HEAVY METALS BACKGROUNDS IN SOILS FROM THE ROVIGO PROVINCE (NE, ITALY)

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Abstract

This research is aimed at characterizing the geochemical composition of the alluvial soils in the province of Rovigo in order to identify the provenance of the sediments from which they originated. To carry out this characterization soils were sampled in 5 distinct areas of the province next to the towns of Stienta, Fratta Polesine-Bosaro, Boara Polesine, Concadirame, Lusia. The samples were preliminarily characterized by grain size investigation and then investigated by wavelength dispersion x-ray fluorescence analysis (WDXRF). The data show a considerable compositional variation of the major elements that are strongly influenced by grain size; sandy soils are enriched in SiO_2 in relation to the prevalent presence of quartz, while fine soils are comparatively enriched in Al_2O_3 and K_2O that indicate the prevailing presence of clay minerals. Samples from Stienta are similar to soils of the Ferrara province developed by Po river sediments, whereas gradual differences are observed on soils sampled in the middle part of the province in the neighbors of Fratta Polesine-Bosaro. More marked differences are observed in the northernmost areas close to the towns of Boara Polesine, Concadirame and Lusia. This compositional heterogeneity is due to a different contribution from the two river systems feeding the sedimentary basin, i.e. the Po River and the Adige river. Although not systematically, high values in Cr and Ni that characterize the floods of the river Po (Amorosi et al. 2002, Amorosi, 2012; Bianchini et al. 2012; 2013) are also recognized in the northern areas of the province. In the northern soils we also observed comparative enrichment in Pb and Zn. Our preliminary hypothesis proposes that this enrichment in chalcophile elements is a proxy of the sedimentary contribution of Adige river, which is characterized by a drainage basin including mining zones in which sphalerite and galena were extracted in the past centuries.

Keywords: *heavy metals, alluvial soils, sediment provenance, geochemical back-grounds*

Introduction

The understanding of sedimentary dynamics in alluvial plains is helped by the use of geochemical tracers that represent powerful tools for the identification of source areas, pathways that link sediment sources and sinks, and general sediment-system behaviour. The understanding of these processes is of fundamental importance for

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a correct definition of geochemical backgrounds, which are site-specific. In fact, the definition of a "baseline" is necessary to distinguish the natural background from anthropogenic concentrations of Potentially Toxic Elements (PTE) such as heavy metals, and is important to develop guidelines for environmental legislation. Due to the fact that the background concentrations strongly depend on geological characteristics such as mineral composition, grain size distribution and organic matter content, there is the need to collect data and to create exhaustive data sets.

The application of geochemistry for the characterization of soils developed on alluvial sediments of the Padanian Plain of northern Italy has been extensively carried out in the Emilia-Romagna Region, especially in the Province of Ferrara (Amorosi et al., 2002; Amorosi 2012; Bianchini et al., 2012; 2013; Di Giuseppe et al., 2014a; 2014b) where hundreds of soil samples have been analysed by X-Ray fluorescence, whereas similar investigations are scarce in the neighbouring province of Rovigo (Veneto region), which is located between Po and Adige rivers (Fig. 1). This contribution is therefore aimed to carry out, for the first time, a systematic geochemical study of this area in order to define the origin of the sediments and their specific backgrounds of PTE such us heavy metals.

Sampling strategies and analytical methods

Soil sampling have been performed in 5 distinct areas of the Rovigo province (Fig. 1) in order to collect representative samples of the various sedimentary facies.

