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EVOLUTION OF ENRICHMENT OF SEDIMENTS BY TRACE METALS (NI AND ZN) IN A DAM OF URBANIZED WATERSHED

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Abstract

Sediments represent and provide a response to the condition of the environmental system in which they are found, acting as a point for deposition of contaminants. Upon developing studies of sediment columns, it is possible to obtain the historic records of substances arising from activities developed in the watershed over recent years. In this context, the present study investigates the enrichment of the sediments produced in an urban residential catchment area by the trace metals zinc and nickel. The sediment fraction smaller than 63 µm was analyzed in three sediment cores from the dam. Chemical analyses verified the concentration of trace metals by the acid digestion method EPA 3050. In carrying out analyses of the evolution of urban settlement in the watershed, the values corresponding to natural and human-impacted areas were determined through the use of different remote sensing products: aerial photographs (from 1972 and 1991) and high resolution satellite images (from 2003 and 2008). Natural areas were reduced in four decades, and population density in already human-impacted areas proved to be an important factor for understanding the urbanization process of the area. All sediment samples analyzed showed Zn and Ni concentrations above the local background value and with a pattern of growth.

Keywords: urban sediments, urbanization, zinc, nickel.

1. INTRODUCTION

Rapid and unplanned urban expansion, together with inadequate soil use, is one of the main factors responsible for degradation of natural resources. Its consequences on water resources are severe damages not only through hydrological changes, but especially through the transported pollutant load, with impacts above all on the quality of life of the population, affecting the environmental balance of the areas drained by the watershed (Nascimento et al. 2005).

Among the human activities that characterize urbanization, the impacts generated by substitution of the original vegetation by impermeable areas (Packman et al. 1999), the emissions of large quantities of sewage without treatment, and the addition of chemical contaminants through the most diverse sources (Poleto & Laurenti 2008) stand out. Nonpoint source or diffuse pollution is associated with soil use activities whose generated load affects waterways mainly by rainwater activity (SEPA 2011) and this becomes a difficult obstacle for watershed management due to the nature of the processes involved and the difficulty of developing procedures for elimination or minimization of the impacts, which are quite varied (Simões 2004). The runoff generated on impermeable surfaces, in addition to increasing the risk of flooding, may be highly polluting, being composed of a mixture of hazardous substances such as metals, pesticides, oils and hydrocarbons, aggregated to sediments or otherwise, and which compromise the quality of waterways. Most of waste water discharges do not receive any treatment before entering in rivers or streams and this may lead to severe ecological damages through reduction in dissolved oxygen available to the biota and also through bioaccumulation of toxic substances (EA 2011).

According to Edwards (2007), sediments may be defined as rock and soil fragments separated by weathering processes, as well as particles of organic origin. The mineral particles have permanent charges due to the imbalance on the mineral surface, which leads to the attraction of cations (Inda Jr et al. 2004); there are also the variable charge components, which maintain a relationship with the pH of the medium. Moreover, organic surfaces or surfaces covered by organic matter have functional groups that may act as coordinating sites to which the metallic cations may bind (Mariani 2006).

Sediments act as a record, a deposit for the contaminants present in the environment in which they are transported. Upon analyzing the surface sediments, it is possible to determine the extent, the distribution, the origin and the possible risks of real contamination. The study of sediment columns, for its part, provides the historical record of the substances arising from human activities, or otherwise, which are developed in the watershed over recent years (Müller et al. 1977) through the overlaying of layers deposited in lentic environments.

In this context, the present study investigates the enrichment of sediments produced in an urban residential catchment area through the trace metals zinc and nickel, seeking to make deductions regarding the relationship between the urbanization process and the diffuse sources of pollution with the quality of the sediments deposited in the Mãe d'Água Dam Reservoir.

2. MATERIAL AND METHODS

2.1 Study area

The Mãe d'Água watershed is one of the components of the headwaters of the Dilúvio stream, an important waterway that extends to the municipality of Porto Alegre, capital of the state, cutting across it in an east-west direction and home to more than 250,000 inhabitants. The study area is located in the municipality of Viamão, in the Porto Alegre Metropolitan Region (RMPA), in the state of Rio Grande do Sul, Brazil (Figure 1).

