

## THE RELATIONSHIPS OF METALS IN RIVER SEDIMENTS (NAHR-IBRAHIM, LEBANON) and ADJACENT FLOODPLAIN SOILS

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### ABSTRACT

Metals in bed-load sediments are multi-source, namely: weathered rocks, metals discharged directly into river, and eroded riparian soils. Contaminated flood plains represent storage system for releasing pollutants to rivers. An essential first step towards modeling this storage is to investigate the relationship. The objective of this study is compare the composition (CaCO<sub>3</sub>, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn) of bed sediments and adjacent flood soils to evaluate compositional relationships for a river (Nahr-Ibrahim, Lebanon) characterized by marked differences in discharge between winter and summer. The comparison would draw first preliminary conclusion of floodplain storage for future pollution and second would approximate sources of metals in bed sediments. The bedrock of the river is mainly limestone, the floodplain soils are mostly Mediterranean type, and water column is saturated with carbonate species.

Bed-load sediments and soil samples pairs were collected from the mouth of the river up to 13 km, during the dry season. The sediment and soil samples were digested using *aqua regia* to extract metals, and quantified by ICP-MS techniques. Inorganic carbon was determined by back titration and organic carbon by LOI method. The data revealed that compositional CaCO<sub>3</sub> in sediments is higher than soils, explained by carbonate precipitation from Ca-saturated water column. The metal content of Fe and Mn are derived naturally in soils and sediments. Down stream, near coastal urban zones, as river opens to floodplain soils, the pollutant metals are highly enriched in soils than sediments. Therefore, sources of these metals are due to eroded soils during wet season and precipitating carbonates in dry season reduced original metal pollutants metal concentration. However, upstream (in inland) the concentration of the metals Cd, Cr, Cu, Pb and Cd were higher in sediments than soils; this suggested that sources of metals in bed sediments are due to direct discharge of these metals into river channel.

**Key words:** contaminated floodplains, bed-load sediments, pollutant storage system, metals, sources of metals, Nahr-Ibrahim River, Lebanon.

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## 1. INTRODUCTION

Lebanon has abundant surface and ground water yet current rates of water consumption are unsustainable because the supply of potable water has been reduced by pollution resulting from population growth, industrial development and expansion of irrigated agricultural land (El-Fadel et al., 2000). Unregulated exploitation and over-pumping of private wells along the populous coast have impaired aquifer quality through saline intrusions (Jurdi, 1992, 1998, Khair et al., 1994, Sene et al., 1999).

There are neither metal mines nor smelters in Lebanon. The industrial sector is limited to food and beverage manufacture, tanneries, soap and detergent production, paint manufacture, textiles, plastics and metal processing (DAR AL-HANDASA, 1996, ECODIT, 2002). Many of the industries discharge their wastes to land or nearby streams without treatment (Stephenson et al., 1997; Jurdi, 1992, 1998). Lebanese rivers are also subject to pollution from other sources: urban run off, vehicle exhaust fumes containing lead, domestic waste discharges and agricultural discharges (Jurdi et al., 2002). Greater use of surface water is now considered a priority to meet national water demands (Jurdi et al., 2002): e.g., technical studies have recommended that Lebanon build 16-23 dams in various parts of the country (Amery, 2002). A better understanding of the transport and chemistry of metallic pollutants in Lebanese rivers is perceived as an important contributor to framing an effective water usage policy.

Potentially toxic metals (e.g., Pb, Zn, Cd, Cu) in effluents enter rivers and sorb to or, later, desorb from suspended and bed sediments. When a river floods, contaminated sediment is deposited on the soils of the floodplain. Floodplain soils are also subject to contamination by industrial discharges direct to land. Surface wash and bank collapse return these sediments to the channel. Further downstream contaminated sediments may be redeposited on the floodplain in a repetitive cycle of transportation, deposition, mobilization and further transportation until, eventually, the contaminated sediments reach the river mouth. There are many knowledge gaps in the understanding of this sediment delivery process.

Floodplains are a temporary storage system for metal pollutants in river basins (Bradley and Cox, 1990). Depletion by leaching through soil is slow but residence times are shortened substantially by physical remobilization (Foster and Charlesworth, 1996). Walling et al. (1998) have suggested that, for two British rivers, between 39 – 49% of the annual suspended sediment load is subject to overbank deposition thereby entering long term storage. Recognition of this long-term storage property of overbank sediments has stimulated interest in using alluvial soils for regional geochemical mapping (Macklin et al., 1994; Otteson et al., 1989). However, there are few studies which relate bank soil and

bed sediment composition or which identify suspended sediment provenance (Carter et al., 2003). The published literature is dominated by work on soil contamination from the industrialized countries (Förstner and Müller, 1981; Bradley and Cox, 1986; Macklin and Dowsett, 1989; Ligh, 1994; Brüggmann, 1995; Kern and Westrich, 1995; Leece and Pavlowsky, 1997; Lin et al., 1998) and there is a paucity of information for underdeveloped countries such as Lebanon.

This research took place in the lower Nahr-Ibrahim valley of Lebanon. It is a continuation of work investigating the transport, deposition and speciation of potentially toxic metals in this nationally important river in the Lebanon limestone terrane (Korfali and Davies, 2000, 2003, 2004). The objective of the study is to compare the composition (CaCO<sub>3</sub>, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn) of bed sediments and adjacent floodplain soils to evaluate compositional relationships for a river characterized by marked differences in discharge between winter and summer. The comparison would draw, first, preliminary conclusion of flood plain storage for future pollution and second would approximate sources of metals in the bed load sediment.

## 2. STUDY AREA

The Nahr-Ibrahim basin covers 330 km<sup>2</sup> in central Lebanon and stretches 30 km inland from the Mediterranean Sea. The main sources are the Afqua and Roueiss springs located at an altitude of 1200m on Mount Lebanon. Much of the valley is a canyon-like, occasionally broadening out to narrow floodplains, before entering the coastal lowlands. The river discharges into the Mediterranean Sea south of the city of Byblos (Jubayl). This research is concerned with the lower 13 km and comprised five sites numbered sequentially upstream from near the mouth (Table I, Fig. 1).

Site 5 is located in a gorge section; and the river (potentially) receives urban effluents and industrial effluents from plastic, pesticide, paint, galvanizing, electroplating works and ferrous metal fabricating from the town of Yahschouch. Lower down, Site 4 is adjacent to a large (now-closed) limestone quarry. Between 1996 and 1999 the character of Site 3, a narrow floodplain section, changed from rough agricultural land to horticultural (glasshouse) use. On a limestone ridge overlooking the valley there is major urban development and plastic and insecticide factories. Effluent from one of these been seen draining down the limestone bluff. Urban effluents and a variety of industrial sources potentially affected sites 1 and 2. Discharges in the river basin are generally unregulated.