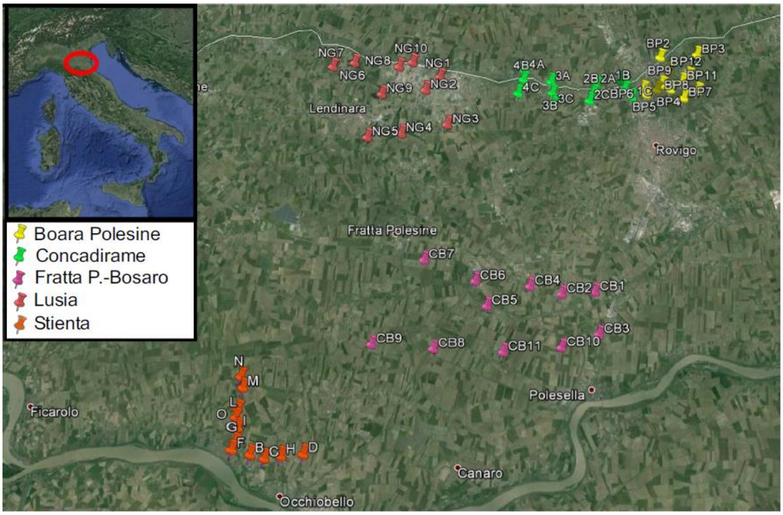

Figure 1. *Perspective view of the sampling area located in the Province of Rovigo between the Adige and Po river streampaths.*

Taking into consideration that the depth of tillage involving soil digging, stirring, and overturning is usually ca 50 cm, according to the procedure delineated by Facchinelli et al. (2001) and Ungaro et al. (2008), two samples were collected by an Eijkelkamp hand auger (Fig. 2a) at each selected site: one representative of the plough horizon (just beneath the roots zone, at a depth of 20 cm) and the other representative of the underlying undisturbed layer (at a depth of 100 cm). In other words, the shallower samples are considered representative of the soil portion interested by agricultural activity, while the deepest samples are interpreted as representative of horizons unaffected by anthropogenic activity. The comparison allows the evaluation of the magnitude of the possible Top Enrichment Factor (TEF) induced by anthropogenic contributions.

Samples were air-dried and divided into distinct aliquots for grain-size and chemical/mineralogical investigations. The grain size determination was obtained, following the notional classification of Wentworth as described by Salemi et al. (2010), by sieving the sandy fraction from the fine (<63 μm) one, and then using wet gravitational separation in deionised water to divide the clay from the silt fraction by a "Micrometrics Sedigraph 5100".

For the geochemical analysis, sediments were finally powdered in agate mill (Fig. 2c) and an amount of about 4 g of powder were pressed with addition of boric acid by hydraulic press to obtain powder pellets (Fig. 2d). Simultaneously, 0.5–0.6 g of powder was heated for about 12 h in a furnace at 1000 °C in order to determine the loss on ignition (Fig. 2e).

This parameter measures the concentration of volatile species contained in the sample. The Wave length dispersive X-ray Spectrometry (WDXRF) analysis of the powder pellets was carried out using an ARL Advant-XP spectrometer Thermo Scientific (Waltham, MA, USA, Fig. 2f). Calibrations were obtained analysing certified reference materials, and matrix correction was performed according to the method proposed by Trail and Lachance (1966). Precision and accuracy calculated by repeated analyses of international standards (with matrices comparable with the studied samples, Di Giuseppe et al., 2014b) were generally better than 3% for Si, Ti, Fe, Ca and K, and 7% for Mg, Al, Mn and Na. For trace elements (above 10 ppm), the errors were generally better than 10%. In order to assure the quality of the analyses, the described WDXRF system has been involved in an intercalibration project on the analysis of soils and sediments, that confirmed the reliability of the presented results (Vittori Antisari et al., 2014).

