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Baseline

Heavy metal concentration in mangrove surface sediments from the north-west coast of South America

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ABSTRACT

Mangrove ecosystems are coastal estuarine systems confined to the tropical and subtropical regions. The Estero Salado mangrove located in Guayaquil, Ecuador, has suffered constant disturbances during the past 20 years, due to industrial wastewater release. However, there are no published data for heavy metals present in its sediments and the relationship with anthropogenic disturbance. In the present study, metal concentrations were evaluated in surface sediment samples of the mangrove, showing that B, Cd, Cu, Pb, Se, V, and Zn levels exceeded those declared in international environmental quality standards. Moreover, several metals (Pb, Sn, Cd, Ag, Mo, Zn and Ni) could be linked to the industrial wastewater present in the studied area. In addition, heavy metal levels detected in this mangrove are higher than previous reports on mangrove sediments worldwide, indicating that this mangrove ecosystem is one of the most disrupted on earth.

Mangrove ecosystems are unique coastal estuarine systems confined to the tropical and subtropical regions (Bayen, 2012). These zones are dominated by mangrove plants, which are highly productive and play a vital role, both as the major primary producers in estuarine ecosystems (Bayen, 2012; Wang et al., 2013), and by providing great ecological services (Lewis et al., 2011). Among the services provided are: protection from tidal events and erosion, as well as hosting approximately 4100 species of flora and fauna, presenting an overall economic value around 181 billion dollars (Alongi, 2002; Du Laing et al., 2009; Lewis et al., 2011). Also, mangroves generate economic benefits to adjacent human populations, mainly due to tourism and fishing (Bayen, 2012; Franco-Dias et al., 2012).

In spite of their great ecological relevance, mangrove forests are rapidly disappearing, with up to 50% being lost over the past 50 years due to direct anthropogenic interference (Bouillon, 2011). Today, less than 10% of the mangroves around the world are considered protected areas (Lewis et al., 2011). Due to their high ecological relevance, many cities have settled in nearby mangroves, which are, therefore, constantly exposed to severe disturbances (Valiela et al., 2001). These disturbances are caused by exposure to a variety of chemicals, including heavy metals, which are considered to be among the most potentially serious anthropogenic pollutants in mangrove ecosystems (Wang et al., 2013). In addition, heavy metals are particularly relevant because of their ability to accumulate in surface sediments, leading to a gradual increase in their concentration in this area (Bayen, 2012).

The content of heavy metals in sediments of various mangrove systems worldwide has been determined; for example, As, Cd, Co, Cr and Ni in Malaysia (Sany et al., 2013); Cu in the Gulf of California (Soto-Jiménez and Páez-Osuna, 2001); Pb in Portugal (Caeiro et al., 2005) and Zn in Puerto Rico (Mejias et al., 2013) among others. These studies make it clear that the long-term pollution processes observed in many mangroves are caused by human activities (Bayen, 2012; Tam and Wong, 2000), with a direct effect on the ecological conditions of the sediments (Essien et al., 2009), biogeochemical cycles (Lenoble et al., 2013) and primary production of the ecosystem (Bai et al., 2011; Bayen, 2012; El-Said and Youssef, 2013). In contrast, there are some mangrove systems with undetermined levels of heavy metals, such as the present in the northwest coast of South America.

The Gulf of Guayaquil is one of the most productive areas on the northwest coast of South America. This area contains 81% of the Ecuadorian mangrove system (Monserrate et al., 2011), covering 615 km of coastline. One of the principal parts of the mangrove, the Estero Salado, has been surrounded by Ecuador's most populous city, Guayaquil (2°11′44″S, 79°53′18″W). In this sense, the Estero Salado has suffered constant disturbances during the