Most of the rocks exposed in the Nahr-Ibrahim valley are fractured karstic limestone of Cenomanian age and fractured karstified massive dolomite, dolomitic limestone and bedded limestone of the Jurassic period. Sandstone and sandy limestone are of limited occurrence. Rocks outcropping in the bottom of the valley belong to the middle and upper Cretaceous period (Sannime and Maameltain Formations) and comprise limestones and dolostones. Basaltic rocks outcrop upstream and Quaternary

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alluvial deposits floor the coastal fringe at the river mouth (Abedel-Rahman and Nader, 2002). The X-ray diffraction data for sediments from Nahr-Ibrahim (Korfali and Davies unpublished) indicate the dominance of calcite, dolomite and quartz in the mineralogy of the < 75  $\mu\text{m}$  fraction. The water chemistry is dominated by carbonate bedrock, saturated with carbonate species (Korfali and Davies, 2004).

Zurayk (1995) has described Lebanese soils based on physiographic zones. Red Mediterranean soils, associated with hard limestone occur widely in Lebanon. They can be found in the coastal area, southern plateau, Lebanon mountain range and central Bekaa valley, under a variety of vegetation and climate (Darwish and Zurayk, 1997). Loosely described as Red Mediterranean soils, they are taxonomically, Ustalfs and Xeralfs. Solum iron contents range from 6.5 – 24.8%  $\text{Fe}_2\text{O}_3$  and colors (hue) are reddish and reddish brown (2.5YR and 5YR) (Darwish and Zurayk, 1997). The riparian soils of Nahr-Ibrahim vary from calcareous brown lowland soils (formed on the marls and the soft limestone) to typical Red Mediterranean soils.

The climate of Lebanon is “Mediterranean type”, that is humid to sub-humid in the wet winter season and subtropical in the summer dry season. Precipitation is limited to 80-90 days in winter and most rain falls between November and April: there is no rainfall in June, July and August (Sene et al., 1999; Amery, 2002). The annual average precipitation for Lebanon is 832 mm; the high mountain areas receive 1400 mm annually and the study area receives 1200 mm annually (Khair et al., 1994). The average annual discharge of Nahr-Ibrahim River in the year 1999 was 10.70  $\text{m}^3/\text{s}$ , the average discharge for the wet season was 17.47  $\text{m}^3/\text{s}$  and that of dry season was 3.99  $\text{m}^3/\text{s}$  (Lebanese Ministry of Hydraulics and Electrical Resources-MHER).

Active down stream flushing of sediments is restricted to the high discharge episodes of the winter-wet season. In the wet winter season, the river carries a brownish suspended sediment load whereas during the dry season, the river runs clear. The discharge is low and the water flow is maintained by bed recharge from ground water (Korfali and Davies, 2000, 2004) In Classical Greek time the Nahr-Ibrahim was known as the river “Adonis” for it was believed the reddish winter color was due the blood of a fertility God (Grant, 1988). Thus, accelerated soil erosion has been a characteristic feature of the catchment for over 2000 years. The high vulnerability of the soil cover to erode is due to intensive run-off on sloping terrain, rendering the surface very unstable with various forms of mass movements (Khawlie, 1985). An estimated 50 –70 tons/ha/year of soil is eroded in areas of high topographic relief (FAO, 1986). This vulnerability of soil erosion has been discussed by Khawli et al. (2002) and it constitutes a major environmental problem.

### 3. MATERIALS AND METHODS

#### 3.1 Sampling methods

The sampling locations (5 sites) chosen for this (1999) study were the same as those described by Korfali and Davies (2000) for work in 1996 (Fig.1). The sites were chosen according to the observed possibility of metal contamination by domestic wastes, industrial discharges and heavy vehicle motion and accessibility to the river channel.

Bed sediments, adjacent floodplain soils and water samples were collected simultaneously during the dry season (October). The method of collecting bed sediments, followed published methods for collection in shallow streams (Chapman 1992, Lewis and McConchie, 1994) and these methods were reported in full in an earlier paper (Korfali and Davies, 2000, 2004). At each location site, composite samples were taken (four sub-samples) and bulked in one polyethylene bag. Representative composite flood plain samples were taken (six sub-samples) for each location site; the soil samples ranged between 15 – 25 m from river's channel.

Measurement of water pH, temperature and electrical conductivity were made in the field using a Hach Model 44600 Meter. Water samples were collected using one-litre polyethylene bottles that had been soaked overnight in 10% (v/v) nitric acid. The bottles were rinsed twice with the river water, and then filled with water running in the direction of river flow. The water bottles were labeled with the corresponding sampling site, time and date of collection. The method of collecting water followed those described by Rump and Kirist (1992) and APHA et al. (1995).

