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Mercury baseline levels in Flemish soils (Belgium)

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Capsule: Increased mercury levels may reflect human activity

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Abstract

It is important to establish contaminant levels that are normally present in soils to provide baseline data for pollution studies. Mercury is a toxic element of concern. This study was aimed at assessing baseline mercury levels in soils in Flanders. In a previous study, mercury contents in soils in Oost-Vlaanderen were found to be significantly above levels reported elsewhere. For the current study, observations were extended over two more provinces, West-Vlaanderen and Antwerpen. Ranges of soil Hg contents were distinctly higher in the province Oost-Vlaanderen (interquartile range from 0.09 to 0.43 mg/kg) than in the other provinces (interquartile ranges from 0.7 - 0.13 and 0.7 - 0.15 mg/kg for West-Vlaanderen and Antwerpen, respectively). The standard threshold method was applied to separate soils containing baseline levels of Hg from the data. Baseline concentrations for Hg were characterized by a median of 0.10 mg Hg/kg dry soil, an interquartile range from 0.07 to 0.14 mg/kg and a 90% percentile value of 0.30 mg/kg. The influence of soil properties such as clay and organic carbon contents, and pH on baseline Hg concentrations was not important. Maps of the spatial distribution of Hg levels showed that the Province Oost-Vlaanderen exhibited zones with systematically higher Hg soil contents. This may be related to the former presence of many small-scale industries employing mercury in that region.

Keywords: soils, mercury, baseline concentrations, background concentrations, spatial distribution

Introduction

The metal mercury is known since antiquity. It was known to ancient Chinese and Hindus and was found in Egyptian tombs from around 1500 BC (Hammond, 1975). The huge incident of mass poisoning with Hg in Minamata Bay in Japan during the fifties has in a dramatic way drawn the attention on the significant hazards associated with its use (ATSDR, 1999).

As a result of natural processes such as volcanic activities and weathering of rock and ores, an estimated amount between 2 700 to 6 000 metric tons of Hg is released yearly in the environment. This is about 1.3 to 3 times the rate of release by anthropogenic sources (ATSDR, 1999). An estimated 10 000 metric tons of mercury are mined each year, although there is a considerable year-to-year variation (ATSDR, 1999). One third of that amount is used in the chlorine-alkaline-industry. Other uses include application in electronic industry, production of paints, paper industry, pharmaceutical industry, dentistry and agriculture (Sznoppek & Goonan, 2003).

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Mercury is naturally present in soils, although in very low concentrations. Worldwide mercury concentrations in soils range between 0.01 to 0.2 mg/kg soil (Adriano, 1986). Some concentrations reported for various countries are listed in Table 1.

Due to the long-distance aerial transport of trace elements, true geochemical background concentrations can be difficult to estimate. Geochemical background concentrations should represent natural concentrations, which exclude human influence. Geochemical baselines, in contrast, represent concentrations that are time and area specific. These are not always true background levels (Kabata-Pendias & Pendias, 1984; Kabata-Pendias *et al.*, 1992).

In a previous contribution (Tack *et al.*, 1997) we estimated baseline concentrations of trace elements for Flanders, Belgium, based on up to 461 observations (156 for Hg) in the province Oost-Vlaanderen between the cities of Gent and Antwerpen. The 90% percentile values for baseline Hg contents were estimated between 1.1 and 1.9 mg/kg with a median of 0.17 mg/kg. These levels were significantly higher than those reported elsewhere (Table 1). For this study, additional sampling was performed in other regions in Flanders to investigate whether these relatively high levels are common to different areas in the Flemish region and to improve estimates of baseline Hg-levels for the Flemish region.

Materials and methods

Compilation of the data

Mercury baseline concentrations were derived from data obtained from 316 sampling locations in three provinces, West-Vlaanderen ($n = 100$), Oost-Vlaanderen ($n = 116$) and Antwerpen ($n = 100$) in Flanders, Belgium (Figure 1). Soils in Flanders are mostly derived from recent sedimentary parent materials deposited during the Pleistocene or Holocene periods. The geographical regions in the studied area have been described previously (Tack *et al.*, 1997).

