Assessment of heavy metal pollution in urban soils and dusts from Regla town (Havana, Cuba) using X-ray fluorescence analysis

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Abstract
Ni, Cu, Zn and Pb contents in urban dust and surface soils from Regla town (Havana, Cuba) are determined by X-ray fluorescence analysis. The obtained results are compared with the metal contents reported for Havana and industrial areas from other Cuban and worldwide cities. The application of pollution, ecological risk and toxicological indexes allows to evaluate the impact induced by the local industries emissions to the population health. The calculated hazard index and cancer risk of all studied heavy metals suggested the acceptable range for both noncarcinogenic and carcinogenic risk to children and adults.

Key words: *X-ray fluorescence analysis; heavy metals; urban areas; dust; soils; Cuba.*

Evaluación de la contaminación por metales pesados en suelos y polvos urbanos del pueblo de Regla (La Habana, Cuba) mediante fluor escencia de rayos X

Resumen
Se determinan las concentraciones de Ni, Cu, Zn y Pb mediante Fluorescencia de Rayos X en muestras de suelos y polvos urbanos de la localidad de Regla (La Habana, Cuba). Los resultados obtenidos se comparan con los contenidos de metales reportados para La Habana y áreas industriales de otras ciudades cubanas y del mundo. El empleo de indicadores de contaminación, de riesgo ecológico y toxicológico, permitió evaluar el impacto que inducen las industrias locales sobre la salud de la población. Los valores calculados de los índices de peligrosidad y de riesgo carcinogénico para los metales estudiados indican un rango aceptable de riesgo no-carcinogénico y carcinogénico para niños y adultos.

Palabras clave: *análisis por fluorescencia de rayos X; metales pesados; áreas urbanas; polvo; suelos; Cuba.*

Introduction

The increment of the industrialization and urbanization has deep attempted against urban environment [1]. Urban soil and street dust are strongly influenced by anthropogenic activities [2, 3], and receives a major proportion of potential toxic metals emissions from different sources, such as atmospheric deposition, vehicular traffic, industrial emissions, construction, building deterioration, mining activities, etc. [4]. These pollutants could remain in soil and dust for a long time and accumulate in human fatty tissue and internal organs via direct inhalation, ingestion and dermal contact absorption [5, 6], representing a risk to human health because of their toxicity and non-degradability, especially in children [7-9].

The actual Regla town is a Villa founded in 1687. It is located in the back side of the Havana port and it has been always considered an industrial villa. Since the end of the XVIII century, local industries were related to naval construction, sugar and packaging industries, distilleries, refineries, etc. Nowadays, Regla is one of the municipalities of the Havana City (population 42939 inhabitants with the 20.7%, corresponding to children below 14 years of age [10]. Furthermore, and in its surroundings are located well-know polluting industries as an oil refinery, an electric power station (closed few years ago), a concrete plants and a metal smelter. Moreover, due to its proximity to the Havana port, another source of heavy metals in Regla urban environment is the exhaust emissions of heavy traffic.

It should be also noted that medical studies aimed at evaluating the impact of internal and external pollution sources on the population's health have concluded that Regla is the Havana municipality with the highest epidemiologic risk due its environmental pollution. Furthermore, they also found that some diseases as tumor, congenital malformations, chronic renal insufficiency, high arterial tension [11] as well as respiratory infections and acute diarrheal morbidity in children below 15 years of age [12].

In this sense, the objectives of this study were; (1) to investigate the concentration of nickel (Ni), copper (Cu), zinc (Zn) and lead (Pb) in the urban dust and surface soils throughout Regla town in order to evaluate its environment quality in terms of metal contamination and (2), assess the potential risk such pollution represents for the local population developing carcinogenic and non-carcinogenic diseases.

Materials and methods

Regla is located in the coordinates 23°07′30″N - 82°19′55″O. The municipality is limited north by the Havana bay and the oil refinery Ñico López, south by the municipality of San Miguel del Padrón, east by the refinery and the municipality of Guanabacoa and west by the Havana bay. The major part of its territory is bordered by high heavy traffic speedways as Via Blanca and Avenida del Puerto.

