

Metal Contents in Sediments (Cd, Cu, Mg, Fe, Mn) as Indicators of Pollution of Palizada River, Mexico

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Abstract

Some heavy metals and trace metals reach aquatic ecosystems from natural and anthropogenic sources, and are considered some of the most important environmental contaminants due to their toxicity, persistence and tendency to accumulate in aquatic organisms. Thus, their study is needed due to the environmental risk they pose. Concentrations of Cu, Cd, Mg, Fe and Mn in recent sediments of the deltaic lagoon-river system of the Palizada river, Campeche, Mexico were determined for three climatic seasons on the 2010 annual cycle. The results confirmed that the climatic season has great influence over the results variability. The highest levels of Cu, Fe and Mn were found during dry season, which may suggest significant evaporation phenomena in the area. Both Fe and Mn are abundant elements in the Earth crust; their concentrations could be related to the study area's characteristics, given the conjunction of two sedimentary provinces: terrigenous in the western portion and carbonated in the eastern. On the other hand, the results suggest a high relationship of Fe-Mn ($r = 0.5131$), Fe-clay ($r = 0.5978$), Cu-Mn ($r = 0.8707$), Cu-clay (0.8501) and Mn-clay (0.9311). The latter confirms the high dependence of these elements and the great affinity of some metallic elements for finer sediments. In conjunction, the climatic season and the sediment's characteristics are essential for metal mobilization and transport. Likewise, the Cd and Cu levels reported are lower than international parameter, indicating value ranges that could cause effects in exposed organisms.

Keywords: trace metals, Palizada river, Gulf of Mexico

1. Introduction

Coastal ecosystems are currently exposed to high amounts of contaminants coming from industrial and urban activities being poured discharged into the systems, which contributes to increasing the concentration of certain contaminants (Cuong et al., 2005; Vane et al., 2009).

Most heavy metals released into the environment reach aquatic systems through direct discharge, wet and dry deposition and erosion. Sediments can accumulate the heavy metals reaching the lake environment; on the other hand, changes in physicochemical conditions can remobilize and release metals into the water column. These contributions can be transferred through the food chain to individuals.

The study of sediments in an aquatic ecosystem allows a comprehensive assessment of a site contamination, since they are the main recipients of most of the contaminants deposited from the water column by precipitation. The metals associated to the organic fraction can form solutions and become available for aquatic life, making it possible to establish a relationship between the sediments heavy metals and the living organisms from a given environment (Aguilar et al., 2012).

In this sense, Santos et al. (2003) & Lima et al. (2005) proved the importance of sediments as water pollution indicators. Characteristics such as size of grain sediments and the amount of organic matter are of great importance as they determine the presence and availability of certain contaminants such as heavy metals and trace metals (Sauvé et al., 2000).

1.1 Background of the Study Area

Terminos lagoon is located in the Gulf of Mexico; it is one of the most studied ecosystems in Mexico due to its importance as a nesting site for many bird species and holding many flora and fauna species. It covers an area of 705,016 hectares, making it in one of the largest protected natural areas of Mexico (INE/SEMARNAP, 1997). The northern continental shelf of the Laguna is highly productive in oil extraction, and it has been shown that the latter can be a source of contaminants (hydrocarbons, heavy metals and others). Besides the rivers (Palizada, Chumpán and Candelaria), some inland lakes such as Pom, Atasta, del Corte, San Carlos, del Este, Balchacah y Panlau –which flow into the lagoon in the south– are a source of agrochemicals and other contaminants.

The oil extraction industry located in this area emits different contaminants into the air, water and soil, including nickel, chromium, lead and cadmium (RETC, 2011).

Considering the area's characteristics, it is important to monitor metal concentrations in order to detect the contamination source and the sediment quality.

2. Method

2.1 Sediment Recollection

Three sampling campaigns were conducted during the rainy, dry and north winds climatic seasons. With that purpose, ten sampling stations were established, which were identified in a previous tour using a global positioning system (GPS). Surface sediment samples were collected at each point using a 0.1-m³ Van Veen dredge. All sediment samples collected were stored in polypropylene containers previously washed with a solution of 10% HCL.

Figure 1 shows the study area, highlighting the sampling points.