Figure 1. Location of the study area.

The watershed under study is composed of four streams, for a total area of 3.52 km^2 and has the Mãe d'Água Dam as its mouth, which is situated in Federal University of Rio Grande do Sul (Figure 2). Climate in the region, according to Livi (1999), being situated at latitude 30°S and 100 km from the Atlantic Ocean, is classified as humid subtropical (Cfa), according to the Köppen (1928) classification. It is found in the southeast part of Santana Hill, an elongated granite formation with lightly rolling topography.

At the mouth of the watershed, there is an artificial lake, the purpose of the Mãe D'Água Dam, which was inaugurated in 1962, was to provide for the needs of the Federal University of Rio Grande do Sul (Fujimoto, 2001). Urban settlement of the area was spurred by establishment of the University campus. Evolution of settlement of the watershed, with population increase and different forms of intervention in the environment, led to transformations in its characteristics throughout the years. Part of the urbanization is legally regulated; nevertheless, there is also a large portion of habitations in areas of risk, such as riparian zones which are permanent conservation areas. In these irregular areas, there is no connection to the sewer line or even the septic tank and leaching-field type sewage systems, and access to other services occurs in a clandestine manner (Rangel 2008; Ungaretti 2010).

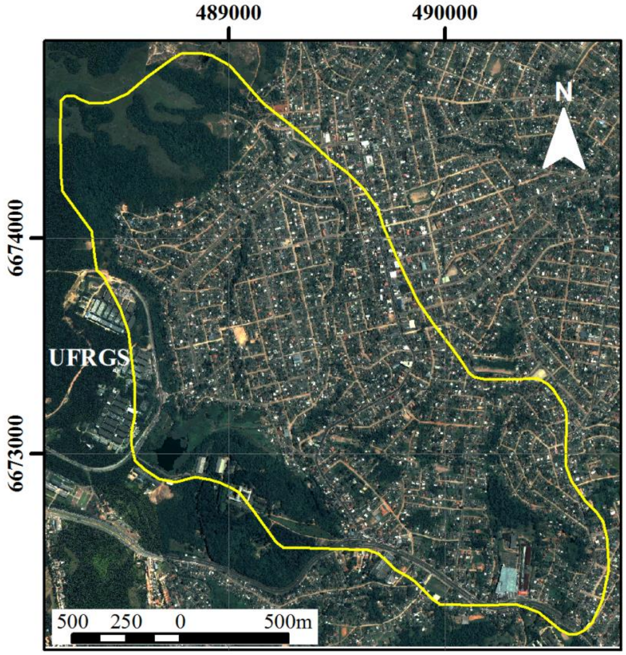

Figure 2. Satellite image of the watershed under study

2.2 Sampling of the sediment profiles

Sediment profiles were collected in December 2009. Three sediment cores were extracted and the location to each one of them is presented in Figure 3. The technique of percussion drilling was used, which consists of introducing a rigid cylindrical tube in the sediment at the bottom through impact produced in a manual fashion.

The sediment cores were cut into 5 cm sections, generating a large quantity of subsamples. For optimization of expenses related to metal concentration analyses, a selection of subsamples to be chemically analyzed was performed. For each sediment core, according to its total length (C1: 0.60 m; C2: 2.01 m; C3: 1.65 m), some strata were selected, seeking to distribute the interval between the subsamples selected in each sediment core in a uniform way. Thus, the subsamples selected for determination of metals are shown in Table 1.