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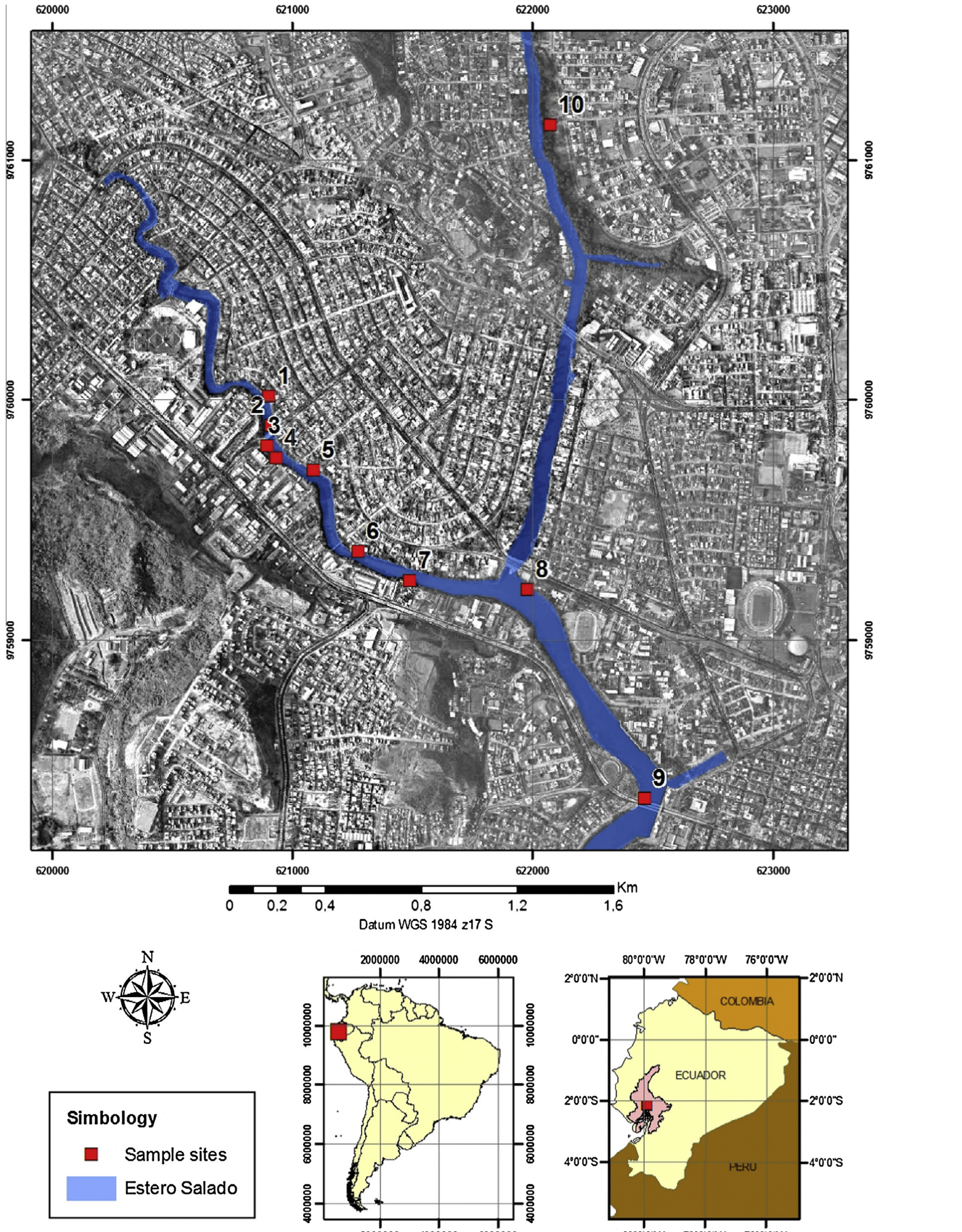

Fig. 1. Aerial photography showing the geographic localization of the 10 sampling sites analyzed in this study.

past 20 years, mainly due to industrial wastewater release (Monserrate et al., 2011).

The aim of this study was to determine the concentration and distribution of heavy metals in mangrove surface sediments from the Estero Salado and to contrast them with the presence of heavy metals reported for other mangroves worldwide. To evaluate associations between metals and to infer similar biogeochemical origins, a Pearson correlation matrix was performed. Furthermore,

Table 1
Percentages of recovery from reference material (MESS-3 Marine Sediment) to assess analytical quality.

Element analyzed	Percentage of recovery (%)
Li	94.5
Be	73.5
Al	85.7
V	93.4
Cr	104.2
Mn	100.4
Fe	79.6
Co	84.5
Ni	78.5
Cu	85.7
Zn	111.4
As	113.1
Se	116.7
Mo	86.0
Ag	72.2
Cd	119.8
Sn	49.2
Pb	87.4

to determine the possible origin of each metal (anthropogenic or crustal), enrichment factors (EF) were calculated using a chemical normalization against a representative element, in this case Aluminum (Al).