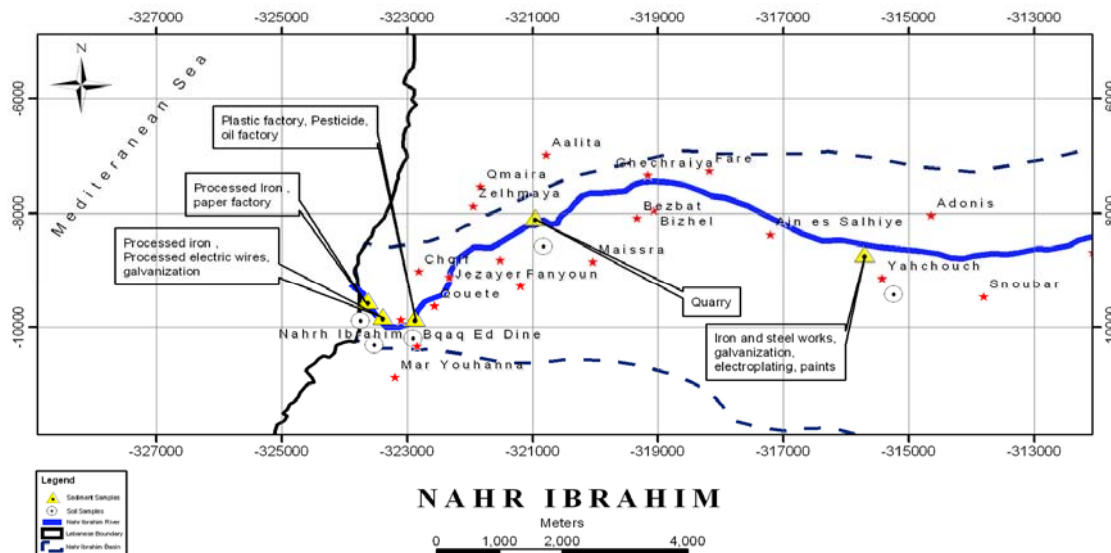


Figure 1. Nahr-Ibrahim sampling sites

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### 3.2 Water analysis

After arriving at the laboratory, water samples were immediately filtered through 0.45  $\mu\text{m}$  pore cellulose filters by vacuum suction into Pyrex™ flasks that had previously been washed in acid. The water samples were immediately taken to store (maximum time of storage 24 hours) at 4 °C. The preservation procedures followed the guidelines of the United States Environmental Protection Agency (US EPA, 1990) and the International Standard Organization (EN ISO, 1995). Samples were analyzed for alkalinity, calcium and magnesium hardness, chloride, nitrate, ammonia, sulphate and phosphate. The various analytes were determined using standard procedures (APHA et al., 1995): - the titration procedures for alkalinity (0.01M  $\text{H}_2\text{SO}_4$ ),  $\text{Cl}^-$  (0.007 M mercuric nitrate), Ca and Mg hardness (0.01M EDTA), and spectrophotometric methods for ammonia (Direct Nesslerization),  $\text{NO}_3^-$  (Cadmium reduction),  $\text{SO}_4^{2-}$  (turbidimetry) and  $\text{PO}_4^{3-}$  (Ascorbic acid).

### 3.3 Sediment and Soil treatment and analysis

Sediment and soil samples were air dried and sieved and the <75  $\mu\text{m}$  fraction was used for analysis. The total organic contents of sediment and soil were determined gravimetrically by the loss on ignition method at 450°C (Davies, 1974) and the amount of inorganic carbonate carbon (% $\text{CO}_3\text{-C}$ ) was determined by acid/alkali back titration method (Nelson, 1982). Total metals in sediment and soils were extracted using one-gram sample with 10 mL hot aqua regia and stepwise heating (2 hrs at 25 °C, 2 hrs at 60 °C, 2 hrs at 105 °C and 3 hrs at 125 °C. (Evan, 1991). Metals in solution were subsequently determined by ICP-MS. Calibration solutions were prepared from Fisher Chemicals USA stock solutions (1 mg metal/ mL in 2%  $\text{HNO}_3$ ). Analytical quality control was monitored by analysis of standard certified references of sediment and soil (sediment RM 8704, Soil SRM 2586 from National Institute of Standards and Technology- NIST, USA). Eight replicates of certified sediment and soil were analysed by the same experimental procedure of metal extraction and analysis: The percentage relative standard deviation (%RSD) was <10%.

## 4. RESULTS AND DISCUSSION

### 4.1 Water

Results for pH, temperature, calcium and bicarbonate ( $\text{HCO}_3^-$ ) concentrations are given in Table 1.

Table 1. Measured master water variables and SI values at all sites

Sampling sites	pH	Temperature °C	Ca <sup>2+</sup> (mmol/L)	HCO <sub>3</sub> <sup>-</sup> (mmol/L)	SI* index (Calcite Mineral)
1	8.4	24.5	1.098	3.65	0.89
2	8.2	24.2	1.047	3.04	0.62
3	8.7	26.8	0.998	3.46	1.13
4	8.4	25.0	0.988	3.03	0.79
5	7.9	19.0	0.998	3.24	0.26

\*SI –Saturation Index

Mean water temperature (23.9°C) is typical for the sampling dates (October) in autumn, late in the dry season. There was little variation along the river except for site 5, furthest upstream, where water temperature was 19°C. The pH range (7.9-8.7) is typical for a limestone drainage basin. The observed calcium concentration was 1.00 – 1.10 (mean 1.03) mmol/L.

Ion speciation was investigated using the AQUACHEM(™) application which interfaces with the United States Geological Survey geochemical modeling program PHREEQC (Waterloo Hydrogeologic, Inc. 1998). Results showed that the computed dissolved Ca<sup>2+</sup> values were rather lower (0.88 – 1.01, mean 0.95, mmol/L respectively) than the observed concentrations.

The saturation index (SI) index of calcium with respect to the mineral calcite was also computed. ( $SI = \log(Q/K_{\text{mineral}})$  when  $Q = IAP$  (ion activity of mineral) and  $K_{\text{mineral}} =$  the solubility product constant of mineral (Langmuir, 1997) The SI values are reported in Table 1. Ranges of  $SI = 0 \pm 0.5$  are generally considered to be within the equilibrium zone of the mineral (Deutsch, 1997). At all sites the SI values of Ca<sup>2+</sup> in relation to calcite are slightly positive (mean SI = 0.61 and Table 1); calcium carbonate would therefore be expected to precipitate from solution.

The oversaturation SI values at most sites and the slightly lower computed dissolved Ca<sup>2+</sup> concentrations compared with the observed values are possibly due to the microfilter size (0.45µm) which conventionally distinguishes between dissolved and colloidal forms. However, Benoit and Rozan (1999) have shown that a much smaller pore size (1µm) is required to exclude colloids.

It is concluded from the water chemistry that Nahr-Ibrahim water column is saturated with carbonate species and forces calcite to precipitate on any convenient nucleus lying on stream bed. Metal pollutants then would be expected to co-precipitate with the carbonates or be sorbed on to the carbonate precipitates.

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## 4.2 Non-pollutant Metals

Calcium, iron and manganese are not normally regarded as polluting metals. In earlier papers (Korfali and Davies, 2000, 2003, 2004) concerned with Nahr-Ibrahim it was concluded that Fe originated from geological sources whereas Zn, Cu, Pb and Cd were anthropogenic in origin. Table 2 provides the data for CaCO<sub>3</sub>, Fe and Mn contents for each site.