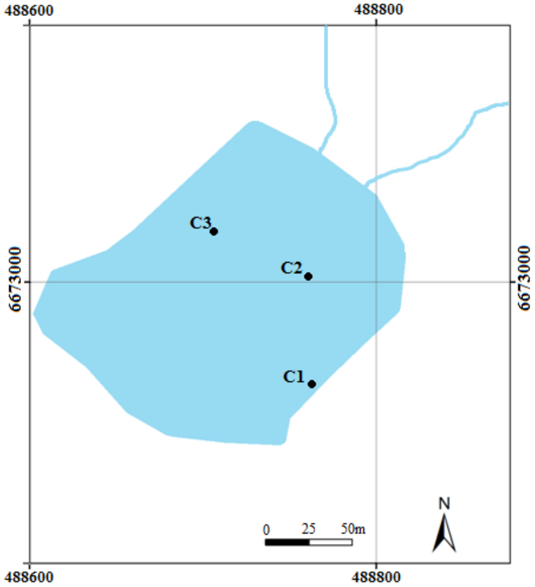

Figure 3. Location of three cores sampled in Mãe d'Água Dam

Table 1. Subsamples selected for analyses of metal concentration

Core 1	Core 2	Core 3
Stratum (m)	**Stratum (m)**	**Stratum (m)**
0.00 – 0.05	0.00 – 0.05	0.00 – 0.05
0.25 – 0.30	0.20 – 0.25	0.25 – 0.30
0.40 – 0.45	0.35 – 0.40	0.45 – 0.50
0.55 – 0.60	0.55 – 0.60	0.65 – 0.70
	0.70 – 0.75	0.85 – 0.90
	0.90 – 0.95	1.00 – 1.05
	1.10 – 1.15	1.20 – 1.25

	1.30 – 1.35	1.45 – 1.50
	1.50 – 1.55	1.60 – 1.65
	1.70 – 1.75	
	1.95 – 2.01	

2.3 Chemical analyses

After drying in a laboratory oven at a constant temperature of 40°C, the selected subsamples were made uniform in an agate mortar and then sieved in plastic equipment with mesh openings of 63 μm. Only the fine fraction (< 63 μm) was considered because it is the most significant in metal accumulation (Horowitz 1991; Mudroch et al. 1997; Poleto & Teixeira 2006; Martinez 2010). Five grams from each subsample were sent to the laboratory for evaluation of total metal concentration. Digestions were made in duplicate plus one blank (a blank sample is made using the same reagents and procedures, but without the addition of the sediment sample) for quality control of the analyses (Poleto & Gonçalves 2006).

The concentration of trace metals was verified by the acid digestion method USEPA 3050, which is directed to analysis of inorganic element concentration in sediments, sludge and soils, and was developed and adopted by the U.S. Environmental Protection Agency (USEPA). This methodology involves strong acid digestions of the samples, dissolving almost all the elements that may become bioavailable. For that reason, elements bonded in silicate structures are normally not dissolved by this procedure since they generally are not mobile in the environment (USEPA 1996). For implementation of the method, 1 to 2 g of sediment were used, which were digested by repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) 30%. After that, hydrochloric acid (HCl) was added for the purpose of releasing more resistant metals (USEPA 1996). In the final stage of the method, a reading of the extracts was made in the inductively coupled plasma optical emission spectrometer (ICP-OES), in the Soil Laboratory of UFRGS.

The background values used in this research were based on the work of Poleto (2007). These values represent a mean concentration representative of the natural concentrations of the metals under analysis in the study area through three compound samples of the region of the headwaters of the Mãe d'Água Dam watershed, in the upper Morro Santana, in areas that did not present human alterations.

For effective quality control of chemical analyses, standard reference materials were used in parallel with the samples analyzed, thus obtaining the range of precision of the samples, as well as allowing corrections of deviations found in the results. This quality control was in the range of 10% of the total samples made; in other words, for each 10 samples of sediment analyzed, at least 01 of the Standard Reference Material was analyzed, according to procedures used by the American laboratories of the USGS (Poleto & Teixeira 2006).

The analytical reagents and the extracting solutions prepared for the analyses had a high degree of purity. The water used for the dilutions was Milli-Q (extrapure) type. All the equipment and glassware used in the sample collection and sample processing procedures were washed with deionized water, remaining in a 14% (v/v) nitric acid solution for 24 hours and once more rinsed with deionized water.