Surface sediment samples were collected from 10 different stations in the Estero Salado mangrove (Fig. 1). At each station, five samples were collected manually using an acid-washed PVC device (upper 10 cm) every 30 cm and along the low tide line. Sediments were stored in 10 ml polypropylene tubes cleaned with HNO_3 at 10%, dried at 37 °C for 3 days and kept at 4 °C until further analysis. Sediment samples were oven dried overnight at low temperatures (50 ± 5 °C) up to constant weight. Concentration of 20 elements (Li, Be, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Mo, Ag, Cd, Sn an Pb) was determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Thermo Fisher Scientific Finnigan X series 2) after digestion of 500 mg of sediment with a mix of concentrated bi-distillate nitric acid and hydrogen peroxide (7:1), using a closed-vessel microwave sample digestion system (ETHOS ONE, Milestone). Analytical grade reagents were used for the corresponding blanks, samples and calibration curves, while analytical quality was checked against a reference material (marine sediment, MESS-3) provided by the National Research Council, Canada (NRC-CNRC) for all metals except B and Ba. Percentages of recovery in the analysis of certified reference material (CRM) are shown in Table 1.

The enrichment factor for 17 elements (Li, Be, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Mo, Ag, Cd, Sn and Pb) was calculated according to the following equation (Herut and Sandler, 2006), where *X* is the metal concentration in the sample(s) and in the reference sediment material – MESS-3 (b).

$$\mathrm{EF} = \frac{X(\mathrm{s})/\mathrm{Al}(\mathrm{s})}{X(\mathrm{b})/\mathrm{Al}(\mathrm{b})}$$

Average concentrations of metals determined in surface sediments from the Estero Salado mangrove are shown in Table 2, together with values of Sediment Quality Criteria for the classification of sediments from the marine water quality in Hong Kong (EPD) (http://www.devb-wb.gov.hk), and the values proposed by the Environmental Quality Standard for Contaminated Soils of Ecuador (EQS) (MAE, 2003). In general, sampling points present a homogeneous distribution pattern of heavy metal concentrations, with values of Co, Ni, V and Zn exceeding those stipulated by either EQS and/or EPD. Maximum values of heavy metals recorded in sediments from several mangroves around the world, including those in the present study, are presented in Table 3. Heavy metal concentrations obtained here far outweigh those obtained for mangroves with no evidence of a pollution process (Ciénaga Grande, Colombia and Sai Kung, China; see Table 3). Moreover, it can be observed that levels of Zn, Cu, Ni, Co, Cr, Ag and V in this ecosystem were higher than those previously reported for any other mangrove sediment, covering a wide range of perturbations, from industrial activities to agriculture, fisheries and transport (Table 3).

There is a positive Pearson's correlation ($p < 0.05$) between the values of Zn, Cu, Ni, Co and Cr determined in surface sediments,

Table 2
Total metal concentration average (μg kg^{-1} dry weight) in superficial mangrove sediment in the Estero Salado – Guayaquil.