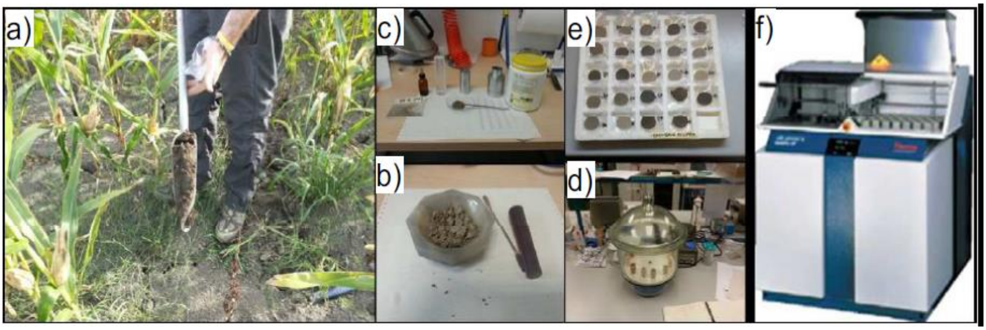

Figure 2. *Sample collection, preparation and analysis of the investigated soil samples. Soil cores sampling procedure (a), powdering in agate mill (b), preparation of boric acid supported powder pellets for WDXRF analysis (c, e, f) and LOI determination (d).*

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Results and discussion

The obtained results show the following compositional ranges for the main oxides constituting the studied soils:

72.79	≤	SiO_2%	≤	35.70
17.24	≤	Al_2O_3%	≤	8.40
11.56	≤	CaO%	≤	1.09
3.50	≤	K_2O%	≤	1.97
10.56	≤	Fe_2O_3%	≤	2.95
6.51	≤	MgO%	≤	2.14

These major elements are strongly controlled by the grain size, and SiO_2 become preponderant in the sandy fraction thus suggesting the prevalent presence of quartz; on the contrary Al_2O_3 an K_2O increase in the fine fractions to mean the prevalent presence of clay minerals.

As concerns the trace elements, the values of the TEF, determined by the ratio between the concentrations of heavy metals in the superficial samples and those recorded at depths of 100 cm, are nearly equal to 1 for most elements at the majority of sampling sites (Table1).

Only few sites display some superficial lead/zinc enrichments, possibly reflecting anthropogenic activities (accumulation of particles related to the use of fossil fuels?). Other sporadic enrichments in surface horizons are observed for copper, possibly in relation to the use of chemical products in agricultural practices.

A general, PTE distribution in the investigated samples is reported in Table 2 and in the box-plots of Fig. 3, that allows to compare medians, quartiles and extreme values in sediments from the distinct zones of the Rovigo province.

Particular emphasis has given to Cr, Ni, Co that are elements used as proxies of the Po river sediments (Amorosi et al., 2002; Amorosi; 2012; Bianchini et al., 2012; 2013; Di Giuseppe et al., 2014a; 2014b). In fact, Po river sediment are characterized by high values of Cr, Ni, Co in relation to ophiolite rocks outcropping in its hydrological basin (Amorosi, 2012; Bianchini et al., 2013). The results show that very high concentration of these metals characterize the zone of Stienta, where compositions are analogous to those recorded of the province of Ferrara (Bianchini et al. 2012, 2013).

In the middle part of the province of Rovigo the Po river fingerprint is less marked (e.g.: samples from Fratta Polesine-Bosaro). The influence of Po river become less significant and progressively vanish in the sites of Boara Polesine, Concadirame, Lusia. In fact, in this part of the province high values of Cr, Ni, Co become sporadic, possibly due to episodic floods of Po river that transported fine Cr, Co, Ni rich particles also in this part of the province. On the contrary, sediments from Boara Polesine, Concadirame and Lusia are characterized by comparative enrichment in chalcophile elements such us (Pb, Zn, Cu) that could be interpreted as the specific fingerprint of the Adige River sediments.

Table 1. *Heavy metals Top Enrichment Factors of soil samples from Boara Polesine* (a), *Concadirame* (b), *Lusia* (c), *Fratta P.-Bosaro* (d) *and Stienta* (e). *TEF values higher than 1.5 (highlighted) are often observed for Cu and Zn, possibly in relation to anthropogenic activities. Two sites from Boara Polesine and one from Stienta display anomalous TEF values for most elements and require further investigations.*

(a)