2.4 Evolution of urban settlement in the area

For evaluation of the evolution of urbanization in the study area, aerial photographs and satellite images were used. Copies of photographs from the year 1972 and 1991 were obtained from the planning and management agencies of the Porto Alegre Metropolitan Region. Aiming to evaluate the most recent urbanization period of the area, an image from the Quickbird satellite was used from the year 2003, with spatial resolution of 0.60 m, which allows monitoring of activities that require high precision, such as urban mapping. For the year 2008, a product of the Sino-Brazilian satellite CBERS, HRC sensor, was used. Delimitation of the watershed was made on the software Idrisi Taiga® as of a digital elevation model (DEM) based on the 20 meter isolines of the Brazilian Army map at a scale of 1:50,000 (Porto Alegre, MI 2987/2).

About urban evolution of soil use, two classification categories were used: anthropic use and natural use. Comparing the years of study, the changes in the watershed can be observed.

3. RESULTS AND DISCUSSION

3.1 Enrichment of Sediments by Zinc

In urban areas, dust particles present in the traffic routes enter in contact with the residues from vehicle emissions and vehicle wear, which is one of the most important sources of trace metals (Sezgin et al. 2003), especially of Zn^{2+}. The vertical distribution pattern of Zn in the three sediment cores (C1, C2 and C3) taken from the Mãe d'Água Dam is shown in Figure 4. All the subsamples analyzed showed values above the local background (47.4 mg.kg^{-1}), indicating anthropic enrichment of these sediments deposited in the Dam. Periodic fluctuations were seen in the concentrations and, as a general pattern, an increase of the association of Zn with the sediments deposited at the bottom of the Dam. This trend is clear above all analysis of Cores 1 and 2.

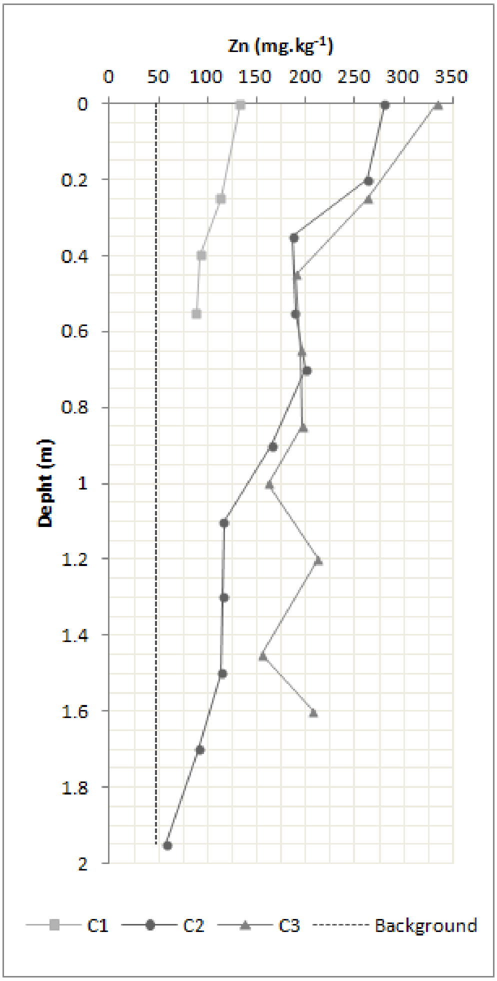

Figure 4. Zinc distribution (mg.kg^{-1}) in the sediments of the three sediment cores collected

The data in regard to variation of the Zn concentrations are shown in Table 2. Sediment core 1 (C1) is located nearest to the shore of the dam and has lower deposition and variation of material. The initial concentration found at the base of the sample was 88 mg.kg^{-1} and increased up to the value of 133 mg.kg^{-1}. The initial concentration found in C2 is very near the background established for the area (47.4 mg.kg^{-1}), showing that this collection point certainly covers a significant period of the history of the sediments deposited in the dam. This sample shows significant enrichment in comparison of the subsamples of the base (oldest extract: 57 mg.kg^{-1}) and of the top (most recent extract: 280 mg.kg^{-1}), showing approximately a fivefold increase (491%). Sediment core 3, for its part, has, in its older subsamples (from 1.6 to 1.0 m), quite oscillating concentrations, not initially presenting a continuous growth pattern in the base to top direction; nevertheless, as of 1.0 m in direction to the top and especially in the last 0.65 m of the sample, it comes to have a behavior similar to C2, with growing increases in Zn concentrations.