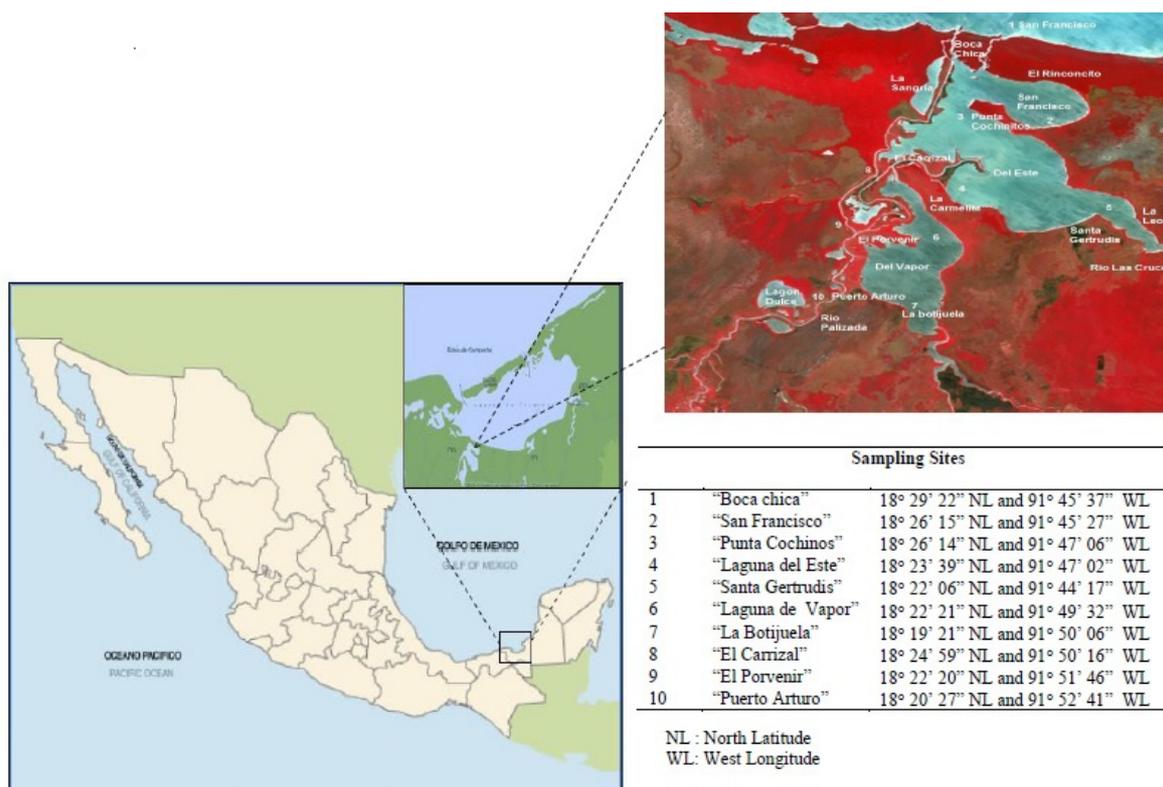


Figure 1. Study area, showing the three sampling areas

2.2 Treatment of Samples

Collected sediments were dried by lyophilization for 24 hours, and homogenized using an agate mortar. A portion of the sediments was used to evaluate organic matter using the technique suggested in the AA-034-NMX-SCFI-2001, which is based on the weight loss on ignition.

Analyses of sediment texture were performed through the Bouyoucos-scale hydrometer technique proposed by Buchanan & Kain (1971). For metal analysis the analytical technique suggested by EPA in method 3050B, consisting of an acid digestion with HNO₃ in a sediment sample was used, the digestion is completed with the addition of H₂O₂, the volume is reduced, finally filtered sample and deionized water to volume for subsequent analysis.

2.3 Analysis of Metals in Sediments Samples

All samples were analyzed by Flame Atomic Absorption Spectrophotometer (AVANTA GBC-A4509), using standard solutions at different concentrations of recognized analytical grade (J.T Baker). The solutions were prepared according to the working ranges of equipment operation, (Table 1). All samples were analyzed by duplicate.

