, contamination index, soil properties.

38 **Introduction**

39 Soil is the most important basic natural resource for the support of agricultural production
40 systems (De Alba *et al.*, 2003) and the maintenance of productivity in these ecosystems
41 depends on their physicochemical and biological characteristics (García *et al.*, 2012;
42 Martínez-Mera *et al.*, 2017). However, soil is very sensitive to environmental variations
43 (Chen *et al.*, 2010). The anthropogenic activities of mining, changes in the use of soils and,
44 use of agrochemicals in conventional agriculture have altered physicochemical properties,
45 decreased edaphic populations and increased concentrations of some pollutants (Jaurixje *et*
46 *al.*, 2013). In this sense, agricultural production systems are a source of pollutants and,
47 according with the physicochemical characteristics of the soil, facilitate their transfer through
48 soil-plant, soil-groundwater and surface-soil water (Kabata- Pendias, 2011).

49 The pollutants in agricultural soils include HM and pesticides (Marković *et al.*, 2010). HM
50 are naturally present in small quantities or traces in the Earth's crust, soils and plants. Many
51 of them are essential for the growth and development of plants, animals and humans (Galán-
52 Huertos and Romero-Baena, 2008; Marković *et al.*, 2010). These natural concentrations can
53 be affected by the implementation of synthetic fertilizers and pesticides, manure and
54 conventional solid waste compost (Wu *et al.*, 2012; Alloway, 2013). High concentrations of
55 HM can affect the environmental health of ecosystems, placing biota at risk (Galán-Huertos
56 and Romero-Baena, 2008). On the other hand, pesticides are used to combat, repel and/or
57 prevent unwanted organisms (plants or animals) during agricultural production (Gilden *et al.*,
58 2010). In Colombia, the intensive use of these products has increased the degradation of
59 agricultural soils (Silva and Correa, 2009). Chemical agriculture models that use synthetic
60 products such as fertilizers and pesticides are an important source of pollutants in edaphic
61 systems (Rueda-Saá *et al.*, 2011; Jiao *et al.*, 2012).

62 Several studies have been carried out on the evaluation of physicochemical characteristics in
63 soils and the presence of contaminants (Silviera *et al.*, 2003; Alloway, 2013; Simón *et al.*,
64 2013). Particularly in Colombia, progress has been made mainly in the departments of
65 Atlántico and Córdoba (Yacomelo, 2014; Roqueme *et al.*, 2014; Marrugo-Negrete *et al.*,
66 2017). The state of physicochemical properties in soils determines the quality and health of
67 the soil because processes of adsorption, transport and degradation of contaminants depend
68 on these characteristics (Bautista-Cruz *et al.*, 2004). In fact, the physicochemical properties
69 of soils, such as pH, OM, texture, mineralogy of clays, potential oxide reduction, carbonates,
70 salinity and iron and manganese oxides and hydroxides, allow for the precipitation,
71 dissolution and solubility of metals and pesticides (Galán- Huertos and Romero-Baena,
72 2008).

73 The use and proper management of soils is the most important method to conserve the soil
74 environment. Otherwise, processes are generated that cause, not only loss of productive
75 capacity, but also negative environmental, social and economic impacts (UPRA, 2013).
76 Therefore, in the present study, the concentration of pollutants (HM and pesticides) was
77 evaluated, as well as the relationship with physicochemical characteristics of agricultural
78 soils in the South of the Atlántico Department. Studies are needed for a baseline for the
79 definition of sustainable management strategies in this type of ecosystem because Colombia
80 does not have a regulatory framework for concentrations of contaminants in soils (Rueda-
81 Saá *et al.*, 2011).

82 **Methodology**

83 **Description of the study area**

84 The municipality of Repelon is located to the west of the department of Atlántico (10° 29'40"
85 N and 75° 08'27" W), with a surface area covers 35,172 ha (10.6% of the total area of the
86 department of Atlántico). The Guájaro reservoir (10° 42 'N and 75° 6' W) stands out, a lentic
87 body that supply the community for drinking water and for different productive activities. In
88 its beginnings, the reservoir was capable of storing about 400,000,000 m³ of water, in an area
89 of 16,000 ha with five meters of average depth. Currently, El Guájaro has an extension of
90 11,647 ha, a perimeter of 114.28 km and an effective volume of 240,000,000 m³ (Figure 1)
91 (IDEAM, 2017). There are quarries in the middle and North area of El Guájaro, from which

92 is extracted mainly construction material (sand, clay and limestone for cement, gravel and
 93 stone) (Carrillo and Cajuste, 1995). The most important economic activity in the region is
 94 the agriculture (CRA, 2014). An irrigation system (Figure 1) was constructed to transport
 95 water to the crops. This ID covering an area 4,000 ha, and it is supplied by the waters from
 96 the El Guájaro reservoir through a catchment channel. The irrigation system consists of a
 97 pumping station that transports water to two channels. From there, the upper distribution
 98 channel (15 km long) and lower channel (12 km long) facilitate irrigation through gravity
 99 (CRA, 2014). In this area, the main crops are, i.e. cotton, tomato, corn, sorghum, cassava,
 100 banana, rice, guava, papaya, and mango. The agricultural activity of transitory crops
 101 intensifies during the months of August-January (Alcaldía de Repelón, 2016).

102 As for the hydrometeorological conditions of the area, the temperatures are between 28 and
 103 32°C. Rainfall varies according to the time of year: during the dry season (January-July), the
 104 average is 39 mm and, in the rainy season, it is 117.2 mm (August-December), with an annual
 105 average of 50 mm (Climate, 2017). However, the hydrometeorological conditions of the area
 106 result from the marked interannual variability, produced by the climatic anomalies El Niño
 107 and La Niña (Ruíz -Cabarcas and Pabón-Caicedo, 2013).