Table 2. Data of Zn (mg.kg^{-1}) in Mãe d'Água Dam sediments

	C1 (mg.kg^{-1})	**C2** (mg.kg^{-1})	**C3** (mg.kg^{-1})
Mean	107	161	213
Standard Dev.	21	51	55
Minimum	88	57	155
Maximum	133	280	334

The zinc concentrations have maximum values in the upper parts of the three sediment cores and decrease in an accentuated manner as depth increases. The oscillations found in Zn sorption to the oldest extracts, as seen in sample 3, is related to the dynamic of the trace metals between the water depth and the sediment particles (Koretsky et al. 2006). According to Rubio et al. (2001), there may be zinc migration to the upper sediment strata during degradation of organic matter. Another factor, indicated by Badr (2009), is the sensitivity of the Zn sorption phenomena to the reducing conditions of the environment, showing the instability in formation of compounds and precipitates. The dynamic nature of interstitial water and the geochemistry of the solid phase of other elements suggest that the trace metals sorbed in the sediments may undergo speciation due to significant seasonal variations in speciation (Koretsky et al. 2006).

3.2 Enrichment of Sediments by Nickel

The results obtained for the trace metal nickel are shown in Figure 5. Different from the results found for zinc, the greatest associations between nickel and the sediments are found in the oldest strata; this is quite evident upon checking C2, which presents its maximum value (24 mg.kg^{-1}) in its base subsample. Recent sediments, represented by the sample at 0.0 m, did not present the greatest concentrations. In Sediment Core 1, this is found at 0.25 m (16 mg.kg^{-1}), whereas for C2, a significant peak is at the depth of 0.35 m (18 mg.kg^{-1}).

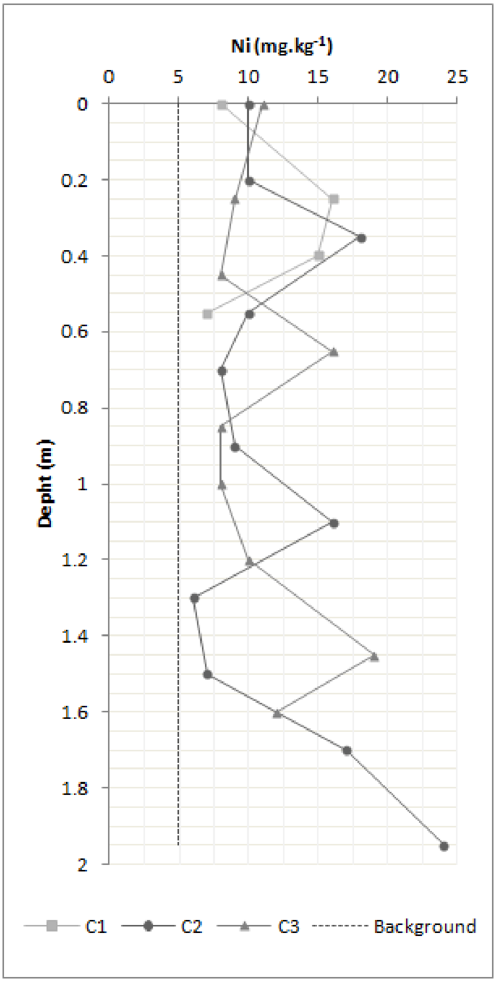

Figure 5. Nickel distribution (mg.kg^{-1}) in the sediments of the three sediment cores collected

The selectivity of sorption between the metals, in which zinc presents greater sorption potential than nickel, and action of the effect of mass exercised by the large quantity of Zn are important factors for understanding the lower concentration of nickel associated with the sediments analyzed. An important fact for understanding of the greater depth in which one finds the higher values of Ni is found in the presence of a metallurgical industry which, according to Bonetto (2010), operated in the narrow period of one decade (1963 ~ 1970), contemporaneous to the beginning of the process of deposition of sediments in the dam (also inaugurated in 1963).