Table 1. Operating limits in detecting elements

Element	Wavelength (nm)	Detection range ($\mu\text{g L}^{-1}$)*
Cadmium	228.0	0.01 – 5.00
Iron	386.0	5.00 - 300
Cooper	222.0	0.10 -60
Magnesium	322.5	0.1- 150
Manganese	219.0	0.2-300

* Data manual operation of the equipment AVANTA GBC-A4509

2.4 Statistical Analyses

All data were evaluated to determine normality by applying the Shapiro-Wilks test, which is used for testing normality of a data set. It states as null hypothesis that a sample x_1, \dots, x_n comes from a normally distributed population (Shapiro & Wilk, 1965). The Statgraphics software was used for this purpose, with a confidence level of 95%, which confirmed the normality of the experimental data.

To determine the heavy metal levels and soil texture variation per sampling site and climatic season, an analysis of variance (ANOVA) test was applied. Finally, a Pearson correlation was applied to determine the relationship between metals and the characteristics of sediment, as well as organic matter.

3. Results and Discussion

In marine systems, heavy metals attach to the sediment by processes such as adsorption and co-precipitation by iron and manganese hydroxides and oxides, adsorption in mineral clays, precipitation with organic matter, hydrolytic reactions of ions and dissolved complexes and other natural mechanisms related to the sediments physicochemical characteristics (Bruder et al., 2002; Sutherland & Tack, 2002).

Marine sediments act as metal integrators and concentrators (García et al., 2004). Depending on the metals physical and chemical properties, they can be mobilized and transported and even pose serious hazards for an ecosystem health. Therefore, their monitoring and control is a necessary action for the conservation of the environment, as well as to define mitigation policies and actions.

3.1 Manganese and Iron

Result obtained (Table 2) of the analysis of heavy metals in sediments of the Palizada river clearly showed that one of the most abundant elements is iron; other studies have found atypical iron concentrations, which are considered indicators of an increase in the rate of fine-material sediments contributed by the rivers (Zarazúa et al., 2011). Likewise, it was considered that there is an influence by the neighboring industries for this metal atypical increase (Mora et al., 2013).

Table 2. Ranges of metals concentrations (annual) in $\mu\text{g g}^{-1}$

Climatic season	Elements				
	Cu	Cd	Fe	Mg	Mn
I Dry	5.8-10.48	1.00 -1.71	216.6 -232.47	3.28– 4.61	127.51- 246.58
Mean	9.658	1.426	225.89	4.365	187.903
*SD	4.0065	0.260	5.2512	0.389	41.407
II Rainy	0.19 -0.256	1.01–1.60	215.4-222.54	14.69-17.91	6.89-13.44
Mean	0.1954	1.335	219.948	16.904	9.50477
*SD	0.056	0.2144	2.93225	1.1129	2.1569
III North winds	1.1-1.23	2.19-2.34	12.9-34.6	5.18-22.16	2.77-15.09
Mean	1.154	2.261	23.769	12.238	6.4262
*SD	0.04409	0.0427	6.846	6.93024	3.0634

*Standard deviation

On the other hand, manganese is another of the most abundant elements in this study and its concentrations increases during the dry season (range 127.1- 246.58 $\mu\text{g g}^{-1}$), which suggests significant evaporation phenomena in the area as well as low mobility of sediments. On the analysis of the results it was determined that there is a high relationship (Table 5) between Fe-Mn ($r = 0.5131$). This feature can be instrumental in their association and pre-disposition to transport other metals, along with their biogenic origin. Thus, it was considered that their levels are proper to the area characteristics, the natural movement of water bodies and sediment transport during different climatic seasons.

It has been shown that most of the iron in coastal systems is bound to the oxidation of organic matter. On the other hand, its presence in sediments increases productivity at shallow coastal regions (Hutchins & Bruland, 1998). Decomposition of organic material produces iron flows into water bodies and near the coast. In this study, the relationship between Fe and the organic matter (Fe-MO), were not significant ($r = 0.1770$) as shown in Table 5.

The statistical analyses show that the climatic season does influence the Fe and Mn concentrations ($p < 0.05$), as shown in Table 3. The highest Fe, Mn and organic matter concentrations were determined during dry season. The same behavior has been identified in other studies, in which the most abundant metals were Fe and Mn, considered as natural input elements (Zarazúa et al., 2011).

