Use of Trace Elements from Historical Mining for Alluvial Sediment Dating

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Abstract

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We examined whether it is possible to relate concentrations of trace elements in alluvial sediments with records concerning the intensity of mining, and use them as a means of dating. We conducted our research in the medieval mining district of the town of Kutná Hora in the Czech Republic. Samples were collected under the pond dam and analysed for clay, silt and sand content and for As, Be, Cd, Co, Cr, Cu, Mo, Ni, Pb, V and Zn concentrations. We observed two main peaks of element concentrations (Be, Co, Cr, Cu, Hg, Pb, V and Zn), independent of grain fractions. The peaks were interpreted as a result of human activity. The concentration curves, stratigraphy and location of the dam/alluvium boundary were compared with historical records of mining production. This means of dam dating into the 16th century agreed with historical dating from written sources. Trace elements were also successfully used as stratigraphic markers. The comparison between concentration patterns of V and other well interpreted elements (Be, Co, Cr, Cu, Hg, Pb and Zn) enabled to recognize a material directly originating from the mines. The elements thus helped to interpret local sedimentation history.

Keywords: archaeological methods; heavy metals; historic metallurgy; human impact; landscape development; Middle Ages

The variability in concentrations of trace elements in sedimentary records is frequently connected with human mining and smelting activities. In this paper, we concentrate on the use of trace elements from medieval mining for the dating and chronological analysis of alluvial sediments. It requires the correlation of element concentrations with historical records concerning mining and/or smelting activities.

This correlation is mostly provided in papers focused on mining of the 19th and 20th centuries (e.g. Lecce & Pavlowsky 2001; Knox 2006; Mihaljevič *et al.* 2006; Bing *et al.* 2011; Xia *et al.* 2011).

Thorndycraft *et al.* (2004) was probably the closest to correlating medieval records concerning the intensity of mining activities with concentrations of trace elements in sediments. In this study, well dated concentration curves of Sn in alluvial sediments were provided, together with data concerning medieval Sn production. Despite this fact, the correlation was not provided. Also Schmidt-Wygash *et al.* (2010) used trace element concentrations as a means of dating related to written sources, but they did not provide a production-concentration relation.

We therefore considered whether concentrations of trace elements in alluvial sediments could be used for the numerical dating of sediments and landscape structures in medieval mining and/or smelting areas.

To answer this question, we determined concentrations of trace elements in alluvial sediments in the region of Kutná Hora (Kuttenberg in medieval German sources), a medieval centre of Ag and Cu mining in the Czech Kingdom. Veselý and Gürtlerová (1996) documented that medieval mining increased the concentration of trace elements in alluvial sediments 20 km below the Kutná Hora mining area, but they did not provide a production-concentration correlation.

In our study, we performed an analysis of the alluvial sediment collected beneath a historically dated fish pond dam built in the 16th century, to obtain medieval sedimentary records not disturbed by any modern human activities.

The aim of this paper was to answer the following questions: (1) Is it possible to relate the concentration of trace elements in alluvial sediments with historic records of mining production? (2) If so, is it possible to use the concentration as a means of sediment dating? (3) Which elements are the best indicators of past mining activities, are there any information-bearing differences between the elements?

MATERIAL AND METHOD

Study site. The studied dam of deserted St. Anne's fish pond is located 4 km northeast of the town of Kutná Hora and 1 km north of the village of Nové Dvory (Figure 1; 49°58'51.457''N, 15°19'31.206''E). The fish pond was built on the Klejnárka River, which originally flowed through the area of the pond in a northeast direction. The alluvium was influenced by sewage waters from mines of Kutná Hora and Kaňk Mt. (352 m a.s.l.) by the Vrchlice River, which collected most of these waters.

The sampling site is situated in the broad alluvium of Klejnárka and Stará Klejnárka Rivers (Figure 1, numbers 9, 11 and 12). The geological bedrock is built mainly of Pleistocene sediments covering the Mesozoic rocks. The cover is formed by fine-grained fluvial sediments. In the southern