Oscillations in the concentrations seen in the three sediment cores are frequent and the data are presented in Table 3. Nevertheless, such abrupt seasonal variations in the availability of sources of this metal for sediments of the area are not very probable, above all if the urbanization rate is considered, which shows growth throughout the period analyzed. According to Zwolsman et al. (1996), variation in the nickel, content in the deeper layers, is due to sorption of the metal to manganese oxyhydroxides. The state of reduction of the sediments determines the mobilization of associated Mn and Ni will occur. Degradation of organic matter, abundant in the material transported to the dam, is a source for possible alterations in the pH and Eh of the deposited sediments, also affecting the mobility of nickel in the sediment profile (Sunderman & Oskarsson 1991) (Badr et al. 2009), such that metal is not adsorbed by the dam sediments.

Table 3. Data of Ni (mg.kg^{-1}) in Mãe d'Água Dam sediments.

	C1	**C2**	**C3**
	(mg.kg^{-1})	(mg.kg^{-1})	(mg.kg^{-1})
Mean	12	12	11
Standard Dev.	5	6	4
Minimum	7	6	8
Maximum	16	24	19

Moreover, Saifullah et al. (2002); Lepp & Madejón (2007) indicate the interaction between trace metals and plants, which may also be considered in the analysis of the data in reference to contamination of the Mãe d'Água Dam, since there is an abundance of macrophytes fixed to the surface substrate of the sediments deposited in the dam, as may be seen in Figure 6(A). Specific factors in reference to the sample collection location should also be taken into consideration; thus, in Figure 6(B), elements found in the subsample corresponding to 1.2 m depth of sediment in Core 3 are shown. These elements can provide a punctual variation, which affects only the location where the sampling was done.

In addition to being active in control of sorption of pollutants to the sediment, the presence of plant residues (OM) and metallic debris, like those found in the subsample at 1.2 m of C3, show the complexity of the material which is transported from urban areas, like the study area, and accumulated in lentic aquatic environments, like Mãe D'água Dam.

In spite of the variations, in analysis of the mean value data, uniformity in distribution of contamination by nickel in time and also among the collection points analyzed is observed. The minimum values detected were similar: 7 mg.kg^{-1} in C1, 6 mg.kg^{-1} in C2 and 8 mg.kg^{-1} in sediment Core 3. Even so, as of evaluation of the background value of the area, an increase in the presence of this contaminant arising from human activity is seen in the material transported by the watershed. The data collected here show the existence of interaction among the sediments of the area and the nickel compounds, but also displays a possible fragility of this interaction in view of the oscillating patterns of association between metal-sediment throughout the sediment column. Solubilization of nickel compounds, known to be carcinogenic, is a risk that needs to be evaluated, above all in case of performing dredging of the material deposited in the dam.

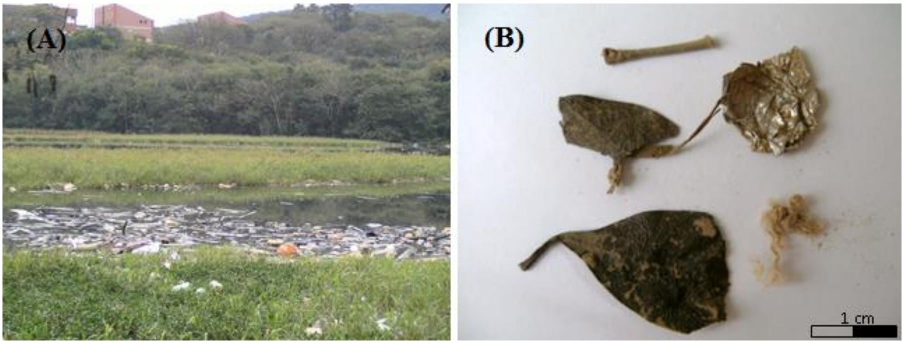

Figure 6. (A) Macrophytes and solid residues on the dam surface (B) Metallic, cloth and plant debris from the 1.2 m subsample of C3