In the present study, 23 urban dust and 11 surface (0-10cm) soil samples were collected in 26 locations in Regla town (fig.1). Locations correspond to schools (4), child parks (3), kinder-gartens (2), parks (7) and industrial (6) and residential (4) areas. Dust samples (around 100g) were collected by gently sweeping an area of about 16 m^2 in the street crossroad using a plastic hand broom, while soil samples were collect by spatula. All samples were transferred to a clean, self-sealed polyethylene bag. In the laboratory, samples were at first dried at 35 °C and large rock, metallic and plastic pieces and organic debris were removed before sieving. The fractions smaller than 2 mm were ground to a fine powder (<63 μm) in an agate mortar. The pulverized samples were newly dried at 35 °C until obtaining a constant weight.

The Ni, Cu, Zn and Pb concentrations were estimated by X-Ray Fluorescence Analysis (XRF) using the Certified Reference Materials (CRM) IAEA-SL-1 *"Lake Sediment"*, IAEA-Soil-5, IAEA-356 *"Polluted Marine Sediment"*, BCR-2 *"Basalt Columbia River"*, SGR-1 *"Green River Shale"* and BCSS-1 "*Marine sediment*" from the Canadian National Research Council as standards. All samples and CRM were mixed with cellulose (analytical quality) in proportion 4:1 and pressed at 15 tons into the pellets of 25 mm diameter and 4-5 mm height. All pellets were measured using Canberra Si(Li) detector (150 eV energy resolution at 5.9 keV, Be windows thickness = 12.0 μm) coupled to MCA. A ^{238}Pu (1.1 GBq) excitation source with ring geometry was used. All spectra were processed with WinAxil code [13]. Detection limits (LD see table 1) were determined according to Padilla et al. [14] (in concentration units) as: LD=3σ/mt, where m is the sensibility in counts.seg-1 per concentration unit, σ is the standard deviation of the area of the background windows (peal windows at 1.14 times the FWHW) and t is the measuring time (6h).

The accuracy was evaluated using the SR criterion, proposed by McFarrell [15]:

$$SR = \frac{|C_X - C_W| + 2\sigma}{C_W} . 100\%$$

where C_X is the experimental value, C_w the certified value and σ the standard deviation at C_X. On the basis of his criterion the similarity between the certified value and analytical data obtained by proposed methods is divided into three categories: SR ≤ 25% = excellent; 25 < SR ≤ 50% =acceptable, SR > 50% = unacceptable.

Figure 1.- Location of Regla town in Havana and the studied stations

The analysis of five replica of the CRMs IAEA-Soil-7 (for soil samples) and IES-951 (for dust samples) is presented in table 1. In both cases, all heavy metals of interest determined by XRF are "excellent" (SR ≤ 25%) and the obtained results shows a very good correlation between certified and measured values. The spatial distribution maps of all studied heavy metals in urban street dust and surface soils from Regla town were generated with ArcGIS software.

The environmental quality of the studied surface soils and urban dusts, was estimated by Integrated Pollution Index (IPI) [16], for each studied location, It is defined as the average value of all Pollution Index (PI) values for each metal of interest:

$$\mathrm{IPI} = \frac{1}{n}\sum_{i=1}^{n} \mathrm{IP}_i; \qquad \mathrm{IP_i} = \frac{\mathrm{C_i}}{\mathrm{B_i}}$$

where, C_i are the concentrations measured for each metal on interest, and Bi their corresponding background contents. IPI is evaluated by 3 categories (see table 2). In the present study, the average metal content in soils from Havana un-urbanized areas [17] were taken as background values: 58 $mg.kg^{-1}$ for Ni, 86 $mg.kg^{-1}$ for Cu, 151 $mg.kg^{-1}$ for Zn and 28 $mg.kg^{-1}$ for Pb.