108 In terms of the general characteristics of the agricultural soils in southern Atlántico, the
 109 Repelón ID is located in quaternary deposits that occupy areas with a flat relief. The western
 110 side of the southern of Atlántico department has 50% of soils formed from clay sedimentary
 111 materials that are low in evolution, superficial, well drained, low to moderate in fertility and
 112 susceptible to erosion. These soils according to the texture, are classified as sandy loam clay
 113 and sandy loam at a depth of 0-30 cm and belong to the Inceptisol order. Table 1 describes
 114 the classification of the agricultural soils of the Repelón ID, suggesting a capacity for use,
 115 productivity and physicochemical characteristics (IGAC, 2008).

116 **Table 1. Soil classification of the Repelón ID (IGAC, 2008).**

Soil	Class I	Class II	Class III	Class IV
Topography	Plains, with deep parts	Moderately to strongly inclined, deep light erosion	Broken topography	Moderately steep, broken with moderate erosion
Drainage system	Moderate	Moderate-Good	Well drained	Moderate to excessive

Nitrogen	Poor	Poor	Poor	Poor
Phosphorus	High	High	Regular	Regular
Potassium	High	High	High	High
Salinity	Medium	Medium	Medium	Medium
pH	Neutral	Alkaline	Slightly alkaline	Slightly alkaline - acid
Crops	Tobacco, beans, corn	Grass	Grass	Grass
Observations	Improve drainages to avoid salinity in roots	Soils near the head. Heavy textures	Soils located in high parts of the municipality	No agricultural aptitude for high permeability is recommended crops of pasture or paddocks

117

118 **Field phase**

119 The field area was divided in three zones: northern zone (soil samples 1 to 4), central zone
 120 (soil samples 5 to 8) and, southern zone (soil samples 9 to 10). In areas with a history of
 121 agricultural activity, during the dry season a total of 10 topsoil samples (0–30 cm depth) were
 122 collected, which were obtained after the removal of the plant material from the soil surface,
 123 each soil sample comprised a composite of three subsamples. The characteristics and the
 124 locations of the sampling sites are shown in Table 2 and Figure 1. Samples for the
 125 physicochemical parameters and HM were stored in polyethylene bags. The samples for the
 126 pesticide analysis were wrapped in aluminum foil before being packed in polyethylene bags.
 127 All samples were transported under a controlled temperature (4 ± 1 °C) to the environmental
 128 laboratory of the Universidad de la Costa (CUC).

129 Table 2. Locations and description of the land uses during the sampling in the Repelón ID.

Soil Sampling	Longitud	Latitud	Types of Plantations
S1	-75.101639	10.509972	Forestry tree with grass
S2	-75.107222	10.519889	Fallow
S3	-75.112106	10.509928	Fallow
S4	-75.122989	10.517156	Annual crops
S5	-75.135569	10.488639	Annual crops
S6	-75.133444	10.483028	Annual crops

S7	-75.138222	10.469369	Fallow
S8	-75.135444	10.464306	Annual crops
S9	-75.142972	10.459889	Fruit tres, Fallow
S10	-75.132278	10.448222	Fallow

130

131 **Laboratory phase**

132 The physicochemical parameters were determined: moisture with the gravimetric method
 133 (IGAC, 2006), pH with the potentiometric method (NTC 5264, 2008), electrical conductivity
 134 ($EC_{1:5}$) with a conductivity meter (model EC300), total N (N total) with the Kjeldahl method
 135 (NTC 5889, 2011), organic matter (OM) from total organic carbon (Walkley-Black method)
 136 using a conversion factor ($\%C * 1.74$) (IGAC, 2006), and cation exchange capacity (CEC)
 137 with the saturation method with ammonium acetate (NTC 5268, 2014). Additionally, the
 138 texture parameters (Bouyoucos-Densimeter Hydrometer) and total phosphorus (Pt) (Olsen *et*
 139 *al.*, 1954) were determined in a certified laboratory (Zonas Costeras S.A.S).

140 Pesticide samples were analyzed with gas chromatography with an electron capture detector
 141 (CG-ECD), using EPA Method 8081B (US-EPA, 2007a) and 8141B (US-EPA, 2007b) for
 142 organochlorine and organophosphorus pesticides, respectively. The detection limit was 2
 143 $\mu\text{g/Kg}$ for organochlorine pesticides and 5 $\mu\text{g/Kg}$ for organophosphorus pesticides. The
 144 quantification of HM zinc (Zn), nickel (Ni), cadmium (Cd), lead (Pb), and chromium (Cr),
 145 0.5g of soil samples were digested with HNO_3/HCl 8:2 v/v in a microwave using EPA
 146 Method 3051A (US-EPA, 2007c). Additionally, concentrations of Hg heavy metal were
 147 evaluated using EPA Method 7471B, where 0.5g of soil sample was digested whit
 148 $\text{H}_2\text{SO}_4/\text{HNO}_3$ 7.3v/v and 5% w/v KMnO_4 at 100 °C for 1h (US-EPA, 2007d). The detection
 149 limits (mg/Kg) varied for the metals (Zn = 5.0, Cr = 3.0, Ni = 20, Pb = 0.07, Cd = 0.002 and
 150 Hg = 0.001). The pesticides and heavy metals concentrations were calculated as the mean
 151 from triplicate determinations. These analyzes were carried out in the Toxicology and
 152 Environmental Management Laboratory of the Universidad de Córdoba-Colombia.

153 **Quantification of soil pollution**

154 ***Geoaccumulation Index (Igeo)***

155 The Igeo for the metals were determined using:

$$156 \quad I_{geo} = \log_2 \left[\frac{C_n}{1.5 B_n} \right]$$

157 There C_n is the concentration of metal examined in soil samples and B_n is the geochemical
158 background concentration of the metal (n). Factor 1.5 is the background matrix correction
159 factor due to lithologic. This index ranges from <0 uncontaminated, $0 < I_{geo} < 1$ low
160 contamination, $1 < I_{geo} < 2$ moderately contaminated, $2 < I_{geo} < 3$ highly contaminated, 3
161 $< I_{geo} < 4$ heavily contaminated, $4 < I_{geo} < 5$ very heavily contaminated and > 6 extremely
162 contaminated (Müller, 1981).