Table 2. Non-pollutant metal content in sediment and soil

Site	1	2	3	4	5	Mean	Shale <sup>‡</sup> )	Soil World Average value <sup>§</sup> )
Fe-SO <sup>•</sup> g/kg	16.96	22.38	30.94	17.22	49.28	27.36	47.25	32.00
Fe-SE <sup>†</sup> g/kg	18.34	20.00	21.00	20.85	30.50	22.14		
Ratio SO/SE	0.92	1.12	1.47	0.83	1.62	1.19		
Fe-SO as % Fe <sub>2</sub> O <sub>3</sub>	2.42	3.20	4.42	2.46	7.1	3.91	6.75	4.58
Fe-SE as %Fe <sub>2</sub> O <sub>3</sub>	2.62	2.86	2.99	2.98	4.36	3.17		
Mn-SO mg/kg	120	79	387	250	535	274	850	761
Mn-SE mg/kg	90	83	172	217	290	170		
Ratio SO/SE	1.33	0.95	2.25	1.15	1.84	1.61		
% CaCO <sub>3</sub> - SO	16.7	38.3	30.6	39.7	15.1	28.1		
%CaCO <sub>3</sub> -SE	23.9	45.3	41.6	47.8	25.9	36.9		
Ratio SO/SE	0.7	0.92	0.74	0.83	0.58	0.76		

<sup>•</sup> SO-Soil

<sup>†</sup> SE-Sediment

<sup>‡</sup> Shale value adapted from Li (2000)

<sup>§</sup> Soils world's average values form Ure and Berrow (1982)

### 4.2.1 Calcium carbonate

The values for %CaCO<sub>3</sub> (strictly, CaCO<sub>3</sub> equivalent, since other carbonates, especially MgCO<sub>3</sub>, may contribute to the neutralizing value of soils and sediments) range

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from 23.9 – 47.8% (mean = 36.9%) for bed sediments and for soils 15.1 – 39.7% (mean = 28.1%) and the sediment/soil ratio is (mean) 1.4.

A visual inspection of the Nahr-Ibrahim channel bed at the five sites suggested a dominance of limestone clasts. However, although limestones constitute a substantial proportion of the bedrock in the Mediterranean Basin they yield very little sediment (Rendell, 1997). This is consistent with the prevailing theory of landscape evolution in karst terranes, that development is dominated by limestone solution not comminution. The excess of  $\text{CaCO}_3$  in the sediments compared with adjacent soils can be explained by carbonates precipitating from the Ca-saturated water column.

#### 4.2.2 Iron

It was noted earlier that the red-brown color of the river when in spate is generally ascribed to suspended sediment derived from erosion of the Red Mediterranean soils widespread in the drainage basin. From a selective sequential extraction investigation of Nahr Ibrahim sediments from the previous (1996) dry season sampling it was shown that Fe occurred largely in the residual fraction (73.5%) with little (3.2%) in the combined exchangeable and carbonate fractions (Korfali and Davies, 2000).

Darwish and Zrayk (1997) reported goethite ( $\alpha\text{-FeOOH}$ ) as the dominant Fe mineral in Lebanese soils. The solubility ( $\text{FeOOH} + 3\text{H}^+ = \text{Fe}^{3+} + 2\text{H}_2\text{O}$ ) of this mineral at pH = 8 is  $<10^{-24}\text{M}$  (Lindsay, 1979). Iron will therefore not dissolve or become mobile when eroded soil enters the river. There is a possibility that low redox (Eh) potentials in the bed sediment might cause ferric iron to reduce to ferrous and therefore become mobile. However, at the prevailing pH of the river water it is unlikely that sufficiently low Eh values would be encountered for  $\text{Fe}^{2+}$  to be a stable species (see Garrels and Christ, 1965, Figure 7.8b). Immobility and preponderance in the residual fraction therefore make Fe a good reference element for evaluating a soil contribution to bed sediments in karst terranes.

The observed prevalence of limestone clasts in the river bed nonetheless raises a possibility that the fine sediment analyzed in this work is dominantly limestone. Abdel-Rahman and Nader (2002) have published multielement data for the rocks of the Nahr-Ibrahim gorge where the Sannine Formation of the Middle Cretaceous is dominant in the westerly part of the valley. The average value of Fe in Sannine limestone formation is 3.65 g/kg. Our mean value in sediments is 22.14 g Fe/kg (range 18.34 – 30.5) i.e. a factor of 6.1 greater. Whatever the source of the  $\text{CaCO}_3$  in the sediment, a dominantly limestone origin does not account for the observed Fe content.

It is conventional to report Fe concentrations in soil and rock as % oxide ( $\text{FeO}$  or  $\text{Fe}_2\text{O}_3$ ). The mean sediment  $\text{Fe}_2\text{O}_3$  content is 3.17% (range 2.62 – 4.36%). Förstner and Wittmann (1979) proposed that river sediments are best compared with reference shale compositions as a quick and practical means of assessing metal enrichment or depletion. One widely used compilation is the North American Shale Composite (NASC) where the

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mean is 6.29%  $\text{Fe}_2\text{O}_3$  (Gromet et al., 1984). Li (2000) has reported a standard shale value for Fe as 6.75%  $\text{Fe}_2\text{O}_3$ . Taking a mean 6.5%  $\text{Fe}_2\text{O}_3$  then the sediment values range from 40.3 – 67.1% (mean 48.8%) of a shale standard. When the mean Fe value is adjusted to allow for the  $\text{CaCO}_3$  component (36.9%) the adjusted mean is 6.91%  $\text{Fe}_2\text{O}_3$ . These analyses and calculations support the hypothesis that the fine bed sediment is derived from eroded soil.

For the soils the range is 2.4 – 7.1%  $\text{Fe}_2\text{O}_3$  (mean 3.91%), values which are lower than the range of 6.5 – 12.1 % $\text{Fe}_2\text{O}_3$  reported by Darwish and Zurayk for lower altitude soils in Lebanon but comparable with the world mean of 4.58% (Ure and Berrow, 1982; Table 2).

Figure 2A is a scattergram of Fe in soil and Fe in sediment. In order to decrease the positive skewness of the two sets of values the data have been transformed to their common logarithm equivalents. There is a good relationship between the two sets and 78.5% of the variance of the sediment values can be ‘explained’ by the soil values. The mean ratio for Fe-soil /Fe-sediment is 1.2 and the regression line intercept is 6.3%  $\text{CaCO}_3$  in sediment. This excess of calcium carbonate in the sediment can be explained by precipitation from the water column.