Table 3. Statistical p values from the ANOVA and normality tests

Variation sources	Analyzed element				
	Cd	Cu	Fe	Mg	Mn
Site	0.2099	0.4753	0.8541	0.641	0.4467
Season	0.0001*	0.0002*	0.0000*	0.0000*	0.0000*
Annual mean	1.674	3.6673	156.55	11.69	69.81
Standard deviation	0.4685	4.925	95.68	6.68	91.70
Normality test (p)	0.00194**	0.0000**	0.00002**	0.0063**	0.0000**

* Significant values with a confidence value of 95%

** For the normality test, it means that data is normally distributed and the confidence value is less than 0.05

Fe and Mn are essential in marine sediments and constitute a source of minerals for different flora and fauna marine species. Although it is highly difficult to distinguish between anthropogenic and natural source, concentration increases between seasons are an important element for analysis.

These results of the influence of soil texture on transport, mobility and association with sediments are shown in Table 4.

Table 4. Ranges in percentages of sediment texture and organic matter content in $\mu\text{g g}^{-1}$

Climatic season	Sand	Silt	Clay	Organic matter
I Dry	27.29-38.2	10-26.09	34.8-57.94	1.46-13.33
Mean	31.755	17.372	50.883	4.589
*SD	3.535	5.995	8.272	4.3392
II Rainy	7.6-73.2	22.4-80.8	2.4-11.6	2.22-4.54
Mean	44.086	50.377	5.937	3.66722
*SD	21.412	20.2202	3.3814	0.84433
III North winds	15.2-85.6	10.4-76.4	2.4-11.6	2.02-4.18
Mean	50.633	42.717	6.65	2.99503
*SD	24.3834	22.546	3.0188	0.70282

*Standard deviation (SD)

The concentrations of sand and clay are predominant in the area and in the dry season show the highest level, showing certain relationship between metal levels detected and the climatic season. These results show high correlation values between iron and manganese with the sediment clay particles (Table 5). In particular, the iron relationships with clay particles showed high correlation ($r = 0.5978$).

Table 5. Correlation matrix of variables

	Analyzed elements							
	Cd	Cu	Fe	Mg	Mn	Sand	Silt	Clay
Cu	-0.2477							
Fe	-0.8953	0.3908						
Mg	0.0273	-0.6637	-0.1488					
Mn	-0.0404	0.8707*	0.5131*	-0.7129				
Sand	0.1552	-0.3060	-0.3234	0.4609	-0.3541			
Silt	0.1836	-0.5574	-0.1925	0.3137	-0.5929	-0.4717		
Clay	-0.3371	0.8501*	0.5978*	-0.7424	0.9311*	-0.4370	-0.5866	
Organic matter	-0.3024	0.1630	0.1770	-0.1421	0.2087	-0.0957	-0.1315	0.2214

* Significant values with a confidence value of 95%

Several studies have reported this behavior (Aguilar et al., 2012), which constitute significant factors in the mobility of heavy metals.

3.2 Cooper

Other studies have reported significant relationships between manganese and copper with organic matter (Karbassi et al., 2011); nevertheless, those relationships did not show in this study. Still, there is high affinity between Mn and Cu ($r = 0.8707$). These results are shown in Table 5 and may indicate association phenomena between these elements or common sources.

Regarding copper, levels can be considered low compared to other studies that report up to $18 \mu\text{g g}^{-1}$ in the sediments of the rivers flowing into Terminos lagoon (Aguilar et al., 2012). Previous studies have reported up to $23 \mu\text{g g}^{-1}$ of Cu in surface sediments at Baja California, Mexico with an increasing trend compared to areas considered free of contamination (Villaescusa et al., 2002). In this study copper showed a high relationship with the sediment finer particles, (Cu-clay $r = 0.8501$), which shows its high affinity to sediment providing greater absorption and ionic attraction due to negatively charged clay particles. This causes permanence in deposited sediments and bioavailability to the animal species whose habits make them susceptible to ingest these contaminants. Mn and Cu show high affinity with the sediment clay particles, which may suggest that there are

association phenomena of Cu-Mn and the sediment finer material. This increases the possibility of retention and accumulation of copper in the sediments.