Samples were available from several sampling campaigns aimed at assessing baseline trace element concentrations in Flanders. Samples from the province Oost-Vlaanderen were taken during a survey study on soil and plant element contents, conducted by the Laboratory of Applied Analytical Chemistry and Ecochemistry in 1979 and 1980 (Cottenie and Verloo, 1985). Up to 494 points were sampled in an area covering approximately 630 km² between the cities of Gent and Antwerpen, of which 116 samples were analysed for Hg. Samples from the provinces West-Vlaanderen and Antwerpen were taken during a study in 1998 (Gellinck, 1998; Van Rompaey, 1998) using the same sampling approach as the Oost-Vlaanderen study.

Sampling locations were selected in agricultural fields (61% of all samples), forest (12%), pasture (20%) or fallow land (7%). In all studies, samples were taken from the topsoil (0-20 cm). Each sample is a composite of between four to eight samples randomly taken within 30 m around the selected grid point. Samples were stored in polyethylene bags, air-dried upon arrival in the laboratory. They were subsequently sieved through a 2-mm screen and stored in dark and dry conditions at room temperature.

Analytical procedures

Soil pH is based on measuring the pH of a 1:5 soil: water suspension (Van Ranst *et al.*, 1999). Soil clay and sand contents are based on classical wet and dry sieving techniques and the pipette method (Gee & Bauder, 1986). For the Oost-Vlaanderen samples, clay contents were in most instances estimated from clay contents determined in neighbouring similar soils (Tack *et al.*, 1997). Organic carbon was estimated using the Walkley & Black method (Nelson & Sommers, 1982).

From the more than 300 soil samples available from West-Vlaanderen and Antwerpen, 100 for each province were randomly selected to analyse the Hg content. Mercury data for the Oost-Vlaanderen soils were taken from the previous studies (Tack *et al.*, 1997; Tack & Verloo, 1996). To check the quality of these data, 17 samples were re-analysed. A paired t-test revealed no significant difference between the previous and current analyses.

Total soil mercury was analysed with flameless atomic absorption after a specific destruction procedure. In a destruction flask 60 mg V₂O₅ and 10 ml 65% ultrapure HNO₃ (Merck, Darmstadt) was added to 0.5 g of soil. The suspension was then heated at 165 °C for 30 minutes on a digester. Then, 15 ml of concentrated H₂SO₄ (18 mol/L) was added and destruction was continued for one hour. This procedure releases different forms of Hg in the form of the mercuric cation. For measurement, the suspension was transferred to a glass stoppered glass tube. After addition of 10 ml 10% SnCl₂ solution in 2% concentrated H₂SO₄, the tube was connected to the mercury analyser (Coleman Mercury Analyzer MAS-50B). The solution added reduces all mercury to the metallic form. Air is blown through the suspension that conducts the volatile elemental Hg through the apparatus such that a transient absorption signal is obtained. The apparatus was calibrated with standards of 0, 0.125, 0.25, 0.5, and 1 µg Hg. A typical calibration curve yielded absorbance values of 0.004, 0.053, 0.102, 0.197 and 0.377, respectively. The detection limit (2σ) was approximately 0.03 µg Hg/kg.

The accuracy of the method was verified through analysis of a calcareous loam soil CRM 141 R standard reference sample (Quevauviller *et al.*, 1996). The certified total Hg content of CRM 141 R is 0.25 ± 0.02 mg/kg Hg. Triplicate analysis yielded a value of 0.24 ± 0.05 mg/kg, with both uncertainty intervals reported as the half width of the 95% confidence interval of the mean.

Data processing

Statistical analysis was performed using R, an Open Source language and environment for statistical computing and graphics (<http://lib.stat.cmu.edu/R/CRAN/>) (Ihaka & Gentleman, 1996). To segregate the population of soil containing baseline Hg levels from the collected data, probability plots were used to evaluate the presence of multiple populations within the data sets. Multiple populations will form distinct straight-line segments on a probability plot provided each population is normally distributed. Where two populations overlap, an inflection point will form. This point, referred to as the "threshold point" by Fleischhauer & Korte (1990), represents the separation point between two populations. The threshold was taken objectively as the value that gave the skewness of the resultant background population nearest to 0 as described in Fleischhauer and Korte (1990) and Tobias *et al.* (1997). The highest values were consecutively removed from the data and the skewness of each resulting population was calculated. The threshold point was identified as the data point for which the resulting population showed the skewness nearest to 0. Through this method, populations can be identified and segregated from one another for further analysis. Segregation of

the data will be partial where overlap exists; however, the statistical bias probably will remain small (Fleischhauer and Korte, 1990). Baseline mercury concentration ranges were estimated from the segregated dataset using non-parametric statistics such as the median and percentile values.