163 ***Contamination Factor (CF)***

164 The level of contamination of soil by metal is expressed in terms of a CF calculated as:

$$165 \quad CF = \frac{C_{metal}}{C \text{ background value}}$$

166 Where C_{metal} is the concentration of metal examined in soil samples and C background
167 value is the geochemical background concentration of the metal. This index ranges from CF
168 < 1 refers to low contamination; $1 < CF < 3$ means moderate contamination; $3 < CF < 6$
169 indicates considerable contamination and $CF > 6$ is very high contamination. The CF is a
170 single element index (Hakanson, 1980).

171

172 ***Contamination Degree (Cdeg)***

173 The $Cdeg$ represents the sum of all pollution factors for all elements examined in a given site
174 and it is determined using:

$$175 \quad Cdeg = \sum_{i=1}^n CF$$

176 Where n are all the metals evaluated. There are four classes: $Cdeg < n$ (low degree of
177 contamination), $n < Cdeg < 2n$ (moderate), $2n < Cdeg < 4n$ (considerable) and $Cdeg > 4n$ (very
178 high degree of contamination) (Hakanson, 1980).

179 ***Pollution Load Index (PLI)***

180 The PLI is calculated by obtaining the n-root from the CF that were obtained for all the metals
181 studied (n). The PLI was determined using (Tomlinson *et al.*, 1980):

$$182 \quad PLI = \sqrt[n]{CF1 * CF2 * \dots * CFn}$$

183 According to the CF value, the PLI was categorized as <1 uncontaminated and >1
184 contaminated; with this value the relationship with soil quality was determined (Iqbal *et al.*,
185 2016).

186 Taking in account that in Repelón Municipality (Colombia) does not have local information
187 about values of background or natural concentration elements, the values of background were
188 taken from the limits proposed by the NOAA, which presents screening concentrations for
189 contaminants (Buchman, 2008).

190 **Information Analysis**

191 Multivariate cluster analysis is presented in a dendrogram for metals and soils. The normality
192 of the data was evaluated prior using Shapiro-Wilk test (Yap and Sim, 2011). As HM
193 concentrations and soil properties did not show normal distribution, values were log-
194 transformed. With the log-transformed data a multiple comparison was evaluated using a
195 canonical correlation analysis, and find out the contribution of physicochemical properties
196 of soils and heavy metals. Statistical analysis was performed with R.

197 **Results**

198 **Physicochemical parameters**

199 The physicochemical characteristics of the soil samples are shown in Table 3. The soils
200 showed differences between the sites sampled. The pH was found to be slightly acidic (6.4)
201 to slightly alkaline (7.2). The soil moisture was low, between 0.91-5.99%. The organic matter
202 and total phosphorus presented values between 2.90-6.45% and 76.2-113.0 mg/Kg,
203 respectively, which were high (IGAC, 2008; Martínez *et al.*, 2008; Novello and Quintero,
204 2009). The total N of the soils varied from very low (0.09%) to low (> 0.15%) (Hoskins,
205 2017). On the other hand, the EC_{1:5} presented values <0.35 in all of the soil samples,

206 indicating that the soils do not have salinity problems (Andrades and Martínez, 2014). The
 207 CEC was high (25-40 meq/100g) (Castellanos, 2016). Finally, three texture groups were
 208 found: silty clay loam (14.3% sand, 45.8% silty, 49.9% clay) and clay (14% sand, 39.8%
 209 silty, 46.2% clay) in the northern zone; silty clay (9.6% sand, 44.3% silty, 46.1% clay) in the
 210 central zone; and clay (6.5% sand, 38.4% silty, 55.1% clay) in the southern zone.

211 Table 3. Physicochemical properties of the agricultural soils in the Repelón ID.

Soil sampling	pH	Humidity (%)	Organic matter (%)	P (mg/Kg)	N (%)	EC (dS/m)	CEC (meq/100g)	Texture
S1	7.2	4.25	2.90	76.2	0.15	0.10	35.4	sicl
S2	6.8	3.95	5.20	90.6	0.07	0.03	35.3	sicl
S3	6.9	5.77	4.05	94.4	0.11	0.01	41.4	c
S4	7.2	3.71	6.00	98.5	0.26	0.02	29.2	c
S5	7.2	1.48	5.60	101.6	0.12	0.07	39.3	sic
S6	7.2	4.81	5.97	113.0	0.11	0.02	48.8	sic
S7	6.5	0.91	3.42	106.3	0.26	0.04	39.1	sic
S8	7.2	5.99	6.45	108.5	0.30	0.01	47.2	sic
S9	6.4	2.43	3.83	102.8	0.21	0.01	54.4	c
S10	6.4	5.61	3.58	111.3	0.16	0.04	51.9	c

212 EC (Electric Conductivity); CEC (Cation Exchange Capacity); Texture: sicl (silty clay loam),
 213 c (clay), sic (silty clay).

214 Pesticides

215 The presence of organochlorine pesticides such as heptachlor benzene, aldrin, endosulfan,
 216 dieldrin, endrin, and 4,4'DDT and organophosphorus pesticides such as malathion,
 217 chlorpyrifos-methyl, fention, tridemofon, diazinon, cis-chlorfenvinfos and dimethoate was
 218 not reported. The results obtained were below the detection limit of the equipment ($<2 \mu\text{g/Kg}$
 219 for organochlorine pesticides and $< 5 \mu\text{g/Kg}$ for organophosphorus pesticides).

220 Heavy metals

221 The concentrations of HM showed variations in the soil samples. The average levels of HM
 222 in the agricultural soils follow a decreasing order as: $\text{Zn} > \text{Cr} > \text{Ni} > \text{Pb} > \text{Hg}$. The HM Zn,
 223 Cr, Ni and Cd showed a similar pattern of concentrations in the field area. In general, highest

224 concentrations (Zn, Cr, Ni, Hg and Cd) were found in the southern area except for the Pb that
 225 showed high concentration in the central zone. Additionally, northern and central zones had
 226 little differences in concentrations for Zn, Cr, Ni and Cd. Similar pattern was showed for Pb
 227 in northern and southern zone. Finally, Hg showed variations between central (high
 228 concentration) and northern zone (low concentration) in comparison with southern zone.