The ecological risk index (RI) was introduced by Hakanson [18] and is used to assess the degree of heavy metal pollution in soil, dust and sediments, integrating the factors of the potential ecological risk for each metal, and associating their environmental and ecological effects with their toxicity. It is calculated as:

$$\mathrm{RI} = \sum \mathrm{Er^i}; \qquad \mathrm{Er^i} = \mathrm{Tr^i} \times \mathrm{C_f^i}; \qquad \mathrm{C_f^i} = \frac{\mathrm{C_x^i}}{\mathrm{C_n^i}}$$

where, RI is the sum of the potential risk posed by individual heavy metals; Er^i - is the partial ecological risk (i.e., the potential risk from a given metal); Tr^i - is the toxic response factor for a given metal (in the present study Ni=Cu=Pb=5 and Zn=1) [18]; C_f^i - is the contamination factor for each metal, C_x^i is the heavy metal content in the studied sample, C_n^i - is the background concentration for a given metal. The ecological risk categories are presented in table 2.

Health risk assessment models were used to quantify the health risk (carcinogenic and non-carcinogenic) for Regla population exposed to heavy metals in urban dust. Local population are exposed to metals in urban dust through three main exposure pathways: direct ingestion, inhalation through mouth and nose, and dermal absorption. The total non-carcinogenic risk was calculated for each metal in urban dust by the summation of the individual risks calculated for the three exposure pathways [19-21].

The average daily dose (ADD) (mg kg^{-1} day^{-1}) for heavy metals in road dust through the three exposure pathways was calculated according to Exposure Factors Handbook [22] and the Technical Report of USEPA [23] using the following equations:

$$\mathrm{ADD_{ing}} = \mathrm{C_{metal}} \times \frac{\mathrm{IngR \times CF \times EF \times ED}}{\mathrm{BW \times AT}} \qquad (1)$$

$$\mathrm{ADD_{inh}} = \mathrm{C_{metal}} \times \frac{\mathrm{InhR \times EF \times ED}}{\mathrm{PEF \times BW \times AT}} \qquad (2)$$

$$\mathrm{AAD_{dermal}} = \mathrm{C_{metal}} \times \frac{\mathrm{SA \times CF \times AF \times ABF \times EF \times ED}}{\mathrm{BW \times AT}} \qquad (3)$$

$$\mathrm{LADD} = \mathrm{C_{metal}} \times \frac{\mathrm{CR \times EF \times ED}}{\mathrm{PEF \times BW \times AT}} \qquad (4)$$

where, the ADD_{ing}, ADD_{inh} and ADD_{dermal} are the average daily dose ($mg.kg^{-1}.day^{-1}$) exposure to metals through ingestion, inhalation and dermal contact, respectively; LADD is the lifetime average daily dose exposure to metals ($mg.kg^{-1}.day^{-1}$) for cancer risk; CR is the contact frequency and is the same IngR used in the calculation of ADD_{ing} [23-25]; C_{metal} - is the concentration of metals in dust; IngR - Ingestion rate of dust ($mg.d^{-1}$):

Table 1. XRF analysis of CRMs Soil-7 and IES-951. SR values and Detection limits (LD)

Element	CRM Soil-7 ($mg.kg^{-1}$)		SR (%)	CRM IES-951 ($mg.kg^{-1}$)*		SR (%)	LD ($mg.kg^{-1}$)
	C_{DET}	C_{REP}		C_{DET}	C_{REP}		
Ni	26 ± 3	26 ± 4	23	38 ± 5	38 ± 3	15	7
Cu	11.1 ± 1.1	11.0 ± 1.0	19	29.9 ± 0.9	30.5 ± 2.3	17	6
Zn	98 ± 7	104 ± 3	12	104 ± 6	96 ± 6	21	5
Pb	56 ± 5	60 ± 4	20	36 ± 4	37 ± 4	24	4

Table 2. Classification of the Integral Pollution Index (IPI) and categories for the Potential Ecological Risk (Eri) and ecological Risk Index (RI).