Diffuse sources of pollution, characteristics of urban residential areas, provide an immense variety of pollutants to the environment and, among them, trace metals such as nickel and zinc stand out. The absence of household sewage treatment and inadequate disposal of these residues provide a large load of organic matter, a fundamental agent in control of sorption and desorption processes of metals to sediments.

3.3 Evolution of urban settlement

In Figure 7, the maps resulting from interpretation of the aerial photographs and satellite images are presented. It may be perceived that already in 1972 (A) most of the watershed area showed human impact. Building of the first structures in the area goes back to the 1950s. In the 1960s, dam construction work and establishment of the University were fundamental for growth of neighborhood.

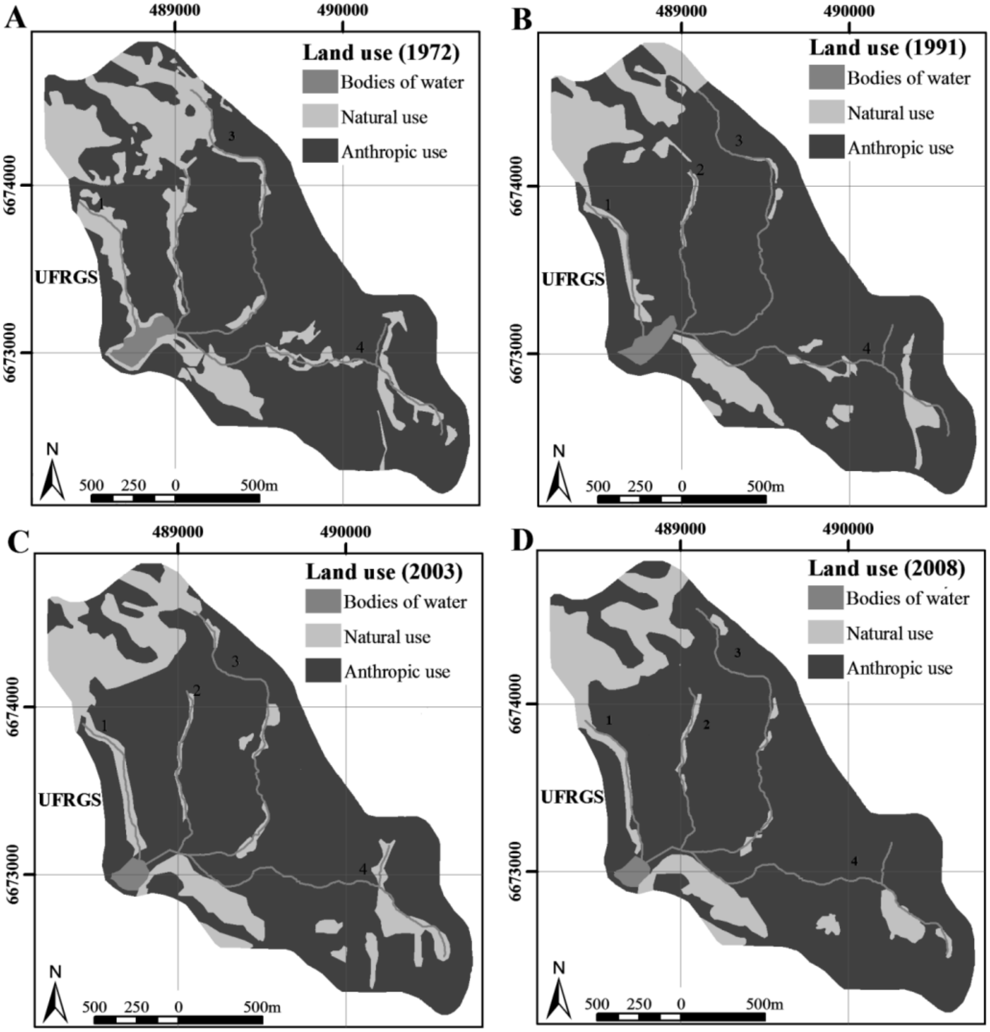

Figure 7. Interpretations of land use in the watershed under study in the years of 1972 (A) and 1991(B) based on aerial photos, and in the years 2003 (C) and 2008 (D) based on satellite images