229 On the other hand, the HM studied were highest than the world soil reference (NOAA) (Table
 230 4). Concentrations of Hg were 8.5 times higher than reference, followed by Ni (3.3 times),
 231 Zn (2.01 times), Cr (1.84 times), Pb (0.34 times) and Cd (0.2 times) that was the HM in
 232 lowest concentration.

233 Table 4. Heavy metals concentrations in agricultural soils from Repelón (Colombia).

Soil sampling	Heavy metals (mg/Kg)					
	Zn	Cr	Ni	Pb	Hg	Cd
S1	94.1	68.5	44.2	4.94	0.09	0.33
S2	99.2	70.4	47.0	5.18	0.09	0.33
S3	91.5	73.8	45.2	5.19	0.10	0.29
S4	75.1	51.0	34.5	3.88	0.74	0.19
S5	94.5	68.0	40.0	4.70	0.90	0.30
S6	92.5	65.9	42.5	6.51	0.92	0.31
S7	109.3	70.6	42.6	5.69	0.10	0.24
S8	98.1	66.6	43.0	5.15	0.51	0.32
S9	103.3	71.6	46.4	6.14	0.73	0.43
S10	109.4	73.9	47.2	6.38	0.69	0.43
Mean	96.7	68.0	43.3	5.38	0.49	0.32
NOAA¹	48	37	13	16	0.05	1.6

234 ¹ Background (mg/Kg)

235 **Geoaccumulation index (Igeo):** The concentrations of Pb and Cd did not represent
 236 contamination in the agricultural soils of the Repelón ID. The HM Zn and Cr presented a
 237 slight degree of contamination, Ni had moderate contamination and Hg had a degree of strong
 238 contamination (Figure 2).

239 **Contamination Factor (CF):** The HM in the Repelón agricultural soils presented variations
 240 in the CF. Figure 3 shows the CF for the evaluated soils. The CF was low for Cd and Pb (0.20

241 and 0.33, respectively); moderate for Cr and Zn (1.84 and 2.04, respectively); considerable
242 for Ni (3.33) and very high for Hg (8.45).

243 **Contamination Degree (Cdeg):** The Cdeg in the northern zone varied between moderate
244 (9.56) to considerable (18.7) where the soil sample S4 presented high contamination degree
245 in this zone. In the central zone, the Cdeg varied between considerable (21.0) to moderate
246 (9.72), in this zone the soil sample S6 showed highest level of Cdeg of all soils. Finally, in
247 southern zone the Cdeg was considerable (20.7).

248 **Pollution Load Index (PLI):** In this study, the evaluation of the overall toxicity status of all
249 soil samples were $PLI > 1$, this value suggest that quality of the agricultural soil is
250 deteriorating (Mir-Mohammad *et al.*, 2016). The pattern of pollution in each soil sample was
251 similar to Cdeg, where soil samples S4 and S6 showed higher values, as the same manner in
252 southern zone.

253 **Multivariate cluster analysis**

254 Dendrogram in Figure 4a. enabled the identification of two major clusters. Cluster 1 is
255 composed of Pb, Hg and Cd. The second cluster is composed of Cr, Ni y Zn. Similarly,
256 sampling points were also analyzed by clustering methods and organized in the dendrogram
257 to identify similar groups (Figure 4b). The sampling sites could be grouped in three clusters,
258 with the majority in cluster 1 with 7 samples, and cluster 2 with 2 samples. In addition, among
259 all the sampling sites, 70% represent cluster 1, with a similar percentage of samples located
260 in the north and center zone of the Repelón ID (Figure 4).

261 **Canonical correlation analysis**

262 The correlations between physical-chemical soil characteristics (G1) and HM concentrations
263 (G2) in the soil samples are shown in Table 5. Six canonical correlations were obtained (L1-
264 L6), of these correlations only L1, L2 and L3 were statistically significant (P-value <0.01).
265 The high correlation coefficient (near 1) means a good relation between variables, if R^2 is
266 more than 0.7, it can be strongly correlated. In L1, the variability (99%) was explained by
267 silt, clay and Cd, they had the biggest positive coefficients. Additionally, Ni and Hg had a
268 high negative standardized coefficient. In L2, the 99% of the variability was explained by
269 silt, Hg, Zn and Cr. Whereas EC had a high negative standardized coefficient. Finally, in L3

270 the 95% of the variability was explained by pH, EC, Ni and Hg. Conversely, Cd, Cr and Pb
 271 had a high negative standardized coefficient.

272 Table 5. Canonical correlations between physical-chemical soil characteristics and heavy
 273 metals concentrations in the agricultural soils of the Repelón ID.

Groups	Characteristics	Canonical correlations						
		L1	L2	L3	L4	L5	L6	
G1	pH	0.24	0.15	1.18	0.35	-0.05	0.18	
	Humidity	-0.40	0.48	-0.15	-0.22	0.12	-0.66	
	Organic matter	0.55	-0.20	-0.30	-0.34	-0.17	-0.52	
	CEC	0.41	-0.92	0.88	1.42	-0.22	0.98	
	EC	-0.38	-0.23	0.20	-0.83	-0.15	0.83	
	Total N	0.16	0.00	0.37	-0.66	0.39	0.21	
	Total P	0.19	-0.05	0.10	0.08	0.45	0.82	
	Texture	Sand	0.36	0.49	0.04	2.27	0.80	1.29
		Silt	1.40	0.74	0.47	3.92	0.02	3.82
Clay		1.12	0.12	0.36	1.27	-0.68	2.64	
G2	Zn	-0.46	0.68	0.09	0.40	-1.67	-0.81	
	Cr	0.09	0.65	-1.69	-1.05	1.90	-0.53	
	Ni	-2.94	0.06	4.80	-1.38	0.64	-0.44	
	Pb	0.59	-0.31	-1.19	0.14	-0.28	1.86	
	Hg	-1.81	0.97	1.60	-0.28	1.08	-0.53	
	Cd	3.16	-0.24	-1.86	1.40	-0.78	0.20	
	R ²	0.99	0.99	0.95	0.82	0.39	0.04	
	P-value	0.00	0.00	0.01	0.32	0.93	0.99	