TEF	BP1	BP2	BP3	BP4	BP5	BP6	BP7	BP8	BP9	BP10	BP11	BP12
Cr	1.0	1.1	1.4	0.6	0.7	0.7	0.8	1.1	1.0	1.3	1.8	1.0
Ni	1.1	1.0	2.0	0.5	0.7	0.7	0.8	1.0	0.9	2.2	1.8	0.6
Co	1.1	1.0	1.2	0.8	0.9	1.0	0.9	0.9	1.0	3.2	1.4	0.9
Pb	1.2	1.1	1.6	2.1	1.9	1.2	1.2	0.8	1.2	1.7	1.3	1.6
Cu	2.8	1.1	2.6	1.3	1.2	0.6	0.9	0.9	1.1	1.8	2.8	2.3
Zn	1.7	1.0	3.6	1.2	1.2	0.9	0.9	0.9	1.1	2.8	1.5	1.0

(b)

TEF	P1A	P1B	P1C	P2A	P2B	P2C	P3A	P3B	P3C	P4A	P4B	P4C	P5A	P5B	P5C
Cr	0.5	0.9	1.1	0.3	1.0	0.9	1.2	1.0	1.2	0.9	1.1	1.0	0.9	1.0	0.9
Ni	0.1	1.0	1.2	0.0	1.1	0.7	1.5	1.1	1.1	0.9	1.2	1.0	0.9	1.2	0.9
Co	0.0	1.1	0.9	0.0	1.0	0.7	1.2	1.2	1.3	0.9	1.1	1.0	0.9	1.3	1.0
Pb	0.3	1.4	1.5	0.2	1.1	0.7	1.2	1.1	1.3	1.1	1.0	1.0	0.9	1.3	1.0
Cu	0.1	1.4	1.4	0.1	1.1	1.4	1.2	1.0	2.1	1.0	1.3	0.8	1.0	1.4	0.9
Zn	0.3	1.6	2.1	0.3	1.0	0.9	1.9	1.1	2.5	1.1	1.5	1.2	0.9	1.8	1.2

(c)

TEF	NG 1	NG 2	NG 3	NG 4	NG 5	NG 6	NG 7	NG 8	NG 9	NG 10
Cr	1.6	1.4	0.9	1.1	1.0	1.0	1.0	0.7	1.0	1.0
Ni	1.1	1.2	0.8	1.3	0.9	1.0	1.1	0.9	1.0	1.2
Co	1.1	1.1	1.0	1.1	0.9	1.0	0.9	1.0	1.1	1.1
Pb	1.0	1.2	1.0	1.3	0.9	1.0	1.0	1.1	0.9	0.9
Cu	1.3	2.4	1.0	1.4	0.8	1.0	1.0	1.1	1.0	1.0
Zn	1.1	1.4	1.0	1.3	1.0	1.0	0.9	1.0	1.4	1.0

(d)

TEF	CB1	CB2	CB3	CB4	CB5	CB6	CB7	CB8	CB9	CB10	CB11
Cr	1.3	1.1	1.1	1.1	1.1	0.9	1.0	1.1	1.1	1.0	1.0
Ni	1.4	1.1	1.1	1.1	1.1	0.8	1.2	1.1	1.2	1.0	1.1
Co	1.1	1.2	1.0	0.9	1.2	1.0	1.1	1.2	1.1	1.0	0.9
Pb	1.0	0.9	0.5	1.7	1.0	1.3	0.9	1.1	1.2	1.1	0.8
Cu	1.4	0.9	0.5	1.2	1.0	0.9	1.3	1.1	1.3	1.2	1.1
Zn	1.2	0.9	0.7	1.4	1.0	1.0	1.0	1.3	2.0	1.1	1.5

(e)

TEF	A	B	C	D	E	F	G	H	I	L	M	N	O
Cr	1.1	1.1	0.9	1.0	1.0	1.0	1.0	1.1	1.0	1.0	1.1	1.6	1.1
Ni	1.3	1.0	0.9	1.2	1.2	1.1	1.1	1.2	0.9	0.9	1.3	1.8	1.1
Co	1.0	1.2	1.0	0.9	0.9	1.1	1.2	1.2	0.9	1.1	1.5	3.1	1.1
Pb	1.1	1.0	1.2	1.4	1.9	1.3	1.2	1.2	0.9	1.1	1.2	1.8	1.1
Cu	2.8	1.0	1.5	1.9	1.5	1.4	1.2	1.3	1.1	1.3	0.7	2.9	0.9
Zn	1.8	1.1	1.1	1.3	1.5	1.4	1.4	1.5	1.3	4.0	2.7	5.3	1.5