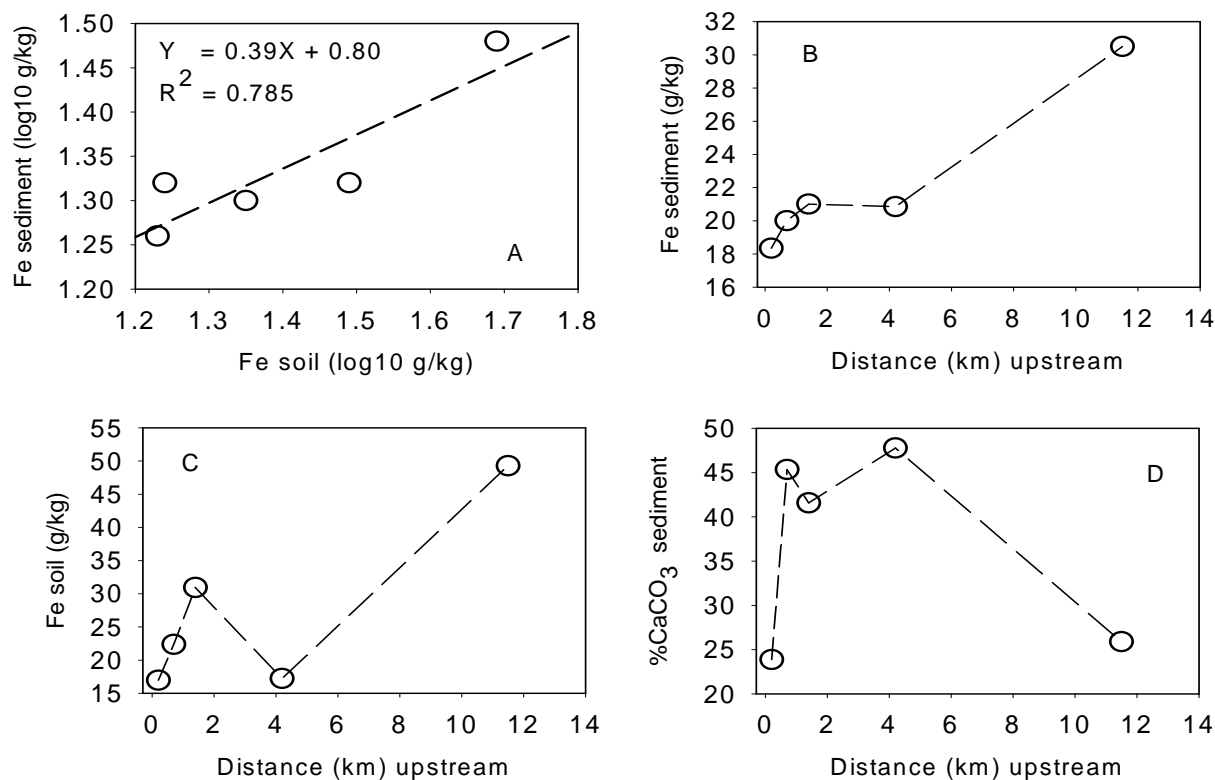


Figure 2. Variation of Fe in sediment and soil and sediment relation to carbonate

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Since the adjusted sediment Fe concentrations are close to those for a standard shale rather than a limestone and since there is a strong relationship between Fe in soil and Fe in sediment it is concluded that the fine bed sediment is most likely derived principally from eroded soil material.

In Figure 2 B, C and D sediment Fe, soil Fe and sediment  $\text{CaCO}_3$  are plotted against distance (km) upstream from the river mouth. In each plot the value for site 5 at 11.5 km appears to be an outlier: Fe contents are relatively high and  $\%\text{CaCO}_3$  relatively low. This may reflect the influence of outcrops of basalt of the Bhanes Formation (Jurassic) but geochemical data are not available for these rocks.

#### 4.2.3 Manganese

Manganese in the sediments ranges from 83.0 – 290 (mean = 170.5) mg/kg. Adjusting the mean value to allow for the calcium carbonate content of the sediment yields a new mean of 270.3 mg/kg. Comparative local rock data are not available. Li (2000) reports a reference shale value as 850 mg Mn/kg whereas the NASC value is 465 mg Mn/kg. The adjusted mean value is only 58% of the NASC reference value. Soil contents are higher than for sediment: mean = 274 mg Mn/kg and range 79 – 534.7 mg/kg. According to Ure and Berrow (1982) the world soil average for Mn is 761 mg/kg. Until data are available for Mn in soils and rocks of the catchment the lower values compared with reference materials cannot be explained. However, Figure 3A shows a strong relationship between the soil and sediment Mn concentrations and soils of high Fe content exhibited a strong relationship with Mn content in soils (Figure 3B); and provisionally, it is concluded that Mn derives from eroded soils

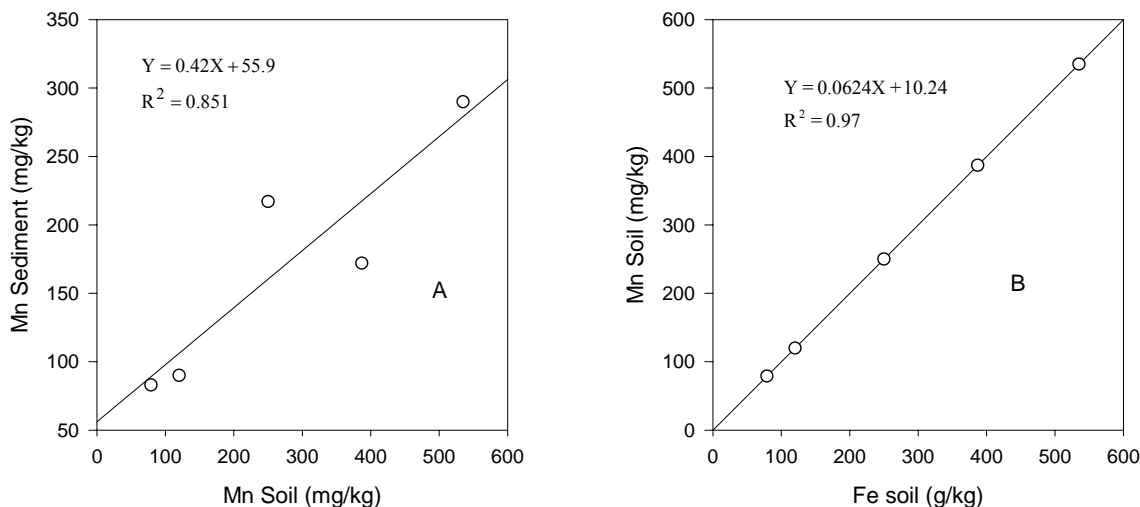


Figure 3. Mn relation in sediment and soil

### 4.3 Pollutant metals in sediment and soil

The metals Cd, Cr, Cu, Ni, Pb and Zn are all classed as potentially toxic metals and excesses in soils and sediments often arise through pollution. Korfali and Davies (2000, 2003) concluded that Cu, Pb and Zn in the sediments of Nahr Ibrahim were anthropogenic in origin and their deposition in the sediments was primarily regulated by the carbonate sediment fraction. The metal data (Table 3) are interpreted in the light of the many pollution sources and in line with the relationship between metal content in sediment and soil.