274 P-value<0.01 Significant correlation.

275 Discussion

276 The soils of the Repelón ID presented adequate physicochemical characteristics. The pH
 277 presented values in the range of 6.5-7.5. In general, the pH of the soils was found within the
 278 range of acceptable values for the development of crops and the availability of nutrients
 279 (Andrades and Martínez, 2014). The low moisture content of the soils was related to the
 280 sampling period (dry season). Similarly, the high OM% is associated with the fallow where
 281 the vegetation cover of the soils (grass), which facilitates the accumulation of organic waste
 282 and the low precipitation prevents water erosion (Guangwei *et al.*, 2006). The Ptotal content
 283 in the soil can be altered by the removal of crops (approximately 80% is absorbed by the

284 plants), water erosion and OM mineralization (Suñer *et al.*, 2001; Novello and Quintero,
285 2009). However, in the Repelón soils, these factors did not influence this nutrient.
286 Conversely, the high content of P_{total} depend to the superficial horizon, where high
287 concentrations of this element are found because plant residues accumulate on the surface
288 (Novello and Quintero, 2009). The low N_{total} content was related to the variability in soil
289 temperature and moisture. High temperatures and low rainfall influence microbial
290 degradation. Therefore, these variables affect the N supply capacity of the soil. A low content
291 of salts can depend, to a large extent, on agricultural practices; during the dry season, the
292 application of fertilizers is very low. Finally, the high CEC was due to the absence of salts,
293 the texture (high clay content) and the OM present in the soil (Hoskins, 2017).

294 In this study, the soils were class I and II due their topography (IGAC, 2008), making them
295 suitable for agricultural activities, and the parameters N_{total} and P_{total} did not present
296 variation in the values previously reported (low and high, respectively). However,
297 parameters, such as pH and salinity, varied. The factors associated with changes in pH in the
298 soil were possibly related to the agricultural history of the soils although, currently the
299 economic agricultural activity is subject to the rainy season. The application of chemical
300 inputs was related to changes in the pH in the soil (Martínez-Mera *et al.*, 2017). Additonally,
301 La Niña phenomenon where the soils were flooded, the soil pH increased with flooding time
302 (Kashem and Singh, 2001). On the other hand, salinity in soils can be caused by natural or
303 anthropic processes. In the former, the soluble salts are found in the subsoil and rock deposits.
304 On the other way, the salinity generated by agricultural activities can be caused by inadequate
305 irrigation methods and the handling of chemical substances (McKenzie, 2013). Taking these
306 factors into account, the decrease in salinity in Repelón soils may be related to the decrease
307 in agricultural activities associated with climate change (dry season) or improvement of
308 agricultural practices over time.

309 Although the use of LorsbanTM 4E insecticide, glyphosate herbicide and NPK 15-15-15
310 (Fertilizer-Triple 15) (Martínez-Mera *et al.*, 2017) has been reported in the Repelón ID when
311 the agricultural activities increase during rainy season, the non-detection of organochlorine
312 and organophosphorus pesticides could be related four reasons: *i*). low agricultural activity
313 during the dry season, *ii*). climatic conditions of the municipality, *iii*). physicochemical

314 properties of the evaluated soils, and *iv*). environmental regulation. In terms of climatic
315 conditions characterized by high rainfall that in some periods have caused flooding (winter
316 wave 2010) (Ruíz -Cabarcas and Pabón-Caicedo, 2013); long periods of drought conditions
317 and high solar radiation, during 2016 the variation of rainy was 0.2 mm (January) and 256.11
318 mm (May) with temperature between 36 °C (March) and 32 °C (November) and average of
319 ultraviolet index 7-9 (very high harmful ultraviolet radiation) (Word Weather, 2018); these
320 conditions favor photolysis of pesticides and increased evaporation of volatile or semi-
321 volatile substances (Narváez *et al.*, 2012). Whereas, during periods of high rainfall, the soil
322 becomes saturated, favoring the leaching of pesticides (Uzcátegui *et al.*, 2011; Ruíz-Cabarcas
323 and Pabón-Caicedo, 2013). In relation to physicochemical properties, such as high OM and
324 clay, they have the ability to adsorb or immobilizing pesticides, leaving them unavailable for
325 biodegradation (Cornejo and Jamet, 2000). On the other hand, the Colombian Agricultural
326 Institute (ICA) in Resolution 2189 in 1974, prohibited the sale of fungicides that containing
327 phenylmercury, chemical inputs were use over 50 years ago. Additionally, in Resolution 366
328 in 1987, and Resolutions 531, 540, 723, 724 and 874 in 1988, prohibited the sale of
329 insecticides containing active ingredients such as aldrin, heptachlor, dieldrin, chlordane and
330 camphechlor; it is likely that these pesticides characterized by their persistence, have
331 degraded during the 28 years that have elapse and these regulations are being complied with.
332 The commonly used pesticides are classified as organophosphates, with high toxicity and
333 low chemical stability; therefore, they have high degradability (Silveira *et al.*, 2003). Taking
334 into account the fact that the average life span of Lorsban™ 4E (30-60 days) and glyphosate
335 (1-130 days) vary depending on the soil and climatic conditions, this condition is associated
336 with the absence of pesticides in the analyzed soils because the highest frequency of
337 application occurs during the rainy season.