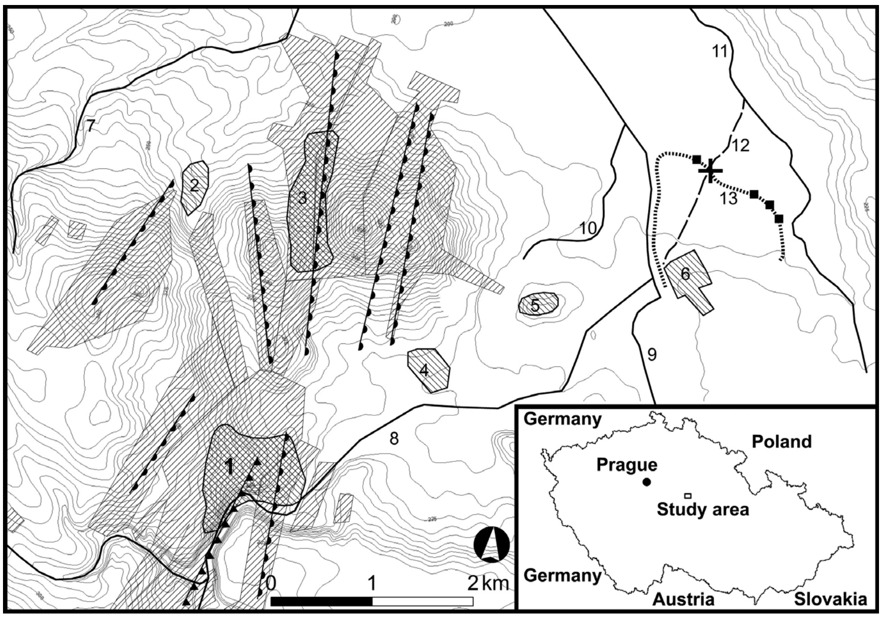

Figure 1. Location of the study site in the Czech Republic and detail plan of the study site; numbered features: hatched from left to right the town of Kutná Hora (1), mining villages of Grunta (2) and Kaňk (3), Sedlec monastery (4), Malín (5) and Nové Dvory (6). Solid lines represent Hořanský stream (7), Vrchlice River (8), Klejnárka River (9), Beránka stream (10), Stará Klejnárka stream (11) and the original Klejnárka stream is represented by the dashed line (12); the dotted line shows St. Anne's fish pond dam (13); not numbered features: hatched from right to left undermined areas, lines with semi-circles represent the main ore zones; line with triangles represents the Osel ore zone; the dagger in the intersection of stream (12) and pond dam (13) indicates the position of the sampling site; the squares on the dotted line (13 – fish pond dam) indicate other four dug probes; the fine solid lines represent contour lines with the spacing of 5 meters

part of St. Anne's fish pond are also loess and deluvial sediments of loess and sand-gravel Pleistocene terrace of the Nové Dvory area. Other Pleistocene terraces are located on the hillsides of Kaňk Mt. and also of the ridge eastward of the fish pond area. Loessic sediments are located mostly westward and southwestward from the fish pond area, on both sides of the lower Vrchlice River. The main soil types in the alluvium are mostly Fluvisols and Phaeozems.

The ore-bearing rocks of Kaňk Mt. are composed of gneiss of various kinds (both types of mica, biotitic, micacitic), micacites, migmatites and migmatitized orthogneiss. The main metal-bearing minerals are native Ag, and high-quality ores with minerals such as tetrahedrite, freibergite, argentite, proustite, pyrargyrite and galenite and also sulphides of Fe, Zn, As, Cu and Pb (Bartoš 2004; CENIA 2012). The altitude of the study site is 205 m a.s.l., the average annual air temperature is between 8 and 9°C and the average annual precipitation is between 550 and 600 mm (Tolasz *et al.* 2007).

Based on the historical records, St. Anne's fish pond was built between 1501 and 1552 (Ledr 1884). The pond was drained in 1775 and its area was transformed into arable land (Lipský & Kukla 2009).

The intensity of Ag and Cu mining greatly changed in the Kutná Hora district over time (Kořan 1950). Written sources which can be used for detailed documentation of metal production date back to the second half of the 15th century. Production by the 15th century was reconstructed on the basis of indirect sources, such as royal taxes and rates of metal trading.

The mining of Ag reached its first and highest peak in the 13th and 14th centuries (Figure 2). The following decline in mining was caused by technical factors and then by economic chaos during Hussite wars in the first half of the 15th century (Kořan 1950; Bílek 2001; Bartoš 2004). Subsequently there was a renewal of mining activities. In the half of the 16th century, the main mine of the Osel zone was closed and the total production rapidly decreased (Kořan 1950; Bartoš 2004).

Field sampling. The sampling site was located in the area of the original Klejnárka River stream, reconstructed according to historical maps and field observations, on the outer side of the dam. We used a 50 × 150 cm probe, dug through the dam and alluvial sediments beneath. The depth of the profile analysed was 160 cm (Figure 3). Beside this probe, other four probes were made at various points of the pond dam to obtain more information about stratigraphy and sediment conditions (see the location of probes in Figure 1).

