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Mineralogy, petrography and geochemistry of sediments used in pollutant sorption experiments

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Abstract. Sediments from the bottom of the Horní Bečva Reservoir (HBS 2, HBS 6, HBV 4) contain identical mineral phases and vary in clay contents (HBS 2 – 1 wt%, HBV 4 – 18.5 wt%). With the increasing clay amount, the contents of Fe-oxyhydroxides and organic matter also increase. Clay minerals in lake bottom sediments are represented mainly by kaolinite (K), chlorite (C) and mixed-layer illite/smectite (I/S) with expandability of ca. 40 % S [(K > C) = I/S]. Neogene argillaceous and silty sandstone from Staré Město (SMA) comprises the highest amount of clay fraction (31 wt%). Phyllosilicates, especially smectite and kaolinite (S = K), surround the rock-forming minerals. H (Hevlín) and Žd (Ždánice) Paleogene and Neogene silty sandstones contain carbonate cement (dolomite > calcite). The dominant clay mineral in both sandstones is I/S with expandability of ca. 95 % S. The H sample contains also a considerable amount of chlorite and discrete illite (S > C > Id). Žd sample comprises less kaolinite plus chlorite and discrete illite [S > (K > C) > Id]. The soil horizon from Lelekovice (Le 4) is typical in the presence of carbonates, weathered amphiboles and epidote minerals (derived from a metabasite source), in higher contents of clay fraction (15 wt%), organic matter and Fe-oxyhydroxides. The main clay mineral in sample Le 4 is mixed-layer illite/smectite with expandability around 65 % S and kaolinite (I/S > K). The soil horizon from Červenohorské sedlo Saddle (SČS 5) is typical in the absence of feldspars, in the high content of muscovite and biotite (derived from mica schists) and in the highest amount of organic matter and Fe-oxyhydroxides. Clay minerals of this sample include mostly discrete illite; chlorite and kaolinite are less abundant (Id > C > K).

Key words: soil, lake bottom sediment, sandstone, clay minerals, smectite, illite, kaolinite, chlorite, carbonate cement

Introduction

Migration of chemical elements under surficial conditions represents an important process affecting the quality of the environment. The behaviour of elements, especially of heavy metals, is controlled by sorption properties of soils and unconsolidated sediments. Sorption properties of soils depend on their mineral composition, the grain-size distribution, and the amount of organic matter. Within the examination of sorption properties of soils and unconsolidated sediments, 8 sample collections were chosen (see Fig. 1), typical for larger areas in Moravia. These sets of samples were subjected to physical, chemical and mineralogical characterisations based on bulk chemical analyses, XRD analyses, microscopic description and grain-size measurements. From these sets, 8 samples were selected for sorption on-column experiments, especially for a sorption of heavy metals (Cd, Cu). For the interpretation of sorption behaviour of samples and their potential modifications during sorption experiments, it was necessary to know their detailed mineralogical composition.

Methodology

Polished thin sections were made at the Faculty of Science of Masaryk University Brno, Department of Mineralogy, Petrography and Geochemistry. The collected samples were strengthened with resin in advance.

Chemical and XRD bulk-rock analyses were performed at MU, Faculty of Science. Free water ($-H_2O$) was determined by drying at 110 °C; water bonded in minerals ($+H_2O$) was analysed employing the Penfield method. SiO_2 and S were determined gravimetrically; TiO_2, Fe_2O_3 and P_2O_5 photometrically; Al_2O_3 and CaO volumetrically by titration with K III. and FeO by titration with $K_2Cr_2O_7$. MnO, MgO, K_2O and Na_2O were analysed employing AAS; Li_2O using flame ionisation photometry and CO_2 by absorption analysis. Organic compounds were roughly determined through calculation – as a mass shrinkage caused by annealing, minus water, CO_2 and S.