338 The indicators to evaluate the contamination by HM presented variations, and showed that
339 in general the ID is contaminated. Pollution sources are associated with natural phenomenon
340 and anthropogenic activities. During 2010 the phenomenon of flooding soil for a long period
341 could provide availability of HM due little aeration of soil, reduction conditions are favored,
342 this condition increases the toxicity of some metals such as Mn (manganese), Cu (copper),
343 Zn, Cd, and Cr (Poot *et al.*, 2007; Reichman, 2002). On the other hand, the anthropogenic
344 activities where the municipality of Repelón is part of the Calamarí Mining District and has

345 27 active mining operations (where 9 are illegal), taking up approximately 50 ha in areas
346 surrounding the El Guájaro reservoir that generate problems in different bodies of water
347 (Alcaldía de Repelón, 2017). In the study area, there are quarries in the middle and north area
348 of El Guájaro, from which is extracted mainly construction material (gravel, sand, stone and
349 limestone), activity that possibly by erosive processes accumulate metals in particular areas
350 (CRA-CARDIQUE, 2002; CRA, 2014), which could reach the El Guájaro reservoir and,
351 later, the agricultural soils of the Repelón ID through the irrigation water. Belmonte-Serrato
352 *et al.* (2010) and Vallejo *et al.* (2016), reported that the extraction of mineral resources
353 (represented by quarries) contributes significantly to increase the concentrations of Zn, due
354 it contributes 10-15% of the total sediments delivered by laminar erosion, which are possibly
355 aggregated by atmospheric deposition and runoff (CRA, 2014). This result corroborate the
356 high concentration of Zn in comparison with the others HM. Additionally, Torregroza-
357 Espinosa *et al.* (2018), reported the presence of HM (Zn, Pb, and Hg) in water and surface
358 sediments of El Guájaro Reservoir, the highest levels were found in the southwest and
359 northern zones of this body of water, it is possible that the pollutants are associated with
360 agricultural and mining activity, floodgate (El Guájaro Reservoir with Repelón ID) and the
361 connection with the Canal del Dique (from Magdalena river) (Figure 1). On the other hand,
362 industrial activities (aquaculture and shrimp farm), municipal waste disposal and agricultural
363 activities, involving irrigation and applications of chemical substances, pesticides and
364 manure, can also be sources of HM (Belmonte-Serrato *et al.*, 2010; Marrugo-Negrete *et al.*
365 2017). The problems associated with irrigation systems depend on the quality of the water,
366 which can be contaminated by the main tributary or receive wastewater discharge, providing
367 heavy metals such as Ni, Pb, Cr, Cd, Zn, Cu, Hg, Mn, and Fe (iron), among others (Kim *et*
368 *al.*, 2015). The application of fertilizers that provide macronutrients (N, P, K) contain
369 impurities of Cd and Pb, which can significantly increase their content in the soil because of
370 regular use since they are fundamental for the growth of the plants. Likewise, phosphate
371 fertilizers provide Cd, Hg, Pb, Co, Cu, As and Zn as impurities (Gimeneo-García *et al.*, 1996;
372 Wuana and Okieimen, 2011; Alloway, 2013). Other fertilizers, such as copper sulfate and
373 iron sulphate, contain Pb and Ni (Gimeneo-García *et al.*, 1996). Even fertilizers from
374 animals, such as manure, can also provide As, Zn and Cu as a product of the animals' diet

375 (Andrades and Martínez, 2014). And some pesticides can also contribute As, Pb, Hg, Cu and
376 Zn (Alloway, 2013; Gaw *et al.*, 2006).

377 During the sampling, intensive applications of chemical products in the evaluated soils were
378 not reported. However, historically, the municipality of Repelón is known as the agricultural
379 pantry of the Atlántico Department. Therefore, the presence of HM in the soils cannot just
380 be associated with mining activities; it is likely that, during the winter season when
381 agriculture intensifies, common practices such as the application of chemical inputs are a
382 source of HM. Mining is responsible for soil degradation. Artisanal mining processes
383 significantly alter the landscape and cause air, soil and water pollution. Quarries are
384 associated with forced migration and loss of soil biodiversity, and destruction of fragile
385 ecosystems (Vallejo *et al.*, 2016).

386 Dendrogram for heavy metals enabled the identification of two major clusters. Cluster 1 is
387 composed with the heavy metals (Pb, Hg, Cd) that are related with anthropogenic activities
388 like wastewater, waste combustion and agricultural activities (fertilization and pesticide
389 application), specifically with the application of phosphate fertilizers (Marrugo *et al.*, 2017;
390 Wuana and Okieimen, 2011). And in the Cluster 2 heavy metals (Zn, Cr, Ni) have
391 relationship with the phenomenon of flooding soil generated by the floods of the Magdalena
392 River or mining activities of quarries, as previously explained. In the dendrogram for
393 sampling sites, two major clusters were observed. Cluster 2 shows the sampling site 1 to the
394 sampling site 7, with the exception of sampling site 4, possibly grouped by agricultural
395 activities. While the second cluster includes sites 9 and 10. These sampling sites are located
396 in the southern zone of the ID of Repelón, and presented high values in the concentrations of
397 all the metals evaluated. In this area, the Canal del Dique connects with El Guájaro Reservoir
398 through two floodgates (Figure 1), where the exchange of 50,000 tons/year of sediment to
399 the reservoir (CRA, 2014) which may explain why those soils are grouped. Similarly, that
400 zone was the most affected by the floods caused by La Niña phenomenon.

401 In the present investigation, a positive correlation was observed between silt and clay with
402 Cd, and a negative correlation between Ni and Hg, with silt and clay. In the second
403 correlation, a positive relationship was observed between silt and Hg, Zn y Cr, and a negative
404 relationship between CEC with Hg, Zn and Cr. In the third correlation a high negative