	Stations											EPD[b]	
	1	2	3	4	5	6	7	8	9	10	EQS[a]	Lower Chemical Exceedance Level (LCEL)	Upper Chemical Exceedance Level (UCEL)
Ag	0.78	1.49	1.17	1.09	1.11	1.06	1.44	0.93	1.27	1.00	ns	1	2
Al	57,737.3	57,136.6	74,219.9	55,876.4	51,553.9	52,218.6	62,131.8	46,214.8	36,552.8	31,159.5	ns	ns	ns
As	2.63	3.86	3.78	3.16	4.03	3.28	5.12	4.26	6.05	7.49	12.00	12	42
B	9.62	9.41	12.28	8.18	6.04	5.72	18.16	12.27	11.54	106.25	2.00	ns	ns
Ba	372.74	361.57	404.25	327.74	322.01	305.68	321.20	249.31	200.25	50.09	750.00	ns	ns
Be	1.24	0.62	0.34	0.39	0.37	0.21	0.39	0.23	0.83	0.59	ns	ns	ns
Cd	0.63	0.93	1.01	0.88	1.03	1.01	1.41	0.91	1.54	0.34	0.50	1.5	4
Co	16.58	16.56	18.40	14.81	15.21	15.40	16.70	14.36	14.24	5.65	40.00	ns	ns
Cr	48.36	62.41	73.85	57.19	64.47	54.78	64.42	43.83	40.74	33.92	65.00	80	160
Cu	139.46	198.82	204.91	167.63	173.54	172.18	206.51	144.38	162.37	47.08	63.00	65	110
Fe	36,431.1	38,326.4	45,423.1	35,445.2	34,859.9	35,134.8	39,284.3	32,046.7	28,326.5	16,471.1	ns	ns	ns
Li	10.21	11.08	13.77	7.85	11.19	12.28	11.89	10.43	11.64	11.52	ns	ns	ns
Mn	359.07	370.47	408.56	340.06	380.33	317.92	382.94	326.41	342.56	156.22	ns	ns	ns
Mo	2.27	3.07	3.33	2.21	2.13	2.52	4.08	3.15	3.46	5.65	5.00	ns	ns
Ni	48.19	56.44	60.91	51.58	52.05	54.91	60.14	42.39	40.80	27.37	50.00	40	40
Pb	37.66	53.67	49.71	48.26	49.33	49.50	56.42	39.80	47.08	20.99	25.00	75	100
Se	2.12	4.38	3.09	3.80	6.59	2.23	9.08	8.25	17.40	34.80	1.00	ns	ns
Sn	1.30	1.65	1.91	1.54	1.92	1.59	3.08	2.09	3.26	1.21	ns	ns	ns
V	124.69	138.40	159.88	126.29	126.87	129.69	144.49	105.24	96.82	124.16	25.00	ns	ns
Zn	331.31	510.49	506.49	450.99	440.06	378.96	527.17	335.35	327.10	93.95	60.00	200	270

ns: not specified by EQS or EPD.
[a] Environmental quality standard – Ecuador.
[b] Environmental pollution department – Hong Kong.

Table 3
Highest values of metal concentrations (μg kg^{-1} dry weight) in surface sediments in mangroves around the world.

Location	Pb	Zn	Cu	Cr	Ni	Co	Cd	Mn	Ag	V	As	Anthropogenic activities	References
Estero Salado, Ecuador	81.3	678.3	253.8	94.5	82.2	20.8	1.9	469.6	3.33	196.3	12.2	Industrial activities	This study
Ciénaga Grande, Colombia	81.7	65	20.3	42.8	19.8	ND	2.99	160	ND	ND	ND	RAMSAR wetland of international importance (Recovery Area)	Espinosa et al. (2011)
Sai Kung – Chek Keng, Hong Kong	31.2	43	2.5	1.2	2.9	14	0.32	111.7	ND	ND	ND	Relatively clean and remote	Tam and Wong. (2000)
South Port Klang, Malaysia	96.02	72.2	24.89	60.19	13.9	ND	1.46	219.1	ND	74.02	63.2	Industrial activities	Sany et al. (2013)
Sado Estuary, Portugal	69	507	195	63	15.09	ND	8	ND	ND	ND	50	Industrial activities	Caeiro et al. (2005)
Las Cucharillas, Puerto Rico	77.6	270.6	89.17	ND	ND	ND	ND	ND	ND	ND	12.2	Industrial activities	Mejias et al. (2013)
Pearl River, South China	32.23	127.41	51.52	104.68	48.14	ND	1.18	770.52	ND	ND	ND	Agriculture activities	Bai et al. (2011)
Fadiouth, Senegal	2.4	5.4	3.5	28.8	2.5	0.9	0.03	21	0.019	14.3	ND	Farming, fishing and mangrove exploitation	Bodin et al. (2013)
Red Sea, Egypt	25	ND	108	ND	22	14.1	1.8	16.1	ND	ND	ND	Receives sediments from outflowing currents coming from the gulf	El-Said and Youssef (2013)
Jambo, India	34.7	98.3	17.9	ND	32.2	32	4.4	ND	ND	ND	ND	Fishing and transportation	Behera et al. (2013)

ND: not determined.