Table 3. Pollutant metal content in sediment and soil

Site	1	2	3	4	5	Mean	Shale ‡	Soil World Average value §
Zn-SO • mg/kg	161	225	110	57	126	136	95	59.8
Zn-SE † mg/kg	110	137	177	75	293	147		
Ratio SO/SE	1.46	1.64	0.62	0.77	0.53	0.92		
Cu-SO mg/kg	49	80	46	28	62	53	45	25.8
Cu-SE mg/kg	88	75	316	111	396	197		
Ratio SO/SE	0.56	1.08	0.15	0.26	0.16	0.27		
Pb-SO mg/kg	276	1080	22	55	52	298	20	29.2
Pb-SE mg/kg	138	172	59	25	62	91		
Ratio SO/SE	2.01	6.31	0.39	2.20	0.84	3.27		
Cr-SO mg/kg	88	78	204	66	106	108	90	84
Cr-SE mg/kg	82	70	79	47	148	86		
Ratio SO/SE	1.09	1.11	2.58	1.40	0.72	1.26		
Cd-SO mg/kg	1.83	2.10	4.50	1.49	1.60	2.30	0.3	0.62
Cd-SE mg/kg	1.49	1.49	1.84	0.81	2.14	1.54		
Ratio SO/SE	1.21	1.41	2.44	1.84	0.75	1.50		
Ni-SO mg/kg	50	36	94	46	116	69	50	33.7
Ni-SE mg/kg	66	56	60	36	101	64		
Ratio SO/SE	0.77	0.66	1.58	1.28	1.15	1.1		
%OC ‖-SO	7.94	23.57	4.15	0.63	5.72	8.40		
%OC-SE	5.17	2.80	1.21	1.87	0.74	2.36		
Ratio SO/SE	1.54	8.42	3.43	0.34	7.72	3.56		

• SO-Soil † SE-Sediment ‡ Shale value adapted from Li ( 2000)

§ Soils world's average values from Ure and Berrow (1982)

‖ OC-Organic carbon

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It is unlikely to have a close relationship between soil and sediment composition at each site, since sediments include material from upstream (we do not know how much and how far). This explains the insignificant statistical correlation of pollutant metals between their content in sediment and flood plain soil (Table 4). Conversely, the paired T-test (Table 4) supports the similarity between bed sediments and catchment soils ( $P > 0.05$ ), which elucidate that catchment sediments are derived from soils erosion. But, when pollutant metal content in sediments and their content in floodplain soils with their special variation were compared, similarities and differences occurred with respect to higher site content and site properties. The metal content variation, relationship of metal content in sediment and soil paralleled with pathway of pollutant metal discharge were all able to anticipate pollutant metals into three groupings.

Table 4. Statistical relation of pollutant metal content in soil and sediment (Pared T-test and Pearson correlation coefficient)

Element	T-paired Test Sig. (P)	Correlation Coefficient (r)
Zn-SO	0.642	0.078
Zn-SE		
Cu-SO	0.097	0.022
Cu-SE		
Pb-SO	0.312	0.856
Pb-SE		
Cd-SO	0.159	0.699
Cd-SE		
Cr-SO	0.330	0.337
CR-SE		
Ni-SO	0.672	0.765
Ni-SE		

#### 4.3.1 Lead, Zinc, Copper

The mean value of Pb in sediment is 91 mg/kg (range 24.7 – 172). For the soils the mean = 297 mg Pb/kg and a range of 22.7 – 1080 mg/kg. Li (2000) records a reference shale value as 20 mg/kg. For soils (Ure and Berrow, 1982) the world mean is 29.2 mg Pb/kg. The mean value of Pb in sediment is 4.6 times higher than the standard shale content; and in soil is 10.2 times more than world's average soil Pb content. Overall, both soils and sediments show evidence for anthropogenic contamination.

For both soil and sediment Pb contents are higher in sites 1 and 2 near the river mouth. Here the river flows through an urban area and under the main coastal road from

Beirut to Byblos: heavy traffic has presumably caused high Pb deposition from vehicle exhaust fumes since leaded petrol is still used in Lebanon

The mean value of Zn in sediment is 147 mg Zn/kg that is 1.5 times higher than shale reference value (Table 3). In soils, the mean content is 136 mg Zn/ kg, which is 2.3 times higher than world soil average. Sediment Zn values increase up to site 3, drop for the quarry site (4) and then rise to a maximum value at site 5. High values at sites 1 and 2 can be ascribed to possible effluents from a galvanizing works and a wire manufacturer. The observed industrial effluent coursing down the limestone cliff marking the floodplain limit explains the increase at site 3. For the soils, sites 1 and 2 are high also and attributable to the same Zn sources yet soil Zn is lower at site 3. Both soils and sediments demonstrate an elevation at site 5 attributable to the many potential polluting sources at or around the town of Yahschouch.

The average Cu content (53 mg/kg) in soil is twice world's average soil value. While in sediments the average Cu content (197 mg/kg) is 4.4 times the shale reference value (Table 3). Soil Cu varies little with distance. Similar to Pb and Zn, the lowest concentration is at site 4 (Quarry). Elevated levels in sediments are at site 3 and 5, and these are attributable to industrial effluents at these sites (Fig. 1).

Figure 4 depicts the ratio of soil to sediment of the studied parameters with distance from river mouth (site 1, 0.2 km and site 5, 11.5 km), viewing the five sampling sites. For both Zn and Pb as river opens to urban coastal floodplain, the soil content is higher than sediments. Except at site 2, the soil Cu was always less than sediment (Table 3, Fig. 4). A strong relationship occurs between Zn, Pb and Cu soil content and organic soil content (Fig. 5). Hence, the soil organic content appears to regulate their geochemistry and their accumulation in soils. This observation could explain the high content of Zn, Pb and Cu in soil at site 2 that has the highest organic content (23.57%). The characteristic localization of Pb near the soil surface in most profiles is related to the surface accumulation of organic matter and it is considered as an important sink of Pb in soils (Kabata-Penduis, 2000). The lack of a relationship in sediment between Zn, Cu and Pb content and organic content could be due to the prevailing low organic content (range only 0.74%-5.7%).