405 correlation was found between the pH and the Cd, Cr and Pb concentrations, and a positive
406 correlation between pH and Ni and Hg concentration. The soils properties play an important
407 role reducing or increasing the availability and toxicity of metals (Galán-Huertos and
408 Romero-Baena, 2008). In this sense, the positive correlation with silt and clay, may be
409 explained by the presence of clay minerals and Fe and Mn oxides associated forming silt
410 sized aggregates (Melo *et al.*, 2000). Additionally, clay presents adsorptive properties that
411 enhanced the retention of metals that decreases its bioavailability (Jung, 2008; Marrugo *et*
412 *al.*, 2017). The CEC is moderately related to the content of clay and OM, thus soils retain
413 heavy metal cations (Ahumada *et al.*, 1999; Marrugo *et al.*, 2017). In agreement with IGAC
414 (2008), the Caribbean soils evaluated presented high content of OM, the higher CEC has the
415 greater the capacity of the soil to fix metals (Ahumada *et al.*, 1999). On the other hand, the
416 negative correlation between CEC and metals concentrations, is possibly explained by the
417 formation of chelate complexes with the OM, due the high CEC (in a function of the content
418 of OM) influences the solubility and assimilation (Angelova *et al.*, 2013). The adsorption
419 and exchange of metals is generally attributed to the properties of the adsorbent, the solvent,
420 the concentration, the valence and the degree of hydration of the cations (Sparks, 2003). Ions
421 with a smaller hydration radius can get closer to the adsorption surface, and adsorption of
422 these can be favored. The average concentration of metals in the analyzed soils was Zn > Cr >
423 Ni > Pb > Hg > Cd; such sequence is not related to the hydration radius of the metals Pb (0.401
424 nm) < Ni (0.404 nm) < Cd (0.426 nm) < Zn (0.43 nm) < Cr (0.461). Finally, the pH affects
425 several mechanisms of metal retention in soils (Carrillo and Cajuste, 1995), and can be
426 considered the most important parameter that influences the processes of sorption-
427 desorption, precipitation and dissolution. Likewise, the formation of complexes and the
428 reactions of oxide-reduction where the bioavailability of the elements is inversely
429 proportional to soil pH (Narwal *et al.*, 1999; Basta *et al.*, 2005; Antoniadis *et al.*, 2008). It is
430 important to take in account that the bioavailability of the metal is determined by several
431 factors including mineralogy, content of matter, capacity of cation exchange and temperature
432 of the soil, additionally, the metal fraction and its location in the soil system (Takáč *et al.*,
433 2009; Nederlof *et al.*, 1993). For this problem, there are different methods for improvement
434 of contaminated soils like removal of contaminated soil, adding uncontaminated soil as a

435 topdressing, use of a soil conditioner depending on the heavy metal on the soil and,
436 improvement of water management (Iwata *et al.*, 1994).

437 In Latin America, Colombia is the third country with the highest water resources, climate
438 diversity and annual precipitation rates, characteristics that favor its role in food production.
439 Therefore, The United Nations Food and Agriculture Organization (FAO) affirms that
440 Colombia has great potential to be a pantry of the world. However, in order to supply the
441 world population, food production will have to be increased by 70% by the year 2050 (FAO,
442 2018). In this context, to ensure food security, regulatory measures should be implemented
443 for the use of chemical products in agricultural activities due it is a problem in areas of great
444 potential and the production, besides being not sustainable and causes serious environmental
445 damage. Particularly, it is probably that in southern zone of the ID of Repelón, the low
446 populations of nitrogen fixing bacterial reported by Martínez-Mera *et al.* (2007) are
447 associated with the soil properties and high concentrations of heavy metals compared with
448 the entire study zona. Puga *et al.* (2006) affirm that the problems with heavy metals including
449 loss of diversity, decrease of the biological potential of the soil, zero agricultural productivity,
450 and effects on public health as chronic-degenerative diseases in people, among others.

451 **Conclusions**

452 In the studied agricultural soils, no organochlorine or organophosphorus pesticides were
453 detected, possibly the scarce agricultural activity at the time of sampling, the variability of
454 the climatic conditions of the area that could favor the natural attenuation of the pesticides
455 and the characteristics of the soils and/or compliance with agricultural regulations. In
456 contrast, the concentration of HM in the soils varied as follows Zn>Cr>Ni>Pb> Hg>Cd. The
457 pollution indicators showed contamination in the study area, that may be related to
458 anthropogenic activities (quarries activities, agricultural practices and, urbanization). And
459 the natural phenomenon such as high rainfall and flooding of soils. Likewise, the canonical
460 analysis showed in the first correlation a positive correlation between silt and clay with Cd,
461 and a negative correlation between Ni and Hg, with silt and clay. In the second correlation
462 showed a positive relationship was observed between silt and Hg, Zn and Cr, and a negative
463 relationship between CEC with Hg, Zn and Cr. In the third correlation a high negative

464 correlation was found between the pH and the Cd, Cr and Pb concentrations. Soil properties
465 like OM, CEC, silt and, clay contributes to HM retention by decreasing tis bioavailability.

466 This research provides knowledge on the environmental health of the agricultural soils of the
467 Repelón ID. Information that can serve as a tool for defining strategies for sustainable
468 management for the implementation of monitoring plans to quantify the vulnerability of the
469 ecosystem and potential risk to human health being that the population depends on these
470 natural resources for the development of their economic activities.