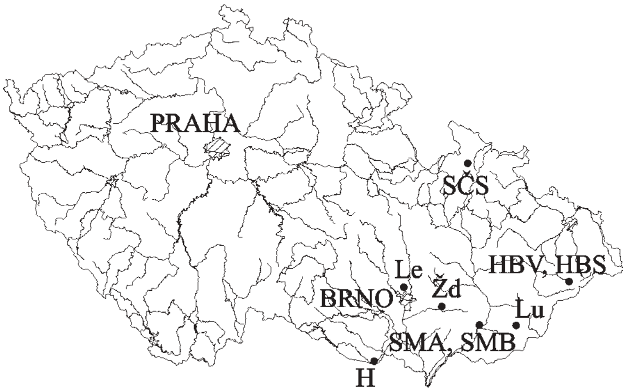

Fig. 1. Topographical map of sample collection sites: HBV, HBS (Horní Bečva Reservoir outlet and its centre), Le (Lelekovice), SČS (Červenohorské sedlo Saddle), Lu (Luhačovice), Žd (Ždánice), SMA and SMB (Staré Město – Velehrad A and B), H (Hevlín).

XRD bulk-rock analyses were performed using the STOE StadiP diffractometer in transmission mode, primary Ge (111) monochromator, $CoK_{\alpha1}$ radiation and PSD detector. The obtained records were evaluated using Win XPOW program (ver. 1.06; STOE Co.) and PDF database.

X-ray records of a < 2 µm fraction were accomplished on an X-ray powder diffractometer X'Pert System Philips with graphite monochromator, 0.05 step 2theta, 2 mm / 0.1 m^2 diaphragm, 40 kV voltage, and 40 mA current. $CuK_{\alpha 1}$ radiation was used. The acquired X-ray diffraction records were evaluated by comparing with modelled records (program NEWMOD, Reynolds 1985). The < 2 µm fraction was separated by sedimentation in distilled water and concentrated by centrifugation and drying. Before the actual separation, the samples in distilled water were disintegrated in the ultrasound. Prior to the collection of the 2 µm fraction, the SČS sample was also purified by removal of organic matter (procedure of Jackson 1974, modified by Šucha et al. 1991). The sample was first placed into sodium acetate buffer (50 ml for 5 g of sample), and 30% H_2O_2 was then added (5 ml for 5 g of sample) and the mixture was heated up to 70 °C (the liquid must not boil) with permanent agitation, rinsed three times with distilled water and centrifuged.

The separated < 2 µm fraction was used for recordings of IR spectra on Perkin Elmer 783 spectrophotometer, operated by a common PC program SpectacleIR (LabControl Co.), at the Czech Geological Survey in Brno. Spectra were recorded in the range of wavelengths of 4000 to 200 cm^{-1}. Pellets 12 mm in diameter were prepared using a standard procedure of pressing with KBr.

Sample surfaces and chemical compositions of minerals were studied using CamScan 4 scanning electron microscope (CGS) equipped with Isis EDX (energy dispersion analyser, Link Co.). Quantitative silicate analyses were accomplished at 15 kV voltage and 2.5 nA current.

Sample characteristics

Four layers of Recent lake bottom sediments (HBV) were collected at the outlet of a drained Horní Bečva Reservoir. All layers can be classified as argillaceous silt. The uppermost layers of all sets of samples were labelled with No. 1, the deepest layers were given the highest numbers. Layer HBV 4 (depth 19–26 cm), selected for sorption experiments, contains the highest amount of clay fraction of lake bottom sediments from HBV and HBS sites (18.5 wt%) and a high amount of organic matter (C_{org} = 3.54 wt%, Bezděk et al. 2002).

In the middle of the Horní Bečva Reservoir, 10 layers (HBS) of Recent lake bottom sediments were collected. These layers differ in grain size. Of these, layers HBS 2 and HBS 6 were chosen for sorption experiments. Sample HBS 2 is typical in its low content of clay fraction (1 wt%) and a low amount of organic matter. Sample HBS 6 contains 8.5 wt% of clay fraction. Layer HBS 2 (depth 12–21 cm) was classified as medium-grained gravelly sand, layer HBS 6 (depth 53–57 cm) as silty fine-grained sand.

Fine-grained silty material is deposited close to the outlet (HBV) while alternating coarser and fine-grained sediments with gravelly sand and argillaceous silt are typical for the basin centre (HBS).

All the lake bottom sediments at the HBV and HBS sites have the same mineral composition. Microscopically, the main mineral is quartz, less abundant are plagioclases, microcline and orthoclase, chlorites (chloritised biotite), muscovite and glauconite (less than 1 wt%). Argillaceous parts of sediments contain concentric oxidation rims, produced by coagulation of Fe-oxyhydroxides during drying.