IPI [16]		Er^i [18]		RI [18]	
Valor	Category	Valor	Category	Valor	Category
IPI ≤ 1	Low polluted	Eri < 40	Low	RI ≤ 150	Low
1 < IPI ≤ 2	Middle polluted	40 ≤ Eri < 80	Moderate	150 ≤ RI < 300	Moderate
2 < IPI	High polluted	80 ≤ Eri < 160	Considerable	300 ≤ RI < 600	Considerable
		160 ≤ Eri < 320	High	600 ≤ RI	Very high
		Eri ≥ 320	Very high		

100 for adults and 200 for children [21]; EF -exposure frequency ($d.y^{-1}$): 350 [26]; ED - Exposure duration (y): 6 for children and 25 for adults [21]; BW - Average body weight (kg): 15 for children and 70 for adults [21]; AT - Average time (d): 365 x ED [20]; CF - Conversion factor ($kg.mg^{-1}$): 1 x 10^{-6} [27]; InhR - Inhalation rate of dust ($m3.d^{-1}$): 7.63 for children and 12.8 for adults [21, 27]; PEF - Particular emission factor ($m3.kg^{-1}$): 1.36 x 109 [24, 25]; SA - Surface area of skin exposed to dust (cm^2): 1600 for children and 4350 for adults [26]; AF - Skin adherence factor ($mg.cm^{-2}$): 0.2 for children and 0.7 for adults [28, 29] and ABF - Dermal Absorption factor (unit less): 0.001 [23, 24].

In order to evaluate the human health risk of heavy metal exposure from urban dusts in Regla, the *hazard quotient* (HQ), *hazards index* (HI), and the *carcinogenic risk* (CR) assessment were applied. The potential risk of carcinogenic and non-carcinogenic hazards for individual metals were calculated using the following equations [19, 30]:

$$HQ = \frac{ADD}{R_f D}$$

$$HI = HQ_{inh} + HQ_{ing} + HQ_{der}$$

$$CR = LDDA \times SF$$

where RfD ($mg\ kg^{-1}\ day^{-1}$) is the corresponding reference dose, which was defined as the intake or dose per unit of body weight. The hazard index (HI) is the sum of the hazard quotient (HQ) from each exposure pathway. HI<1 means there is no significant risk of non-carcinogenic effect that could be ignored, whereas HI >1 suggested that adverse effects might occur [31]. As an estimate of the upper-limit probability of an individual developing cancer because of exposure to a particular carcinogen, CR is used to denote cancer risk. SF ($kg.d.mg^{-1}$) is the corresponding carcinogenic slope factor of the lifetime average daily dose (LADD). When $CR<10^{-6}$, it is considered that the risk is negligible. When CR is in the range of 10^{-6} to 10^{-4}, it suggested that there is a certain cancer risk. When $CR>10^{-4}$, it indicates that there is a significant cancer risk. The RfD and SF values of metals analyzed in the present study are presented in table 3.

Results and discussion

Total concentrations and descriptive statistics of urban dust and surface soil samples are summarized in table 4. The urban dust mean concentrations of the studied metals above to background value ratios decreased as the order of Pb (3,21 times) > Ni (1,72 times) > Zn (1,52 times) , while Cu mean content did not exceed the background values (0,43 times). A similar behavior was obtained for surface soil mean concentrations: Pb (4,14 times) > Ni (3,26 times) > Zn (2,20 times) and Cu (0,92 times).

The standard deviation (SD) and coefficient of variance (CV) indicated that there was wide variation in some metals concentrations in dust and soil showed samples. According to the study by Phil-Eze [33], CV ≤ 20% indicated low variability, 21% < CV ≤ 50% was regarded as moderate variability, 51% < CV ≤ 100% indicated high variability, and CV > 100% was considered very high variability. In this study, concentrations of Ni and Pb maximum variability with CV of 131% and 104%, respectively, while for urban dust was Pb with CV of 116%. The very high CV values of Ni and Pb and high CV of Zn for studied soils, and very high CV of Pb and high CV of Ni and Cu for studied dusts, reflected their heterogeneity in the Regla town soil environment, which further indicates the existence of anthropogenic sources in the studied area [34, 35]. This is in correspondence with the Ni, Pb and Zn distributions in Regla soils (figure 2, left) and studied metals in urban dusts (figure 2,

Table 3. The reference doses and slope factor of metals in the present study [32].

	Cu	Ni	Pb	Zn
R_fD_{ing}	4.00E-02	2.00E-02	3.50E-03	3.00E-01
R_fD_{inh}	4.02E-02	2.06E-02	3.52E-03	3.00E-01
R_fD_{dermal}	1.20E-02	5.40E-03	5.25E-04	6.00E-02
SF	-	8.10E-01	-	-

Table 4. Statistical results of the Ni, Cu, Zn and Pb concentrations ($mg.kg^{-1}$) in urban dust and surface soils of Regla town and the corresponding IPI.