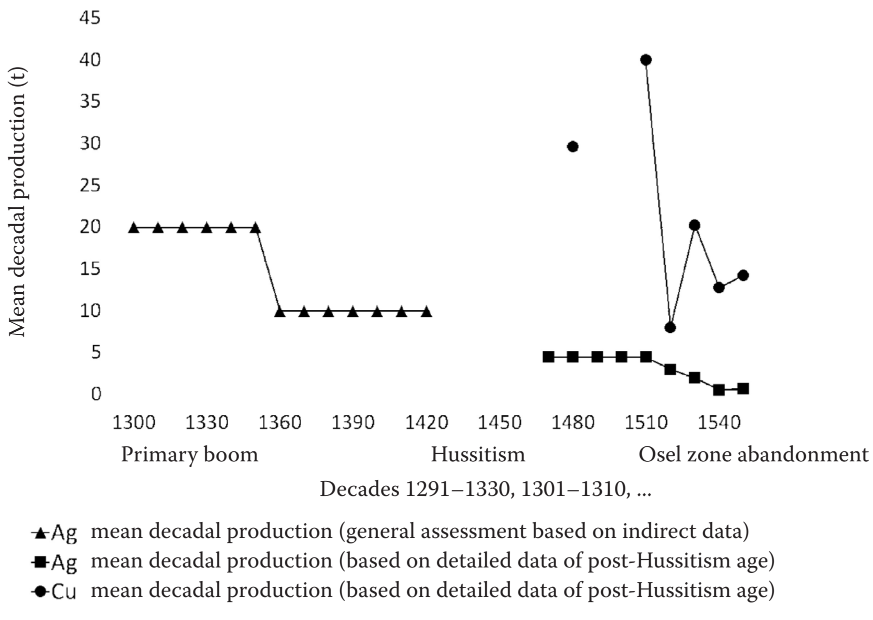

Figure 2. Historical mining production in Kutná Hora mines according to Kořan (1950); silver and copper production was reconstructed according to direct and indirect historical sources; primary boom indicates the first boom of mining in the 14th century; Hussitism indicates a mining decrease during the Hussite revolution in the first half of the 15th century; Osel zone abandonment indicates the termination of mining activities in one of the most important ore zones, the Osel zone; it corresponds with the decrease in metal production after 1510

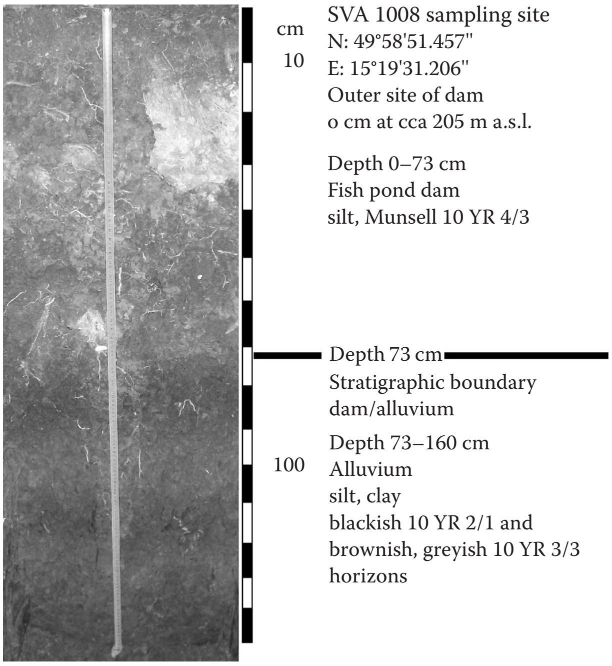

Figure 3. Photograph of the analysed alluvial profile

Sediment samples for the trace element analysis were taken from 75 to 150 cm depth at regular distances of 5 cm, and we collected 16 samples. The fresh weight of each sediment sample ranged from 300 to 500 g.

Data analysis. Sediment samples were sent to the laboratory of the Research Institute for Soil and Water Conservation (www.vumop.cz). Twelve trace elements were determined (As, Be, Cd, Co, Cr, Cu, Mo, NI, Pb, V, Zn). The samples were treated according to Standard ISO 11464 (dried, ground to a size of 0.25 mm or less and dried again). Eleven elements were determined in Varian AA240 apparatus (Varian Inc., www.varianinc.com) after extraction of the sample in 2M HNO_3 solution at a ratio of soil to agent of 1:10. Concentrations of Hg were determined by ashing and amalgamation in AMA254 apparatus (LECO Corporation, www.leco.com). The content of clay (< 0.002 mm), silt (0.002–0.05 mm) and sand (0.05–2.0 mm) was analysed by sieving and sedimentation methods according to Standard ISO 11277. We analysed three categories of elements according to their content in mined ores (Oraský 1987): (1) abundant elements in mined ores (As, Cu, Pb and Zn), (2) attendant and rare elements (Be, Cd, Co, Hg, Mo and Ni) and (3) very rare elements (V).