The high copper concentrations have been related to wastewater discharges and hydrocarbons (González et al., 2006). Concurrently, other studies attributed high levels of determined Cu and Cd to wastewater discharges in lakes in China (Zeng & Wu, 2013). On the other hand, none of the other metals analyzed showed a relationship with organic matter, which might suggest that the source is not biogenic. Recently, Vázquez et al. (2002) and Vázquez and Sharma (2004) related high copper and other elements concentrations in the sediments at the “Sonda de Campeche” to the combustion of gasoline and exploration, hydrocarbon production and shipping in the area. In this sense, the studies of Turner (2013), confirm that the geosolids derived from fine particles in paint used for boat maintenance increase the concentration of heavy metals such as copper in sediments and surrounding areas, even exceeding the limits established by the UK Environment Agency.

Recent studies (Mora et al., 2013), determined copper concentrations above those found in sediments of the Palizada river. Comparing these results with the standard for the protection and management of aquatic sediment by the Ontario Ministry of the Environment (Persaud et al., 1993), the values for copper and cadmium in this study are below those for the effects range low (ERL), defined as the concentration after which the first adverse effects are observed on benthic organisms.

Moreover, the results showed that the levels of heavy metals are below the ERL in the case of copper (ERL and ERM reference values for heavy metals can cause biological effects on exposed marine organisms). The effects incidence has been estimated in ranges lower than 25% at concentrations below ERL values (Long et al., 1995). The estimated limits for considering the risk values are $34 \mu\text{g g}^{-1}$ for Cu, but the ERL for cadmium establish a risk value of $1.2 \mu\text{g g}^{-1}$, which is exceeded in this study.

3.3 Magnesium

Another element that comes into attention is magnesium, whose values were higher during the norths season (maximum concentration of $22.16 \mu\text{g g}^{-1}$), as shown in Table 2. This behavior may be due to the particular circulation mechanisms at the coastal area in this season, compared to other climatic seasons (Villanueva & Botello, 1992). The ANOVA confirms that there is significant statistical difference only by climatic season ($p = 0.0000$), but not by site (Table 3).

These determined concentrations may be derived from the amount of water mobilized during this climatic season, as the study area has the highest values in the country, with average annual precipitation of 1169 mm (Villalobos & Mendoza 2010). Concurrently, runoff from agricultural soils is high in fertilizers, which could be a source of increased Mg in sediments.

Additionally, this area is characterized for being highly productive with regards to agriculture with the use of ammonium phosphate fertilizers (Medina et al., 2009). Different studies have found that the use of fertilizers increases the concentration of heavy metals that can even be transferred to food and enhance adverse effects on aquatic organisms through runoff from agricultural soils to water bodies (Conceição et al., 2013). Like many metals, magnesium's highest concentrations are seen in surface sediments, which tend to decrease with depth (Higgins & Schrag, 2010). In this study, magnesium showed no significant relationship with any other element or with sediment characteristics.

3.4 Cadmium

Regarding cadmium, it is considered one of the most toxic element to the environment. Recent studies indicate that cadmium poses a serious ecological threat and contributes greatly to the toxicity response rates, as it is even more toxic than arsenic and lead (Min et al., 2013, Wei & Yang, 2010; Chabukdhara & Nema, 2013; Zeng & Wu, 2013). The results are shown in Table 2.

The concentration profile in relation to the other elements is low. This is consistent with Chakraborty & Owens (2013), who determined cadmium concentrations of up to $125 \mu\text{g g}^{-1}$ along the Australian coast. On the other hand, previous studies in the Terminos lagoon have reported cadmium concentrations between 1.2 and $1.5 \mu\text{g g}^{-1}$ in surface sediments (Ponce & Botello, 1991); likewise, concentrations of $0.40 \mu\text{g g}^{-1}$ in river sediments were recently determined (Aguilar et al., 2012). These levels are below the maximum values reported in this study, which are $2.34 \mu\text{g g}^{-1}$ in climatic season of norths.

The presence of cadmium in marine sediments, it is considered totally extraneous to life and its presence in sediments is mainly due to human action. In the sediments, the main solid cadmium species occurring under oxidizing conditions are CdO, CdCO₃, while under reducing conditions it is CdS (Villanueva & Botello, 1992). The oxyanionic species exist at high pH, whereas at low pH the predominant ion is Cd²⁺.