A plausible justification to enrichment downstream in soil and upstream in sediment of Zn, Pb and Cu is due to the pathway through which these metals are discharged (whether to soils or direct to river) and additional is site soil property previously discussed. The Lebanese Ministry of Environment has recently set rules and recommended policies to preserve the surface water; of these is the prohibition of waste discharge (domestic and industrial) into rivers (ECODIT, 2002). However, in inland zones, wastes are dumped into the river channel (Jurdi, 1998, Jurdi et. al, 2002). The high Zn and Cu concentration in sediment at site 5 where ratio of soil to sediment is low (Table 3, Fig. 4) is mostly attributable to the metal rich effluents from the specified industries at this site direct to the river. Mostly, the precipitating carbonate species and depositing in the sediment scavenge these metals, rendering higher amounts in sediment

than soil. But, if polluting metals go first to soil then to river as eroded soils, then metals may be lower in concentration since carbonate coating would dilute and sediments from upstream. This could explain the higher concentration of Zn, Cu and Pb in floodplain soil at site 2, where prohibition of waste discharge into river channel and the waste dumping into floodplain were experiential. Though site 3 is a coastal zone, the concentrations of Zn and Cu in sediment are higher and this is most probably due to the lower organic content in floodplain soil at that site (Table 3). Zinc and Cu entered stream as a runoffs and scavenged with the precipitating carbonate depicting higher concentrations in sediment.

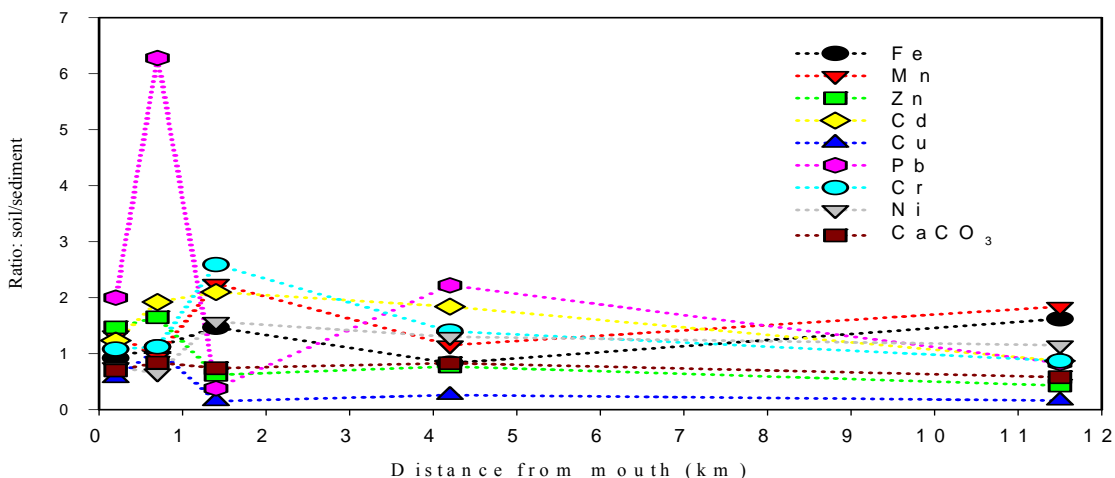


Figure 4. Ratio of metal content in soil and sediment among all sites with distance

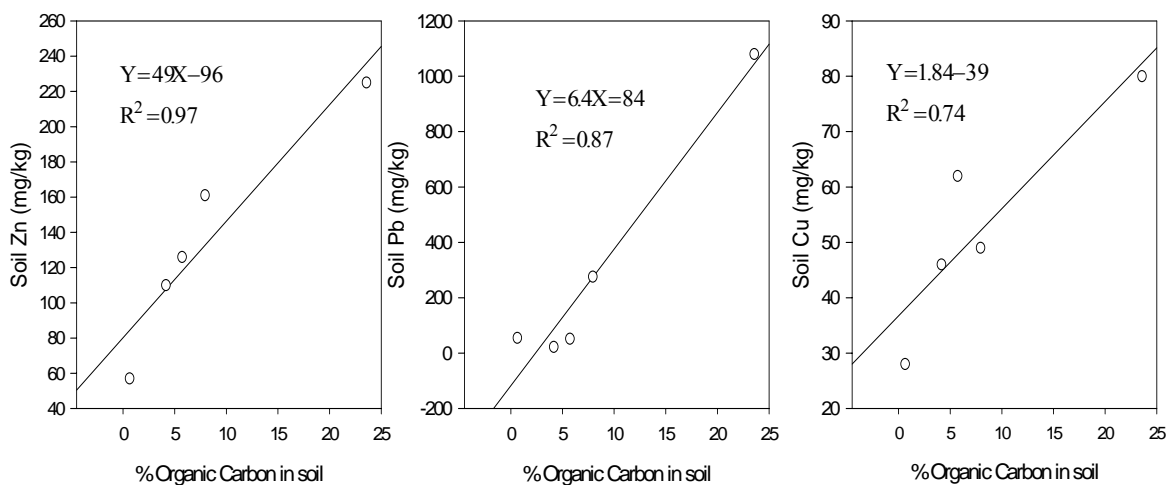


Figure 5. Relationship of Zn, Pb and Cu with organic carbon in soil

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#### 4.3.2 Cadmium and Cr

Cadmium and zinc are closely related geochemically, they are both calcophilic metals and Cd is obtained as a co-product from smelting and refining of Zn from sphalerite. The ratio of Zn to Cd in natural materials is often approximately 100: 1. The world soil Zn/Cd ratio (Table 3) is 96.5 and ratio for reference shale is 316. For the experimental soils Zn/Cd = 61.6 and for sediments = 99.8. These ratios support the suggestion that Zn and Cd in Nahr-Ibrahim derive markedly from different polluting sources and also are supported by the lack of statistical significant correlations between Zn and Cd whether in sediment or soil. While, a statistical significant correlation ( $r=0.88$ ,  $p<0.01$ ) occurred between soil content of Cr and Cd.

The reference shale for Cr is 90 mg/kg. The mean value of 86 mg/kg is comparable with sediments content except at site 5. The reference soil is 84 mg/kg. Most sites were below this level except at site 3 it is anomalous. Pigments containing Cr are widely used and plastic factory at site 3 (Fig. 1) produces colored products that may explain the high values. A source of chromium pollution in soil is the dying industry (Stepnievskis and Bucoir, 2001).