Already in 1991 (B), the advance of urbanization occurred in the direction of the springs and over the areas of riparian forest, which should be under permanent conservation; streams 2 and 3 exhibit thin strips of riparian vegetation, with the areas near the dam were completely altered. The preserved areas, categorized as natural use areas, are found in the upper portion of the watershed, in areas with greater slope and those adjacent to University, located at the east margin of the dam.

The data related to evolution of human settlement in the watershed are shown in Table 4. It can be observed the variation is small in the last two periods evaluated – the years 2003 and 2008 (C and D). This is due to the interpretation criterion established and the settlement characteristics of the area; areas that were already included in the anthropic category had reached their maximum.

Table 4. Results of soil use evolution in the study area

Year	Anthropic (km^2)	Natural (km^2)	Dam (km^2)
1972	2.67	0.80	0.05
1991	2.90	0.58	0.04
2003	2.97	0.53	0.02
2008	2.99	0.51	0.02

In Figure 8, the densification of settlement of the areas may be seen. The growth of low-income families and the lack of alternative areas have created agglomeration of various households on the same lot.

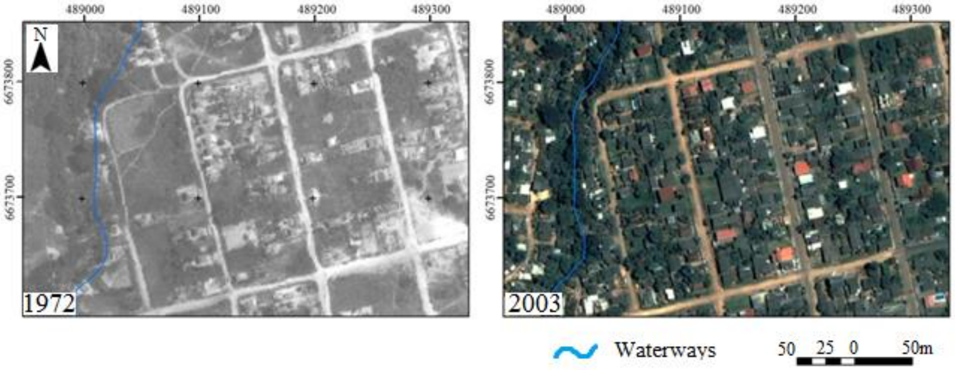

Figure 8. Densification in settlement of human impacted areas, in 1972 and 2003

4. CONCLUSIONS

Sediments with granulometry < 63 µm deposited in the dam showed affinity to the trace metals zinc and nickel, as all the strata of the sediment column analyzed showed concentration above the background value and, therefore, show the existence of enrichment of sediments by these elements. Considering that, the study area historically is characterized by residential settlement, with the predominance of pollution from diffuse sources. The urban dynamic is the main agent which supplies pollutants to bodies of water; therefore, the increase of nickel and zinc concentrations in analysis of the vertical sediment profile shows the increase of pollution transported to the dam in the last four decades.

Zn concentrations increased in a more defined and continuous manner than Ni concentrations, showing growth in the three sediment cores analyzed, affirming the high affinity of this element to the sediments composed of the organic fraction, abundant in the area due to lack of waste treatment. The oscillations identified in the sediment profile for the nickel concentrations may be explained by vertical migrations of it by means of interstitial water, the short past industrial activity in the area, as well as by the effect of mass exercised by zinc, present in large quantity in the sediments deposited in the dam.

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