Cadmium solubility is controlled by the presence of organic matter and Fe-Mn hydrous oxides, which could be a determining factor in the retention of cadmium in the sediments as our results show high Fe-Mn relationships ($r = 0.5131$). Shows that the latter can predispose the presence of cadmium. In addition to low cadmium concentrations, the determining factor for its mobility is the absorption in several soil constituents, which could be Mn oxides > Fe hydrated oxides > Fe crystalline oxides. This also suggests association phenomena with Fe. However, the results show that there is no significant relationship between Cd-Fe.

Cadmium values exceed the limits established by the ERL (ERL and ERM reference values for heavy metals can cause biological effects on exposed marine organisms). The effects incidence has been estimated in ranges lower than 25% at concentrations below ERL values (Long et al., 1995). The estimated limits for considering the risk values are $1.2 \mu\text{g g}^{-1}$ for cadmium.

The ANOVA results, shown in Table 3, confirm that only the season influences the concentration variations of the elements analyzed ($p < 0.05$). However, for the sediment texture fractions, the site is the factor promoting concentration variability as shown in table 6 for the fractions of silt and Clay.

Table 6. Statistical p values from the ANOVA and normality tests

Variation sources	Sand	Silt	Clay	Organic matter
Site	0.0779	0.0006*	0.000*	0.4095
Climatic Season	0.0953	0.0395	0.3055	0.3370
Annual mean	41.627	35.7007	22.82	3.75065
Standard deviation	20.84	23.35	22.68	2.69968
Normality test (p)	0.005374**	0.002252**	0.00007**	0.000009**

* Significant values with a confidence value of 95%

** For the normality test, it means that data is normally distributed and the confidence value is less than 0.05

Also the organic matter content is usually high as compared to that of recent studies in the surrounding areas (Aguilar et al., 2012) since the area is influenced by the extensive mangroves, several macrophyte species and human settlements, thus contributing substantially to the variations of organic matter. This greatly predisposes both retention and mobility of heavy metals, in this study organic matter shows no seasonal climate variability or sampling site.

4. Conclusion

The absence of effective monitoring and control of contaminants at the Mexican coastal environment, the growing industrialization and urbanization, and mostly the lack of real implementation of environmental regulations have caused rivers and lagoons at the Gulf of Mexico to be at risk due to the presence of several contaminants.

It is very difficult to make the difference between human and natural sources, but both have influence on the levels of iron, magnesium and manganese, which were the most abundant in this study, likewise these elements are part of the earth's crust and may form partnerships with the characteristics of sediments primarily with the finest fractions. Magnesium, manganese and iron are natural components of the sediments the atypical increase in their concentrations reveals the human impact on the Palizada river, by the use of fertilizers and other products for agriculture, which is directly related to high concentrations in climatic seasons of dry. Also this fact clearly shows that the amount of metals deposited sediments from climatic seasons and that these levels are lower than those detected in the rainy season climate can infer what the dilution of contaminants.

The study area has a large human influence agricultural activities taking place in the Palizada river, this area also forms part of the Terminos lagoon and the "Sonda of Campeche", where it extracts more than 70 % of production hydrocarbon Mexico, according to current studies, this industry generates numerous emissions into the atmosphere including heavy metals, this may be one of the generation sources of heavy metals as due to the physical and chemical processes that occur these are deposited in water and sediments are precipitated as evidenced by numerous studies in the area.

Making a comparison with other studies, the levels of metals were detected in the Palizada river do not represent a risk factor for the ecosystem, however cadmium levels are slightly higher than international standards and if

there could be considered a risk factor because cadmium does not come from any natural source, which may suggest that there are anthropogenic discharges into the Palizada river.

The seasonal climate has a major influence on the concentration and distribution of heavy metals, being in the case of iron, copper and manganese dry season the most significant climate for magnesium season was the most influential of showers. For cadmium the climatic season Norths was the most significant. Organic matter levels increase during the dry season in the study area are given numerous weather events with the rainy season with very high levels of precipitation and very extreme dry seasons with temperatures close to 40 ° C.

The sediments represent a reservoir of heavy metals, some of which may form associations with sediment texture as shown in this study with high correlation values between copper, iron and manganese clays, thus can be made available for other life forms.

Also the high values of correlations between the metals copper and manganese, iron and manganese may suggest that these elements may have a common source of generation, being the most abundant that were determined in this study.

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