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478

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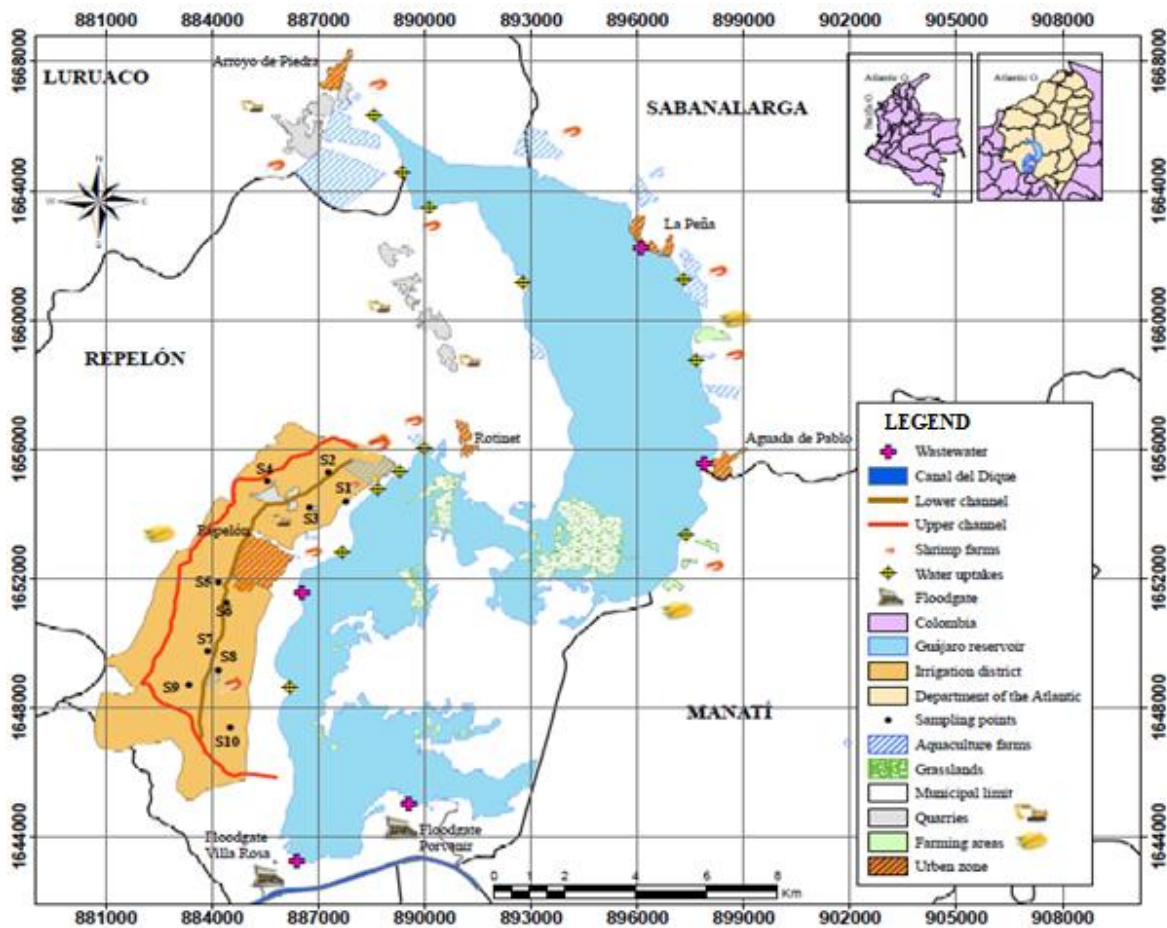
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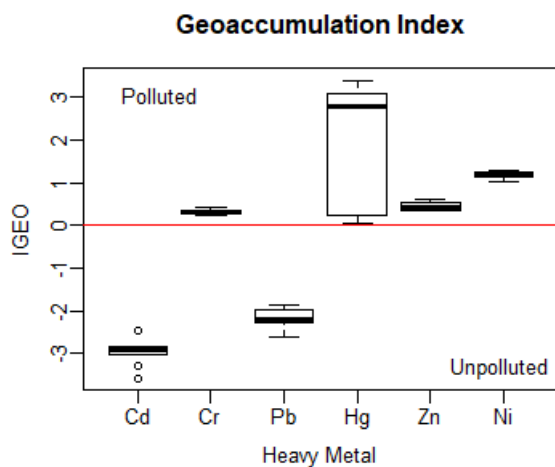
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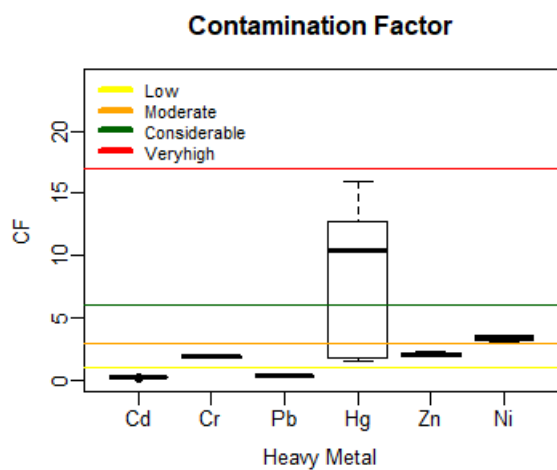
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698 Figure 1. General location of the ID of Repelón.



699

700 Figure 2. Geoaccumulation index (Igeo) of the metals analyzed in the agricultural soils of
701 the Repelón ID.



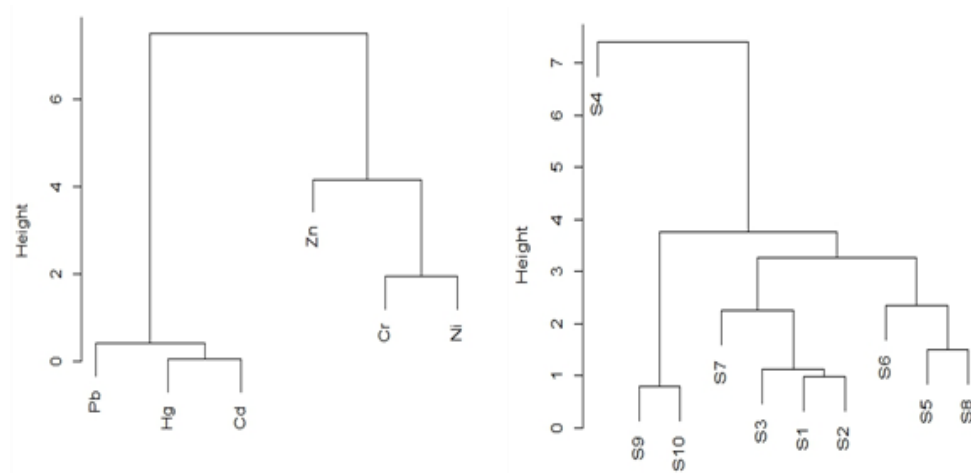
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703 Figure 3. Contamination Factor (CF) of heavy metals found in agricultural soils of the
704 Repelón ID.

705

A)

B)



706

707 Figure 4. Dendrogram obtained by hierarchical clustering analysis for (A) the heavy metals,
708 and (B) the sampling sites.