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Table 2. *Statistical parameters of the heavy metals distribution in soil samples from the Province of Rovigo.*

		Boara P.	Concadirame	Lusia	Fratta P. - Bosaro	Stienta
Cr (mg/kg)	Min	52	40	35	74	108
	Mean	107	93	69	157	192
	Median	128	75	73	178	200
	Max	216	237	96	217	237
Ni (mg/kg)	Min	24	23	19	45	45
	Mean	94	57	38	118	144
	Median	98	42	39	133	143
	Max	171	168	62	181	202
Co (mg/kg)	Min	14	13	10	11	12
	Mean	24	18	17	19	24
	Median	24	17	18	19	25
	Max	33	32	24	25	29
Pb (mg/kg)	Min	23	21	30	17	11
	Mean	57	51	50	36	26
	Median	53	49	47	28	24
	Max	91	77	84	88	65
Zn (mg/kg)	Min	52	51	66	69	16
	Mean	137	105	98	92	90
	Median	131	96	97	88	84
	Max	318	222	153	136	251
Cu (mg/kg)	Min	15	17	24	26	10
	Mean	64	37	50	44	46
	Median	59	38	40	40	46
	Max	95	59	144	100	96

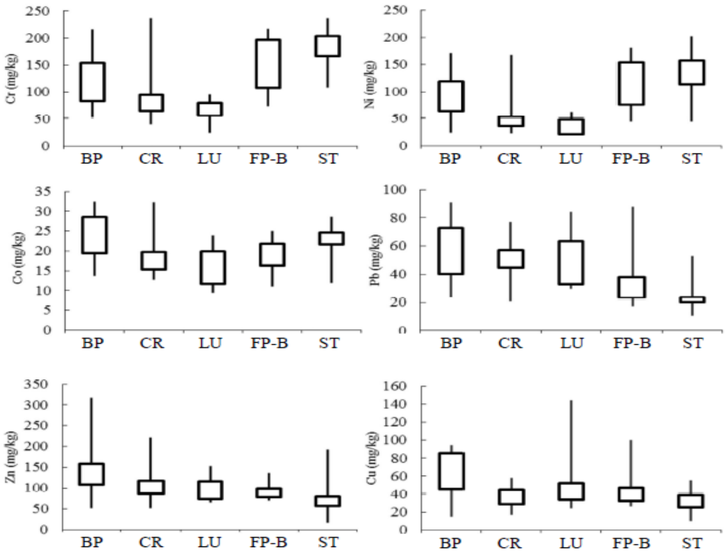

Figure 3
Box-plots of Cr, Ni, Co, Pb, Zn and Cu distribution in soil samples from the Rovigo province.

BP = Boara Pisani, CR =Concadirame, LU = Lusia, FP-B = Fratta Polesine-Bosaro, ST = Stienta.

In this case the enrichment in chalcophile elements could be related to particular rocks and ore deposits outcropping in the Adige catchment (Bianchi and Di Colbertaldo, 1956; Di Colbertaldo, 1965; Murara, 1966; Dessau and Perna, 1966; Brondi et al., 1970; Dessau and Duchi, 1970; Brigo, 1971; Brusca and Perna, 1997; Frizzo, 2004). In fact, although mining activities are not active any more, ore deposits including sulphides have been widely exploited in the Trentino region during historical times for H_2SO_4 production and for Cu, Pb, Zn, Ag, barite and fluorite.