evidencing a common biogeochemical origin (Table 4). These elements are often associated with industrial activities. To gain further insight about the origin of these heavy metals, EF was calculated (Table 5). EF values lower than 1.5 indicate that the metal is entirely provided from crustal contribution and values higher than 1.5 indicate that a significant portion of the element is from non-crustal sources (Herut and Sandler, 2006). Calculated EF values for Co, Ni, Cu, Zn, Se, Ag, Cd, Sn and Pb were above 1.5 in most of the stations analyzed (bold values in Table 5). For Ag, Cd, Cu, Se and Pb, EF values were higher than 5. This result, together with the fact that concentrations of these elements are equal, or greater in order of magnitude, to other mangroves with recognized industrial activity, makes it possible to assume that these elements (Ag, Cd, Cu, Se and Pb) have an anthropogenic origin, for instance industrial wastewater discharges (Caeiro et al., 2005; Mejias et al., 2013; Sany et al., 2013; Xin et al., 2013). In this sense, it is known that, during the last 20 years, the Estero Salado mangrove forest has received water discharges from approximately 190 industries (http://www.ambiente.gob.ec/listado-de-industrias-con-descargas-al-estero-salado/). The main activities which have been reported as sources of contamination in other mangroves in the world are also present in the Ecuadorian mangrove system, including aquaculture activities, beverages, preparation and storage of food (Cu, Zn, Co, Ni, Pb, Cd) (Behera et al., 2013; Tam and Wong, 2000), smelting activities (Cu, Pb, Zn, Cd) (Liu et al., 2011), glass production (Cu, Cd, Zn, Pb, Mn, Ni, Co, Cr, V, As) (Sany et al., 2013), release of agricultural input products (Cu, Zn, Mn, Cr, Ni, Cd, Pb) (Behera et al., 2013), pulp, paper, fertilizer (Cd, Pb, Zn, Cu, As, Cr) (Caeiro et al., 2005) and manufacture of textile and plastic products.

According to CLIRSEN (Center for Integrated Natural Resources Remote Sensing of Ecuador), since 2004, the extension of the Ecuadorian mangrove has been reduced to 108,000 Ha, which means the loss of 70% of this ecosystem (http://www.ccondem.org.ec/). Although it is not possible to establish a direct link between this

Table 4
Pearson correlation coefficient matrix showing the relationship between concentrations of heavy metal parameters in sediments from the Estero Salado.

	Li	Be	B	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Se	Mo	Ag	Cd	Sn	Ba
Li	1.00																	
Be	0.36	1.00																
B	0.11	0.14	1.00															
V	0.41	0.14	−0.06	1.00														
Cr	0.29	0.01	−0.47	0.80	1.00													
Mn	0.14	−0.02	−**0.79**	0.39	0.70	1.00												
Fe	0.28	0.05	−0.69	0.69	**0.84**	**0.88**	1.00											
Co	0.10	0.04	−**0.83**	0.47	0.71	**0.92**	**0.93**	1.00										
Ni	0.18	0.,00	−0.61	0.72	**0.84**	0.79	**0.88**	**0.85**	1.00									
Cu	0.11	−0.03	−0.73	0.57	**0.80**	0.77	**0.83**	**0.84**	**0.90**	1.00								
Zn	0.04	−0.01	−0.66	0.60	**0.80**	0.76	**0.81**	**0.81**	**0.91**	**0.96**	1.00							
As	0.02	−0.15	0.62	−0.11	−0.35	−0.50	−0.49	−0.59	−0.44	−0.35	−0.36	1.00						
Se	−0.05	−0.16	0.70	−0.23	−0.51	−0.61	−0.61	−0.69	−0.58	−0.55	−0.53	**0.95**	1.00					
Mo	0.19	0.19	0.72	0.16	−0.16	−0.54	−0.35	−0.51	−0.31	−0.27	−0.29	0.64	0.54	1.00				
Ag	0.15	0.13	−0.09	0.29	0.50	0.12	0.18	0.11	0.31	0.40	0.38	−0.08	−0.24	0.20	1.00			
Cd	0.08	−0.01	−0.55	0.27	0.52	0.57	0.51	0.58	0.62	0.78	0.69	−0.06	−0.29	−0.06	0.38	1.00		
Sn	0.14	0.02	−0.25	0.14	0.31	0.33	0.29	0.34	0.39	0.51	0.46	0.17	−0.01	0.13	0.26	**0.77**	1.00	
Ba	0.00	0.02	−**0.78**	0.49	0.70	**0.87**	**0.90**	**0.94**	**0.83**	**0.80**	**0.82**	−0.63	−0.69	−0.58	0.09	0.40	0.15	1.00
Pb	0.11	−0.06	−0.65	0.51	0.74	0.67	0.71	0.71	**0.84**	**0.92**	**0.91**	−0.32	−0.50	−0.27	0.44	0.75	0.46	0.69

Significant coefficients at a level of $p < 0.05$ (bold numbers).