Soil Cd values at each site (mean= 2.3 mg/kg) exceed the world reference value of 0.632 mg/kg. Similarly, the sediment value (mean – 1.54 mg/kg) at each site exceeds the reference shale value of 0.3 mg/kg. The suspected pollution from Yahshouch area – sit 5 (Fig. 1) is seen for sediment Cd but not for soil. Similar to Cr soil Cd at site 3 is anomalous and the effect of the plastic factory, where Cd is used as stabilizer to preserve decomposition of PVC (Stephenson et al., 1998) is projected. In addition, site 3 is an agricultural zone where pesticides and herbicides fertilizers have added into content of Cd in soils. The high content of Cd and Cr in soils could also be due to the high content of Fe at site 3 (Table 2). A stronger relationship is observed for Cd with Fe content in soils than clay and organic, and Fe oxides reveal in soils the highest affinity for adsorption (Kabata-Penduis, 2000). Hursthouse et al. (2001, 2003) have also reported the strong correlation between Fe and Cr content in soil.

Like Zn and Pb as the river opens to urban coastal floodplain, the soil content of Cd and Cr are higher than sediments (Table 3, Fig. 4), and highest ratio of soil to sediment is at site 3. Again a justification for downstream enrichment of Cd and Cr in soils and upstream in sediment is due to pathway through which these metals are discharged and soil site characteristics. It was discussed previously that Fe soil phase, appears to regulate Cr and Cd accumulation in soil. The higher content of Cr and Cd in sediment than soil at site 5 (Table 3, Figure 4) is again due to metal effluents from industries at site 5 (Fig. 1) direct to river. Whereas, at site 3 with higher Cr and Cd in soils than sediments is due to discharge of metal effluents first to soil then to river as eroded soils.



### 4.3.3 Nickel

The reference shale and soil values for Ni are 50 mg/kg and 33.7 mg/kg respectively. The sediment content does not differ markedly from the reference value except at site 5, industrial zone, battery factory and steel works (Figure 1). The mean soil value (69 mg/kg) is approximately double the soil value and site 5 (116 mg/kg) is seen to be anomalous as site 3, the locality influenced from discharges by plastic factory.

Ni diverted from the other discussed pollutant metals, where at site 5 soil Ni content was slightly higher than sediment Ni content. This diverted pattern of Ni could be ascribed to Ni geochemistry. Nickel is siderophilic element and will join Fe whenever such a phase occurs (Kabatta-Pendius, 2000); a higher Fe content could present a higher Ni content. Figure 6 shows a strong relationship between the soil and sediment Ni content and the content of Ni and Fe in soil. It is concluded for the time being that Ni derive from soil erosion in sediment.

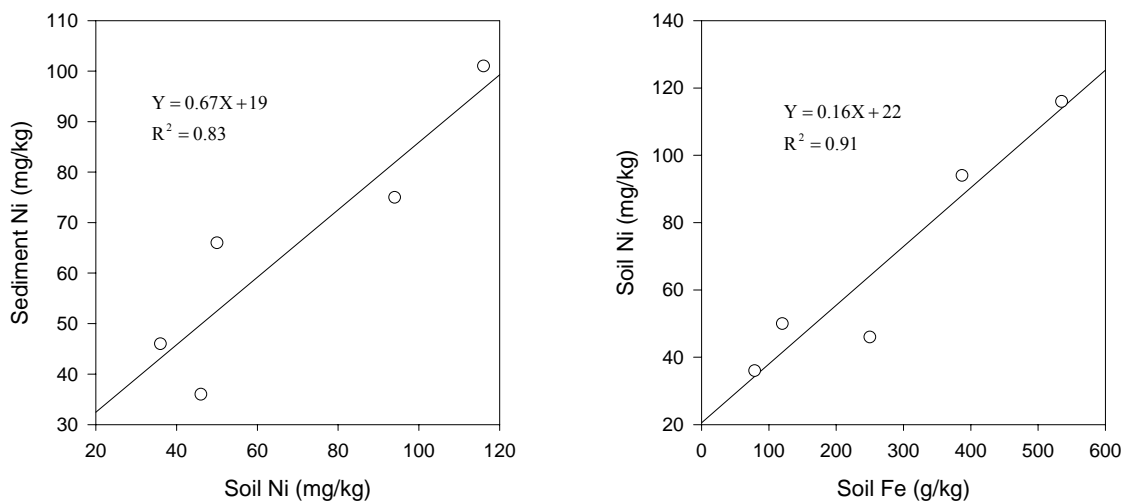


Figure 6. Ni relation in sediment and soil

## 5. CONCLUSION

This study may be considered as a prototype of rivers whose drainage basin is floored by limestone and the water is saturated by calcium carbonate species. As well, in view of the growing recognition of role studies of floodplain soils might make in pollution monitoring and be storage system, the composition ( $\text{CaCO}_3$ , As, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn) of bed sediments and adjacent floodplain soils of Nahr-Ibrahim River (characterized by marked differences in discharge between winter and summer) were compared in this study. Data revealed a higher composition of  $\text{CaCO}_3$  in sediments than in soils, which is explained by carbonate precipitating from the Ca-saturated water column. This saturated carbonate water column acts as a sink through precipitating the pollutant metals in bed sediment. Though, as river opens to floodplain soils the concentrations of polluting metals (Cd, Cr, Cu, Ni, Pb and Zn) are higher in soils than sediment (i.e. these floodplain soils revealed a storage system for future pollution); yet the precipitating Ca-carbonate acted as diluter for metals in bed sediments. These bed sediments are derived from eroded soils during wet season of higher metal content. Whereas, data revealed that upstream in inland site (environmental rules are not strictly abided) concentration of Cd, Cr, Cu and Zn were higher in sediments than in soils. This could be attributed to direct industrial effluents into channel and consequent metal precipitating into bed through carbonate precipitation. Nevertheless, the studied carbonated river basin with its high pH value provide lower de-sorption of metals from bed sediment. This might lead to the ultimate ability of utilization of this river water to cope with Lebanon future shortage of good water quality. Thus, an integrated management plan of this water resource is vital. The plan set should address policy constrains relative to environmental, institutional and financial issues; as well as the development and enforcement of environmental rules and regulations that should be abided by all through river channel (mainly in inland sites). Besides, the whole process of metal cycle among soils/sediment/water necessitates a study of metal concentration in sediment and in soil chemical fractions to draw a better fit for modeling of fluvial pollutant transport and elucidate an improved management plan of this water resource.

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