The complex of elements is of methodical importance for interpretation of the vertical record of element concentrations. One of the most frequent objections to this type of research is potential mobility of elements through the sedimentary environment. The complex of elements contains the elements of various (and contradictory) modes of mobility (Adriano 1986). We use the complex of elements to avoid the problems with possible mobility or to minimize its influence.

In order to evaluate the relationship between the concentrations of all analysed elements and the grain fractions, we used PCA analysis in Canoco for Windows 4.5 (ter Braak & Šmilauer 2002) and the CanoDraw programs. We also used linear least-square regression in the Statistica 9.0 program (www.statsoft.com).

RESULTS

The dug probe (Figure 3) discovered the body of the dam and also the alluvium beneath. The dam was made of silty homogenized material of Munsell colour 10 YR 4/3. The structure was mainly blocky (both angular and sub-angular), with aggregate size about 1 cm. The root systems and disturbance were observed. The alluvium was mainly composed of clay (or silty clay) and was characterized by alternation of blackish (10 YR 2/1) and brownish (10 YR 3/3) horizons. These horizons indicated repeated flooding and A horizon development on the alluvial surface. The structure was prismatic; the size of aggregates was about 1 to 2 cm, rarely up to 5 cm. The root system extent and disturbance were observed only rarely (see Figure 3), much less than in the dam body. No animal disturbance was observed.

Silt and clay material in the dam body was characteristic also in other dug probes with the exception of the most eastern one. There was a visibly higher content of sand fraction.

The stratigraphic boundary between dam and alluvium was distinguished due to a change in the texture and structure of sediments and also due to the identification of alluvium indicated by repeated A horizon development. This boundary indicated by a darker horizon was found also in the most eastern probe. Other probes did not reach the boundary.

The mean proportion of clay, silt and sand over the analysed profile was 50, 40 and 10%, respectively, and this proportion was relatively stable within the profile (Figures 4 and 5). The sediment was classified as clay to silty clay.