Table 5
Enrichment factors of heavy metals in superficial sediments in the Estero Salado. EF values higher than 1.5 are shown in bold.

	Stations									
	1	2	3	4	5	6	7	8	9	10
Li	0.19	0.21	0.20	0.15	0.23	0.25	0.20	0.24	0.34	0.39
Be	0.94	0.47	0.20	0.31	0.31	0.17	0.27	0.22	0.99	0.82
V	0.70	0.79	0.70	0.73	0.80	0.81	0.75	0.74	0.86	1.29
Cr	0.56	0.73	0.67	0.69	0.84	0.71	0.70	0.64	0.75	0.73
Mn	1.41	1.47	1.24	1.38	**1.67**	1.38	1.39	**1.60**	**2.12**	1.13
Co	**1.74**	**1.75**	**1.50**	**1.60**	**1.78**	**1.78**	**1.63**	**1.88**	**2.36**	1.10
Ni	**1.67**	**1.98**	**1.64**	**1.85**	**2.02**	**2.10**	**1.94**	**1.83**	**2.23**	**1.76**
Cu	**6.12**	**8.81**	**6.99**	**7.60**	**8.53**	**8.35**	**8.42**	**7.91**	**11.25**	**3.83**
Zn	**2.38**	**3.71**	**2.83**	**3.35**	**3.54**	**3.01**	**3.52**	**3.01**	**3.72**	1.25
As	0.14	0.21	0.16	0.17	0.24	0.19	0.25	0.28	0.51	0.74
Se	**3.21**	**6.72**	**3.64**	**5.96**	**11.21**	**3.74**	**12.81**	**15.64**	**41.72**	**97.86**
Mo	1.21	**1.65**	1.38	1.22	1.27	1.49	**2.02**	**2.10**	**2.91**	**5.58**
Ag	**7.65**	**14.78**	**8.94**	**11.07**	**12.24**	**11.52**	**13.12**	**11.39**	**19.67**	**18.17**
Cd	**2.76**	**4.12**	**3.45**	**4.00**	**5.05**	**4.90**	**5.77**	**5.01**	**10.67**	**2.76**
Sn	1.35	**1.73**	**1.54**	**1.65**	**2.22**	**1.82**	**2.96**	**2.71**	**5.34**	**2.33**
Pb	**2.60**	**3.75**	**2.67**	**3.45**	**3.82**	**3.78**	**3.62**	**3.44**	**5.14**	**2.69**

fact and specific pollution sources, it is possible to assume that there is a relationship between the industrial activities present in the mangrove and the high levels of metals in the sediments.

The present study provides a baseline record of heavy metals in mangrove surface sediments on the West Coast of the Pacific, specifically in the inner part of the Estero Salado in Ecuador. These results show that many elements are present at high levels in the sediments of this mangrove. This is the first evidence of heavy metal pollution in the Estero Salado mangrove. Also, these results could be indicative of the general heavy metal levels in other unstudied mangroves in northwestern South America, affected by similar anthropogenic impacts.

Numerous metals, including those elements considered highly toxic, such as Cd, Cu, Cr and Pb, are stored in sediments of this mangrove ecosystem, which represents a potential ecotoxicological risk. According to guidelines previously established for the management and policy of sediment research (Salomons and Förstner, 2010; Förstner and Salomons, 2010), the development of public policy programs for restoration and recovery of natural resources, such as mangrove systems, must focus on the integration of socio-economic aspects and natural sciences. In this sense, it is advisable that the implementation of management programs, such as the one being carried out by the local government to save and protect the mangrove ecosystem, known as "Guayaquil Ecológico program", should also consider the potential risk that the eventual mobilization of the sediment represents, taking into account that this mangrove is one of the most disrupted worldwide.

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