	Metal	Maximum	Minimum	Mean	Median	Skewness	Kurtosis	SD	CV(%)
Urban dust	Ni	364	30	100	59	2,02	3,30	96	96
	Cu	110	16	37	27	2,05	4,94	27	73
	Zn	371	82	230	222	<-0,01	0,34	68	30
	Pb	378	21	62	46	4,33	19,63	72	116
	IPI	4,0	0,5	1,7	1,0	2,0	3,7	0,8	47
Surface soil	Ni	798	44	189	92	2,15	4,05	248	131
	Cu	180	14	75	73	1,55	4,35	42	53
	Zn	1048	87	285	247	2,63	4,85	284	86
	Pb	431	28	116	73	2,31	5,71	121	104
	IPI	4,9	1,2	2,6	1,9	0,5	-1,7	1,4	54

right), where the respective metal content hot spots are clearly identified, allowing the identification of the potential pollution sources. On the other hand, the Cu distribution in soil is more homogenous through the studied stations.

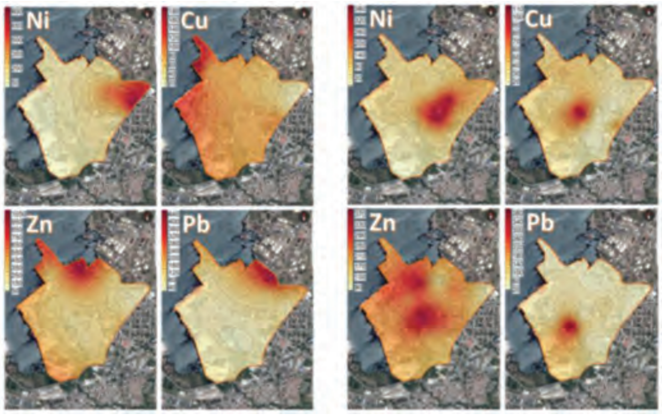

Figure 2. Spatial distribution of concentrations of Ni, Cu, Zn and Pb in surface soils (left) and urban dusts (right) from Regla town.

The skewness and kurtosis (K–S) test confirmed that the concentrations of the studied metals did not follow a normal distribution, which was normal in geochemical variables. The skewness values of practically all studied metals in surface soils and urban dust were positive. It manifested that their distribution patterns were right-skewed related to the normal distribution. The skewness of Pb was maximum in both, soil and dust. The exception was Zn in urban dust showed negative, indicating that its distribution pattern is left-skewed compared with the normal distribution. However, its absolute values of skewness is lower than 1, which confirmed that Zn--content in dust practically follows a normal distribution.

The spatial distribution of Integral Pollution Index for surface soil and urban dust (figure 3) was an extremely crucial tool, in order to assess the potential main pollution sources in the studied area. The behavior of the soil

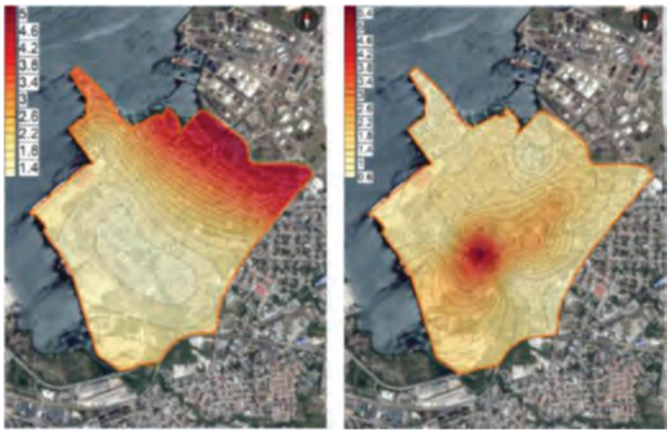

Figure 3. Spatial distribution of the Integral Pollution Index in surface soils (left) and urban dust (right) from Regla town.