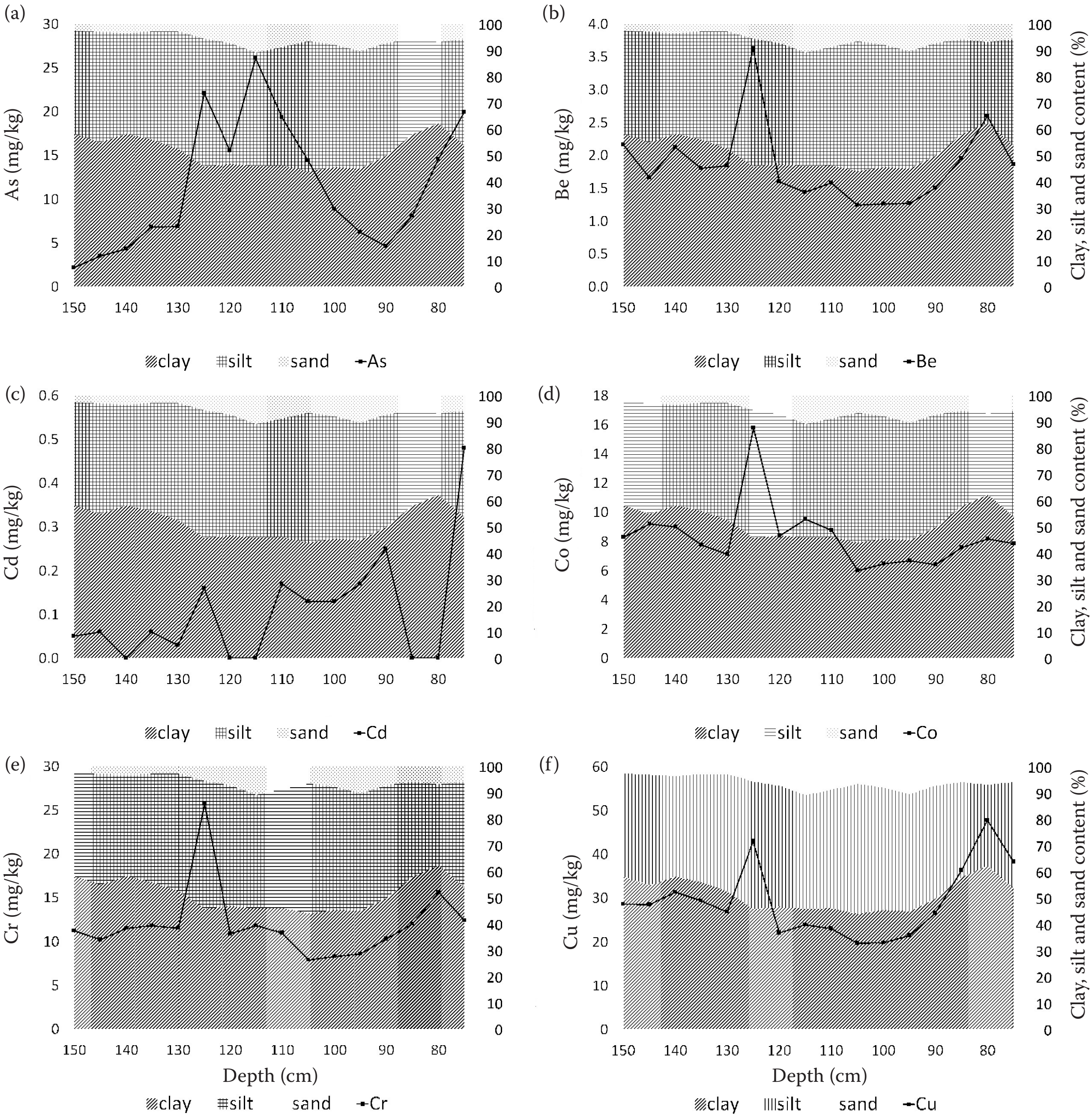

Figure 4. Concentrations of (a) As, (b) Be, (c) Cd, (d) Co, (e) Cr and (f) Cu in the analysed profile; the right *y* axis indicates the proportion (in %) of clay, silt and sand at the particular depth; the charts represent heavy metal content values at depths between 150 and 75 cm; the stratigraphic boundary between the dam and the alluvium was at a depth of 73 cm (for details see Figure 3)

Concentrations of elements reached hundredths (Hg), tenths (Cd and Mo), mg/kg (Be and Co), and tens of mg/kg (As, Cr, Cu, Ni, Pb, V and Zn). The PCA analysis explained the 42.4% variability of analysed data on the first axis and 88.9% variability on the four ordination axes together (Figure 6). According to the diagram, similar chemical properties were recorded in samples collected from depths ranging from 85 to 90 and 130 to 150 cm, as marks for these depths were close together in the diagram. The second cluster of similar samples was recorded for depths ranging from 95 to 120 cm. Samples collected from depths of 75, 80 and 125 cm highly differed in chemical properties from the above-mentioned clusters.

The relationships between element concentrations and grain fraction content are depicted in Table 1 (only significant results with $P < 0.05$).