Contour maps displaying the spatial distribution of Hg in each province were produced based on geostatistical analysis using the package GeoR (<http://www.est.ufpr.br/geoR/>) (Ribeiro & Diggle, 2001). Spatial interpolation was performed using ordinary kriging. Covariance parameters were estimated by fitting a parametric model to an empirical variogram using ordinary least squares. For other settings, program defaults were used (Ribeiro & Diggle, 2001).

Results and discussion

Soil properties and mercury contents

A statistical summary of the observations grouped by province and for the combined data is provided in Table 2. Soils of West-Vlaanderen are characterised by higher clay and pH values compared to soils in the other provinces. The soils in Antwerpen tend to be sandy and acidic. The soils of Oost-Vlaanderen have intermediate characteristics. For all data, organic carbon contents ranged from below 1% to 24%, but typical soils in the Flemish region have organic carbon contents between 1.5 - 2.5% , corresponding to about 3 - 5% organic matter.

Mercury contents ranged from lower than the detection limit (0.03 mg/kg) to 4.2 mg/kg dry soil. This is a wide range, well above soil mercury ranges reported for non polluted soils (Table 1). Percentile values show that the area sampled in Oost-Vlaanderen clearly has higher soil Hg-contents than the other provinces. The median for Oost-Vlaanderen, at 1.8 mg/kg, is almost twice that of the other provinces and that for all data. One quarter of the Oost-Vlaanderen soil samples had mercury levels above 0.37 mg/kg.

Figure 2 shows a boxplot of the data, where the difference between Oost-Vlaanderen and the other provinces is very apparent. Values larger than 1 mg Hg/kg were not plotted. Many of the data of Oost-Vlaanderen are considered 'outliers' in the boxplot of the whole data, i.e., they extend further than 1.5 times the interquartile range and are plotted as dots in the boxplot of the entire data.

Derivation of baseline values

Concentrations ranges commonly found in the majority of soils of the region are considered baseline concentrations ranges. To estimate baseline concentration ranges from the data, the “standard threshold method”, described by Fleischhauer & Korte (1990) and Tobias *et al.* (1997) was applied. In this approach it is assumed that elemental contents will be distributed according to a log-normal distribution. When a different population is represented in the data, it will show up as a deviation from the log-normal distribution.

The normal probability plot of the mercury contents is shown in Figure 3. Low values deviate from linearity because these were under the detection limit and had all been assigned a value of 0.03 mg/kg. Two different linear segments revealed the presence of two different populations. High data points were removed until the skew of the distribution became minimal. That point was considered the threshold, delineating the baseline-population from soils from a different population. The

threshold was located at $\log \text{Hg} = -0.39$, which corresponds to a Hg-level of 0.41 mg/kg. Values higher than 0.41 are therefore assumed not to belong to the population of soils with baseline Hg levels. Data below the threshold were retained to estimate background Hg contents in Flemish soils from their statistics.

Percentile values for baseline mercury contents are listed in Table 3. These values can be used as reference values to determine whether a soil might be contaminated or not. Such an indication of contamination only implies that the total contents are higher than one would normally expect in unpolluted areas. No indications may be inferred about potential hazards that could be associated with the observed element contents.

Correlations with soil properties

Soil properties are expected to be correlated with baseline contents of metals (Tack *et al.*, 1997). Soil constituents such as clay and organic carbon increase the binding capacity of the soil for metals (Bolt & Bruggenwert, 1976). They decrease the probability that elements released from weathering of the soil minerals or entering the soil through aerial transport and deposition or other sources will be removed by leaching. Baseline contents of metals will therefore tend to be higher in soils with high contents of clay and/or organic matter. Mobility and retention is also strongly affected by soil pH. Cationic elements tend to be more mobile with decreasing pH, while anionic elements will become mobilised at higher pH values (Förstner, 1990).

Scatterplots of baseline Hg contents as a function of soil clay and organic carbon contents and pH (Figure 4) do not reveal distinct trends, although some observations can be made. Mercury contents in soils with high clay contents and high organic C contents (plots A and B in Figure 4) vary over a wider range than those in soils with lower clay or organic carbon contents. This reflects the metal binding capacity of these soil constituents. Mercury, entering the soil system through aerial transport and deposition or released through weathering of soil minerals, will more likely be retained in soils high in clay or organic matter. Also differences in mineralogy and hence lithogenic Hg levels between sandy and clayey soils may constitute a factor. With respect to pH, wider ranges in Hg levels occurred in soils with higher pH-values, reflecting a higher tendency for binding in these soils.