IPI (figure 3, left) shows the high impact induced by the oil refinery, for very long time, to the urban soils of Regla town. The comparison with studied metal distribution in Regla soils (figure 2, left) allows to confirm that Pb, Zn and Ni exhausts from the refinery, are deposited in soils along the town-refinery common border, inducing a very high pollution in this area (an average local soil IPI of 7.5). On the other hand, the hot spot observed for the urban dust IPI spatial distribution correspond to station 7 (IPIdust (st.7) = 4.0), where a new temporal fuel-oil electric power plant was installed, after then the mentioned power station was closed.

The potential ecological risk for each studied metal in soil and urban dust from Regla town are presented in figure 4 (up). It could be found that the severity of pollution of the studied trace metals decreased as follows: for soil as Pb > Cu > Ni > Zn, whereas for urban dust as Pb > Ni > Cu > Zn. However, for the major part of the studied stations, the potential ecological risk must be considered as Low. The only exceptions are Pb-soil-Eri in the stations 1 (considerable) and 17 (moderate),

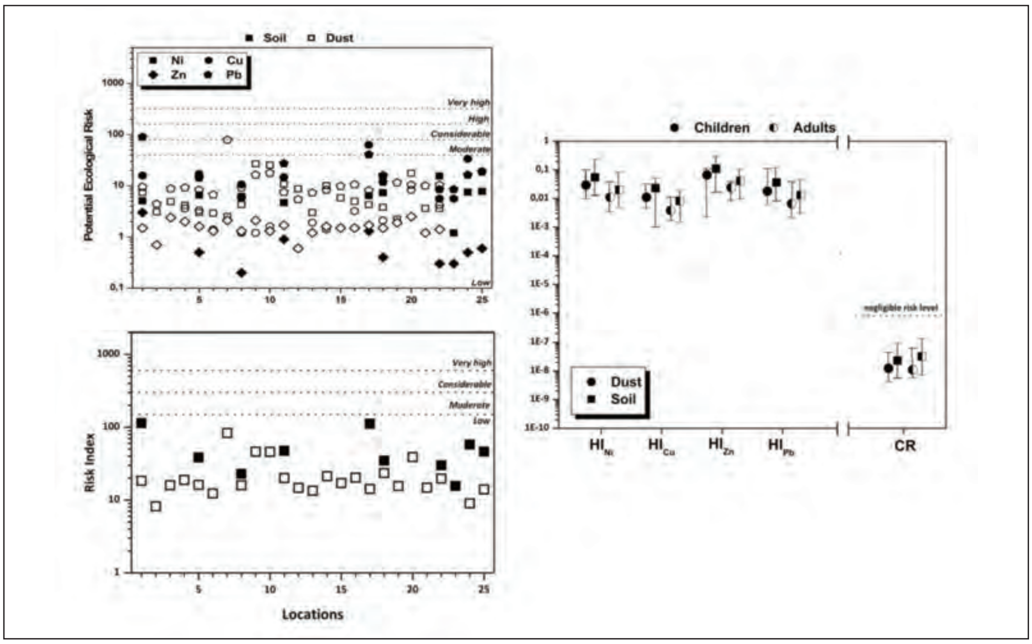

Figure 4. Potential ecological risk (left-up) and Risk index (left-down) and .the averages and ranges of metal Hazard Indexes (HI) and Ni-Carcinogenic Risk (CR) (right) in surface soils and urban dust from Regla town.

soil-Cu-Eri in the station 17 (moderate) and dust-Pb-Eri in the station 7 (moderate). These results are in agreement with the IPI distribution for soil and dust, respectively. The Risk Index (figura 4, down) classified as Low for both, soil and dust, in all studied stations, independently of the relatively high metal contents determined in some of the studied stations. In correspondence, the determined Ni, Cu, Zn and Pb hazard indexes and Ni-carcinogenic risk (figure 4, right) are in normal levels.

Conclusions

In correspondence with the obtained results, the relative high Ni, Cu, Zn and Pb contents, measured in surface soils and urban dusts from Regla town are not the cause to develop by the local population a carcinogenic and non-carcinogenic diseases mentioned in Cuellar Luna and et. al. [11]. In this sense, the study of the content of other heavy metals as As, Cd, Hg, etc., is recommended.

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Recibido: 18 de marzo de 2021
Aceptado: 21 de julio de 2021