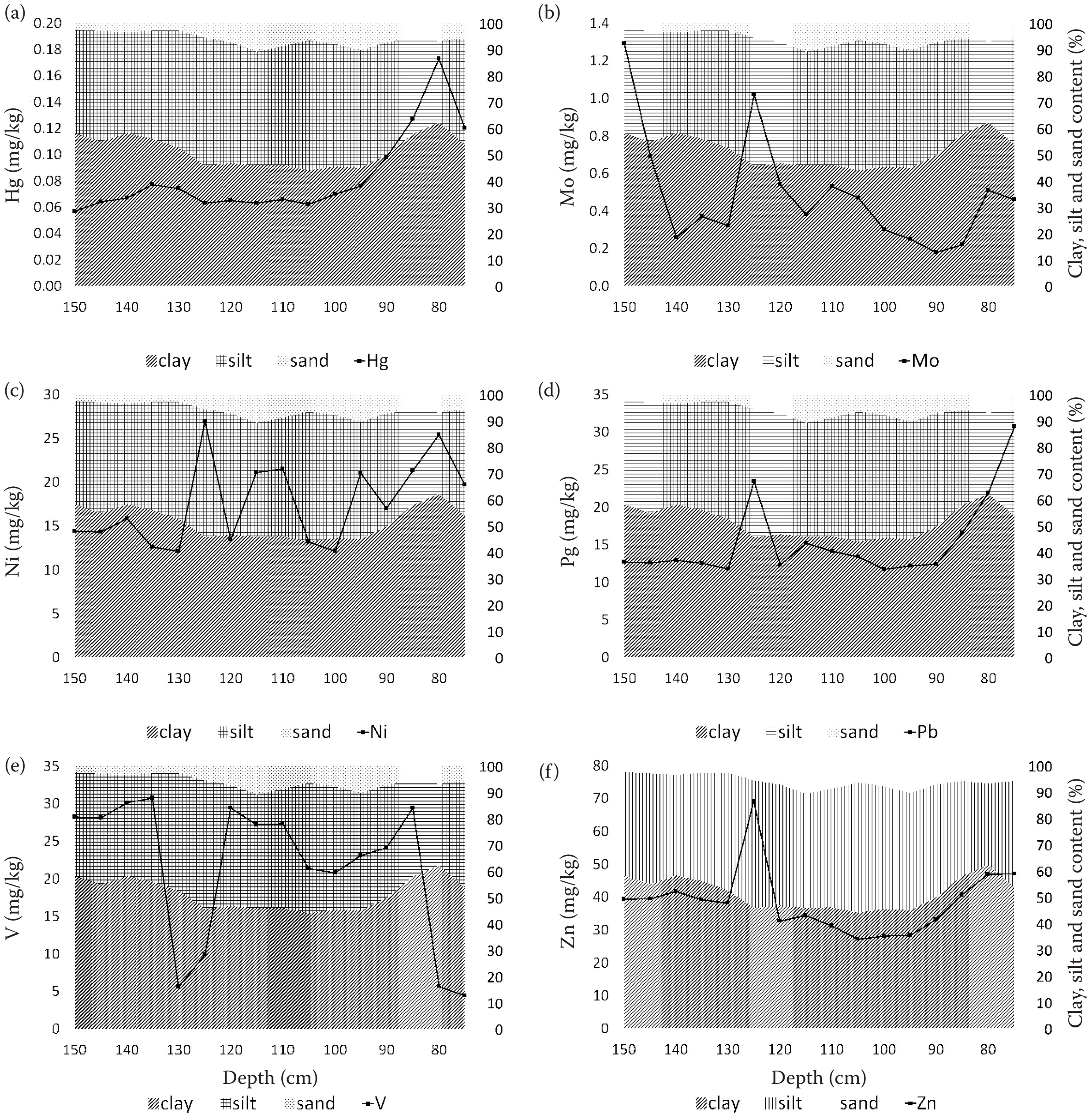

Figure 5. Concentrations of (a) Hg, (b) Mo, (c) Ni, (d) Pb, (e) V and (f) Zn in the analysed profile; the right y axis indicates the proportion (in %) of clay, silt and sand at the particular depth; the charts represent heavy metal content values at depths between 150 and 75 cm; the stratigraphic boundary between the dam and the alluvium was at a depth of 73 cm (for details see Figure 3)

Using all data available, the relationship to clay was observed for Cu and Hg, to silt for Cu and Hg and to sand for As. Using the data without outlying samples (125, 80 and 75 cm) the relationship to clay was observed for As, Be, Cr, Cu, Zn, to silt for Be, Cr, Zn, to sand for As, Be, Cu, Ni and Zn.

Concentrations of individual elements are given in Figures 4 and 5. There was a group of elements with a similar development pattern of positive (Be, Co, Cr, Cu, Hg, Mo, Pb and Zn) and negative (V) peaks not respecting the content of grain fractions. These peaks were found at depths of 125 cm and from 80 to 75 cm (see Figure 6), and were the most significant for Cu (Figure 7). For some of the above-mentioned elements, both peaks were recorded (Be, Cr, Cu, Pb, V and Zn), but for others only the first (Co and Mo) or the second (Hg) peak was recorded.