In line with the wide scatter in the data, linear trends between soil properties and mercury contents were mostly not significant (Table 4). Only for organic carbon was there a significant positive correlation with baseline Hg concentrations. However, only 2.5 % of the total variability in baseline Hg contents is explained by a linear regression model with organic carbon as predictor variable ($\text{Hg (mg/kg)} = 0.098 + 0.0110 \text{ OC(\%)}; R^2 = 0.0252, p = 0.009$). Thus, the influence of soil properties on Hg baseline contents overall is, although statistically significant, not very important, in contrast to trends found for trace elements such as Cd, Cu, Pb or Zn (Tack *et al.*, 1997). While clay and organic carbon contents may be major factors in determining the retention of trace elements that mainly occur in cationic forms (e.g. Zn, Cu, Pb), the more complex environmental chemistry of Hg implies that several mechanisms may control Hg retention. For example, the retention of Hg in soils is not only caused by valence-type ionic adsorption on organic and inorganic materials. It may also be controlled by formation of covalent bonds with organic compounds or by precipitation as highly insoluble carbonates, phosphates or sulphides (Adriano, 1986; Yaron *et al.*, 1996).

Spatial distribution of mercury levels

The spatial distribution of Hg contents as estimated by kriging, is mapped in Figures 5 to 7 for West-Vlaanderen, Oost-Vlaanderen and Antwerpen, respectively. Contours were plotted with respect to the estimated baseline values. White and light gray correspond to levels below and darker grays to levels above the 90% percentile values of the estimated baseline contents. These plots may allow to identify areas where systematically higher levels were encountered, but should not be used to draw conclusions on the parcel level because the sampling grid is too coarse.

Two spots with higher levels in West-Vlaanderen (Figure 5) are based on a single measuring point and should therefore be further investigated before conclusions can be drawn. Also for the third, larger area, no readily explanation for the higher levels is available as it concerns a rural area. The sampled area in Oost-Vlaanderen (Figure 6) shows large areas with concentrations above the 90% percentile value of baseline levels, indicating systematically higher Hg soil contents in that area. A possible explanation of the markedly increased Hg levels in the investigated part of the province Oost-Vlaanderen can be found in the former presence of many small-scale industries employing Hg in that region. The area around the city of Lokeren, for example, was particularly known for the production of felt. In the old processes for the production of felt, mercuric nitrate was used to hydrolyse rabbit fur (ATSDR, 1999). Areas with levels above 1 mg/kg correspond to urban and industrialised areas around Lokeren, Hamme, and Kruibeke.

Figure 7 shows the map for the province of Antwerpen. Most of the area is below the 90% percentile of the derived baseline Hg contents. The south west in particular shows higher contents. It corresponds to a highly industrialised area with non-ferro metallurgical, electro-technical and chemical industry. The area around Antwerpen harbour, another highly industrialised region in the north-west, was not included in the sampling.

In summary, baseline levels estimated from the current data are in line with those reported elsewhere, with a median of 0.102 and a 90% percentile value of 0.229 mg Hg/kg dry soil. Soil Hg levels in some of the areas sampled in Oost-Vlaanderen appeared significantly affected by human activities.

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Figure captions

Figure 1. Map indicating sampling locations in the three provinces, West-Vlaanderen, Oost-Vlaanderen and Antwerpen from left to right, respectively.

Figure 2. Box -and-whisker plot of the data for each of the Provinces and for all data. Whiskers extend to to the most extreme data point which is no more than 1.5 times the interquartile range from the box. Values higher than 1 mg/kg were not plotted.

Figure 3. Normal probability plot of the data with indication of the 'threshold' (see text for explanation).

Figure 4. Baseline Hg contents as a function of soil clay content, organic carbon content and pH.

Figure 5. Spatial distribution of Hg contents in the province West-Vlaanderen with indication of the sampling points. White indicates Hg levels below the median of baseline Hg levels, light gray below the 90% percentile values of baseline levels. Darker colours represent levels above baseline ranges as indicated by the figure legend. Coordinates, which are expressed in meters (m), are according the Belgian Lambert system.