Conclusion

Flood plains are nested hierarchical systems with graded structures that are intimately inter-layered, where geochemical fingerprints reflect composite processes at various scale. Their understanding needs the decomposition of large systems into smaller units, which requires very detailed investigation. In spite of the complexity, the geochemical fingerprint of the sediments in the Province of Rovigo clearly reflects the affinity of the solid load transported by the two main riverine systems that envelope the area, i.e. Po river having sediments characterized by notable concentration of Ni and Cr, and Adige river having sediments comparatively enriched in chalcophile elements such as Pb and Zn. Nevertheless, the obtained concentrations, expressed as median and quartile values provide the current backgrounds of heavy metals in the area that will be useful for future investigation to discriminate geogenic and or anthropogenic contribution to the pedosphere. Obviously, the presented data express the total geochemical budget, and therefore additional analyses will be necessary to discern the labile (i.e. scarcely sequestrated by minerals and mobile) fractions that are bio-available and can be bio-accumulated and transferred to plants and biota, and finally translocated to the human food chain.

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TENORI DI FONDO DEI METALLI PESANTI NEI SUOLI DELLA PROVINCIA DI ROVIGO

Riassunto

La presente ricerca nasce dall'esigenza di caratterizzare la composizione geochimica dei terreni alluvionali nella Provincia di Rovigo per determinare la provenienza dei sedimenti da cui hanno avuto origine. Si sottolinea che, al contrario della Provincia di Ferrara, nella quale centinaia di analisi XRF sono state effettuate sui terreni alluvionali, in Provincia di Rovigo non sono disponibili in letteratura studi analoghi. A tal fine sono stati campionati terreni in 5 distinte zone della Provincia prossime ai paesi di: Stienta, Fratta Polesine-Bosaro, Boara Polesine, Concadirame, Lusia. I relativi campioni sono stati in primis caratterizzati dal punto di vista granulometrico e, dopo essere stati polverizzati con un mulino ad agata, sono stati sottoposti all'analisi in fluorescenza a raggi X per la determinazione degli elementi maggiori ed in tracce. I risultati mostrano una notevole variazione composizionale degli elementi maggiori che sono fortemente influenzati dalla granulometria; terreni sabbiosi risultano essere arricchiti in SiO_2 a sottolineare la prevalente presenza di quarzo, mentre i terreni fini sono comparativamente arricchiti in Al_2O_3 e K_2O che indicano la prevalente presenza di minerali argillosi. In questo contributo, maggiore enfasi viene data però agli elementi in traccia ed in particolare ai metalli di transizione in quanto se presenti in quantità rilevanti possono risultare potenzialmente tossici. I risultati vengono riportati sulla base di una trattazione statistica semplificata per la quale sono stati definiti: valori massimi, minimi e medi, mediane e quartili delle popolazioni di sedimenti relativi alle 5 distinte zone della Provincia. Tali parametri statistici sono stati funzionali per la creazione di grafici "box-plot". Si sono osservate sistematiche differenze ed i campioni della zona di Stienta sembrano essere del tutto analoghi ad alcuni terreni dell'areale ferrarese originati dai sedimenti del fiume Po, mentre graduali differenze si sono osservate sui terreni della fascia mediana della Provincia (Fratta Polesine-Bosaro). Differenze ancor più marcate sono state osservate nelle zone più settentrionali corrispondenti alle zone di Boara Polesine, Concadirame e Lusia. Questa eterogeneità composizionale è da imputare ad un differente contributo relativo dai due sistemi fluviali che alimentano il bacino sedimentario polesano, rispettivamente il sistema fluviale del fiume Po e quello del fiume Adige. In particolare, gli alti valori in Cr e Ni che caratterizzano le alluvioni del fiume Po sono riconosciuti anche nelle zone settentrionali della provincia, ma in modo meno sistematico. In tali zone si nota un arricchimento comparativo in Pb e Zn. Come ipotesi preliminare proponiamo che questo arricchimento in elementi calcofili sia un indicatore tipico delle alluvioni del fiume Adige che è caratterizzato da un bacino idrografico nel quale insistono zone minerarie nelle quali blenda e galena sono state estratte nei secoli scorsi.

Parole chiave: *metalli pesanti, suoli alluvionali, provenienza sedimenti, tenori di fondo geochimico*

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