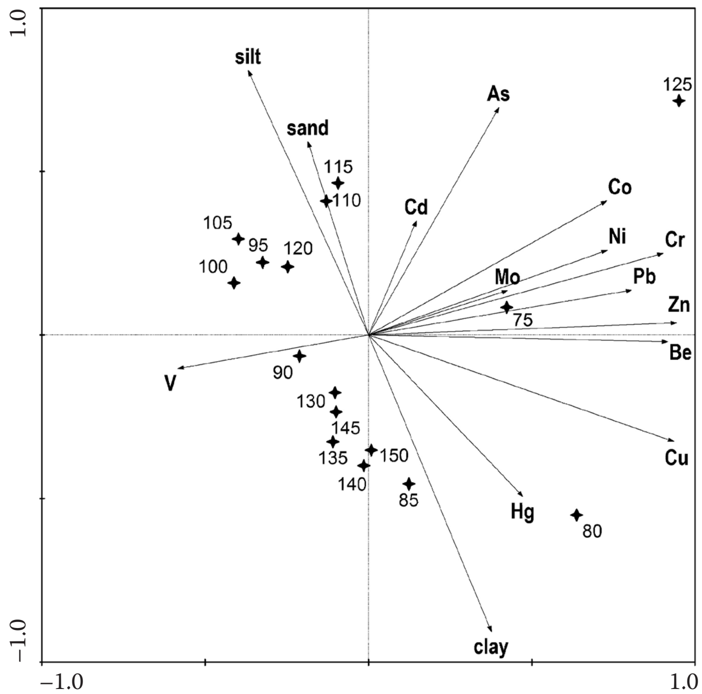

Figure 6. Ordination diagram showing the results of PCA analysis; the number of each sediment sample corresponds with the sample depth in the analysed profile (for details see Figure 3, and also Figures 4 and 5, where the depths of samples are plotted on the *x* axis)

Table 1. Results of linear regression analysis of the relationship between the content of clay, sand and silt and concentrations of elements in sediment samples; only significant results are provided; two types of analysis were performed – for all data together (all) and for data without three of the most outlying samples (without)

Element	Fraction	Analysis	*a*	*b*	*r*	*P*
	clay	without	50.43	–0.80	–0.64	0.020
As	sand	without	0.06	1.61	0.68	0.011
	sand	all	2.20	1.54	0.55	0.027
	clay	without	–0.95	0.05	0.92	< 0.001
Be	sand	without	2.12	–0.08	–0.74	0.004
	silt	without	4.65	–0.07	–0.81	0.001
Cr	clay	without	2.35	0.16	0.64	0.018
	silt	without	22.26	–0.27	–0.70	0.008
	clay	without	–14.78	0.80	0.92	< 0.001
	clay	all	–16.29	0.88	0.63	0.008
Cu	sand	without	31.82	–0.97	–0.59	0.034
	silt	without	80.41	–1.26	–0.94	< 0.001
	silt	all	76.31	–1.11	–0.63	0.009
Hg	clay	all	–0.07	0.00	0.57	0.021
	silt	all	0.30	–0.01	–0.76	0.001
Ni	sand	without	11.31	0.80	0.63	0.022
	clay	without	–9.16	0.87	0.94	< 0.001
Zn	sand	without	42.57	–1.28	–0.74	0.004
	silt	without	87.02	–1.21	–0.85	< 0.001

a, *b* – coefficients from the regression equation $y = a + bx$; *r* – regression coefficient; *P* – probability value

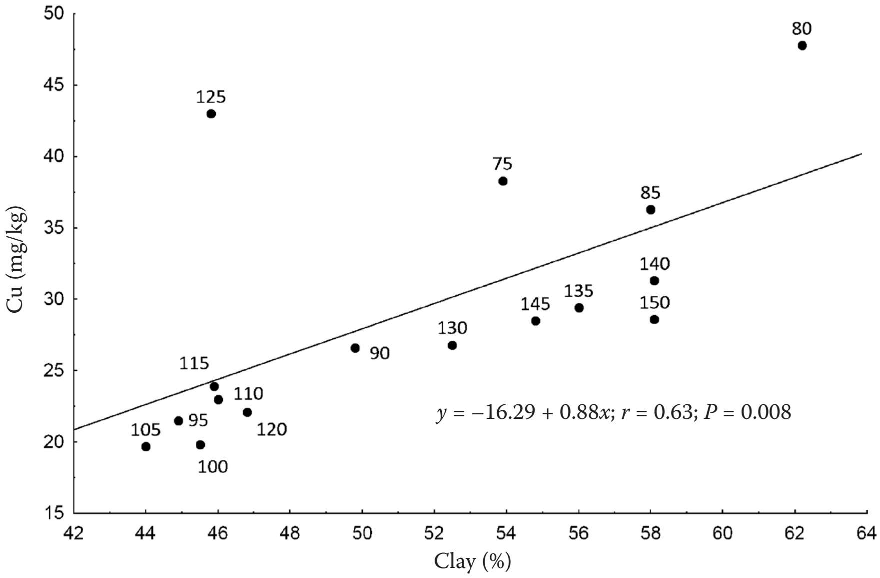

Figure 7. Relationship between the concentration of Cu in sediment samples and the content of clay fraction in the sediment; analysis for all available data together

Negative peaks for V inversely followed positive peaks of many other elements. In the case of Mo, the peak was also recorded at a depth of 150 cm. Concentrations of As, Cd and Ni did not follow an obvious development pattern with respect to the peaks of other elements.