Figure 6. Spatial distribution of Hg contents in the province Oost-Vlaanderen with indication of the sampling points. White indicates Hg levels below the median of baseline Hg levels, light gray below the 90% percentile values of baseline levels. Darker colours represent levels above baseline ranges as indicated by the figure legend. Coordinates, which are expressed in meters (m), are according the Belgian Lambert system.

Figure 7. Spatial distribution of Hg contents in the province Antwerpen with indication of the sampling points. White indicates Hg levels below the median of baseline Hg levels, light gray below the 90% percentile values of baseline levels. Darker colours represent levels above baseline ranges as indicated by the figure legend. Coordinates, which are expressed in meters (m), are according the Belgian Lambert system.

Table 1. Some soil Hg contents reported in literature

		Hg (mg/kg)	Reference
Brazil	Average	0.077	Brabo <i>et al.</i> (2003)
China	Background	0.02-0.03	Li & Wu (1991)
Florida	Baseline	0.0008-0.04	Chen <i>et al.</i> (1999)
Korea	Unpolluted soils (n = 109)	0.045	Kim & Kim (1999)
Ohio, USA	Median	0.06	Cox & Colvin (1996)
Slovakia	Median	0.06-0.13	Curlik <i>et al.</i> (1995)
Czech republic	Background	0.12-0.19	Podlesakova & Nemecec (1995)
Worldwide	Background	< 0.4	Kabata-Pendias & Pendias (1984)

Table 2. Summary statistics of the data

	n	Min.	1st Qu.	Median	Mean	3rd Qu.	Max.
<i>West-Vlaanderen</i>							
Hg (mg/kg)	100	0.03	0.07	0.11	0.13	0.13	1.47
Clay (%)	100	0.6	8.5	12.3	16.7	19.6	58.1
Sand (%)	100	8.3	32.8	54.8	53.6	71.1	99.5
OC (%)	100	0.06	1.29	1.57	2.02	2.55	6.88
pH-H ₂ O	100	3.71	5.92	6.5	6.58	7.49	8.73
<i>Oost-Vlaanderen</i>							
Hg (mg/kg)	116	0.03	0.09	0.18	0.37	0.43	3.07
Clay (%)	108	0.4	5.4	8.3	13.6	16.5	53.8
Sand (%)	108	5.7	40.5	56.9	56.8	77.3	94.7
OC (%)	116	0.36	1.26	1.61	2.05	2.04	10.4
pH-H ₂ O	116	4.3	5.54	6	6.13	6.71	8.1
<i>Antwerpen</i>							
Hg (mg/kg)	100	0.03	0.07	0.10	0.21	0.19	4.19
Clay (%)	100	0	2	3.6	4.6	5.3	30.8
Sand (%)	100	42.4	78.7	87.5	83.9	92.6	98.6
OC (%)	100	0.52	1.52	2.11	2.61	2.71	23.81
pH-H ₂ O	100	3.31	4.2	5.21	5.12	5.8	7.4
<i>All data</i>							
Hg (mg/kg)	316	0.03	0.07	0.12	0.24	0.22	4.19
Clay (%)	308	0	3.7	7.5	11.7	14.0	58.1
Sand (%)	308	5.7	45.4	70.9	64.5	86.1	99.5
OC (%)	316	0.06	1.32	1.74	2.22	2.51	23.81
pH-H ₂ O	316	3.31	5.28	5.95	5.95	6.63	8.73

Table 3. Estimates of baseline ranges for mercury (mg/kg dry soil) based on the percentile values of the data considered to be baseline values.

Percentile	5%	25%	50%	75%	90%	95%
Hg (mg/kg)	0.030	0.067	0.102	0.143	0.229	0.301

Table 4. Pearson correlations between Hg contents and some soil properties. Significance at the 5% level is indicated with "**", at the 1% level with "***"

	Hg (mg/kg)	Clay (%)	Sand (%)	OC (%)	pH
Hg (mg/kg)	1.000	0.033	-0.058	0.159**	0.043
Clay (%)		1.000	-0.844**	0.184**	0.640**
Sand (%)			1.000	-0.087	-0.569**
OC (%)				1.000	-0.037
pH					1.000

Normal Q-Q Plot

West-Vlaanderen

Oost-Vlaanderen

Antwerpen