DISCUSSION

Result interpretation

Concentrations of many elements are in general related to the content of different fractions in sediments (Adriano 1986); therefore we performed as a preliminary step the identification of variability in concentrations of elements caused by the content of clay, silt and sand in sediment samples. The depths with concentrations of elements out of any natural relationship with grain fractions were interpreted as a result of mining and smelting activities. These depths were found to be 125, 80 and 75 cm.

The analysed sedimentary profile was divided into several phases according to concentrations of elements and also according to chronological development. The first phase was represented by samples from 150 to 130 cm, forming a separate cluster in the diagram in Figure 6. This phase clearly followed natural relations to grain fractions, and therefore there was no obvious effect of mining and smelting activities. Sediments at these depths were probably deposited before the second half of the 13^{th} century. The first peak in concentrations of many elements (Be, Co, Cr, Cu, Mo, Pb, V and Zn) was found at a depth of 125 cm. We interpreted this as the start of mining at the end of the 13^{th} century. The next phase was represented by samples collected from depths from 120 to 95 cm. In this phase, the samples created the clearly separate cluster from other depths (see Figure 6), where the concentrations were not related to the clay fraction. We interpreted this as a record of human impact, but not as distinctive as the peak at a depth of 125 cm. Depths of 90 and 85 cm created the same cluster with depths ranging from 150 to 130 cm. We interpreted it as a result of no or minimal mining activities. This corresponds well with historical records of minimal mining during and after the Hussite Wars in the first half of the 15^{th} century (Kořan 1950; Bílek 2001; Bartoš 2004). The last phase was represented by depths of 80 and 75 cm. This peak of elements (Be, Cr, Cu, Hg, Pb, V and Zn) was interpreted as a record of renewed mining activities in the second half of the 15^{th} century.

Some elements (Co, Hg and Mo) reached one peak only, and this was probably due to the mining of partly different ores at different times, or because of different technologies used during manufacturing.

There were also element concentration curves which we do not know how to interpret (As, Cd and Ni). These elements do not follow the pattern of clear peaks as do other elements (Be, Co, Cr, Cu, Hg, Mo, Pb, V and Zn). The fact that the majority of the elements follow a certain clear pattern is the reason to see a certain objective agent (mining activities in this case) and to see the concentrations as non-random. Then why do not these three elements follow the pattern as the other do? The concentrations of As, Cd and Ni can be random, or these elements are bound in molecules prone to vertical mobility, or they record mining activities more precisely than the other elements. These questions demand using more methods of analysis and can be the topic of future research. Mo and its peak at a depth of 150 cm do not enable a clear interpretation whether it is caused by human or natural factors.

Dating of St. Anne's fish pond foundation

The stratigraphic boundary between the dam and the alluvial sediments (Figure 3), which represents the origin of the dam, was recorded in the descending part of the second peak of trace element concentrations (at depths of 80 and 75 cm). We interpreted this decrease in concentrations as a result of the decrease in mining in the first half of the 16th century (Figure 2). The historical dating of the dam construction into intervals between the years of 1501 and 1552 thus agrees with the indirect dating by means of concentrations of trace elements. The crucial factor in the numerical dating of sediments according to concentrations of trace elements is the possibility to correlate the curves of concentrations of many elements with historical or archaeological records, documenting mining and/or smelting activities and their intensity. In the case of our profile, the use of any independent dating method was not possible because no organic materials suitable for ^{14}C analysis were discovered. In the case of our sedimentary record the dating is therefore based only on the correlation of concentrations of trace elements in the sediment and records available about the intensity of mining in the study area.

Vanadium concentration interpretation

The opposite development pattern of V in relation to many other elements well reflected the low V concentration in ores from the mining district. A low concentration of V was therefore a good indicator of mining in the study area and the sedimentation of mined materials. The high concentration of V in sediments represents the sedimentation of materials coming from other sources than mines. The concentration of V in sediments was therefore a very useful tool for interpreting the sedimentary history of the study area.

CONCLUSIONS

We demonstrated that it is possible to relate concentrations of trace elements in alluvial sediments with historic records of mining production. Therefore concentrations of trace elements can be used for the dating of alluvial sediments in mining areas, although the accuracy and correctness of dating according to trace elements highly depend on initial independent dating, sedimentation history and the quality of written sources.

Among twelve elements analysed, eight (Be, Co, Cr, Cu, Hg, Pb, V and Zn) helped identify the human mining impact on the sedimentary record. Absolute values of element concentration are not determinative for the analysis. Different sources of sediments were distinguished by V concentrations. Trace elements can be thus successfully used as stratigraphic markers in archaeological studies.

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