Five layers of a Recent soil profile were collected in Lelekovice (Le). Sorption experiments were carried out with soil horizon Le 4 (silty loam with coarse-grained sand and gravel at a depth of 27–37 cm). Within the Lelekovice locality, horizon LE 4 is characterised by the highest content of clay fraction (15 wt%) and CO_2 content (3.24 wt%; other layers contained 2.7–3.2 wt% CO_2). The Lelekovice soil profile is characterised by the presence of carbonates, weathered amphiboles of actinolite composition and weathered epidote minerals (originating from the metabasite) and also by a higher content of clay fraction (over 10 wt%) and Fe-oxyhydroxides (5.6–6.3 mol.% goethite). Furthermore, most common quartz, plagioclases, K-feldspars and chlorites were microscopically identified in these samples.

Five layers of soil profile were collected at Červenohorské sedlo Saddle (SČS). Horizon SČS 5 – sandy loam with gravel (depth 32–42 cm) – was selected as a sorbent. This horizon has the lowest content of organic matter of all SČS samples (C_{org} = 5.4 wt%, Bezděk et al. 2002) and the highest amount of goethite (7.5 wt%; other layers contained ca. 2 to 4 wt%). The SČS soil profile is typical in the negligible amount of feldspars and high contents of muscovite, biotite and chloritised biotite (derived from mica schists). The prior mineral is quartz; very rare sericitised plagioclase occurs as well. This locality contains the highest amount of detrital organic matter and Fe-oxyhydroxides.

A poorly lithified silty fine-grained sandstone from Ždánice (Žd) belongs to the Ždánice–Hustopeče Formation of the outer flysch belt of the West Carpathians (Upper Oligocene to Lower Miocene). Sample Žd consists mostly of quartz fragments; less common are fragments of K-feldspars, plagioclases and muscovite, surrounded by carbonate cement. This sample contains the highest amount of carbonates (according to the XRD analysis, dolomite > calcite) and was therefore chosen as a sorbent.

The Pannonian of the Vienna Basin is represented by two samples of poorly lithified argillaceous and silty fine-grained sandstones from Staré Město – Velehrad. The sample labelled as SMA was collected on the bottom of a sump reservoir; SMB sample comes from its bank. These samples contain high amounts of phyllosilicates, which surround the rock-forming minerals (quartz, K-feldspars and plagioclases). Muscovite and chlorites were found using optical microscope while kaolinite and smectite are the major clay minerals proved by XRD analysis of fraction under 2 µm and EDS analysis. The SMA sample was selected for sorption experiments due to the highest clay fraction content (31 wt%).

Poorly lithified Eggenburgian silty fine-grained sandstone from Hevlín (H) was collected in the new brickyard. This sample was selected as a sorbent because of its low clay fraction content (2.5 wt%) and higher amount of carbonates (5.9 wt% CO_2). Quartz, muscovite, chlorites, plagioclases, microcline and K-feldspars are surrounded by a small amount of carbonate cement. The sample has a marked stratification with ordered micas and bands of organic matter.

Clay mineral alterations during sorption experiments

The specific surface area of quartz and clay minerals is very different; the behaviour of clay minerals in various environments is diverse. Unlike kaolinite, chlorite is very sensitive to low pH values. For sorption characteristics of sediments is very important to know clay composition (Almon and Davies 1981).

Clay minerals were identified in samples before and after sorption experiments. X-ray diffraction patterns were analysed on air-dried and glycolated oriented samples of the clay-sized fraction < 2 μm. The fraction of < 2 μm (clay fraction) contained mostly clay minerals; very small amounts of quartz, feldspars or carbonates were also observed in SMA and H samples.

X-ray patterns of lake bottom sediments from the Horní Bečva Reservoir are virtually identical. HBV 4, HBS 2 (see Fig. 2) and HBS 6 contain mostly kaolinite and chlorite (K > C), approximately the same amounts or less mixed-layer illite/smectite (I/S) with average expandability between 35 and 42 % S, and a small amount of discrete illite (Id).

Le 4 (Lelekovice) soil horizon comprises mostly I/S with expandability of 60 to 70 % S, lower amount of kaolinite and a negligible amount of Id (see Fig. 2). Le 4k sample (after experiments) contained also a small amount of chlorite. The shape of kaolinite peak indicated the presence of chlorite even in Le 4 sample, although it could not be proved. The chlorite (C) content probably did not increase throughout the experiments. Chlorite very likely originated by chloritisation of amphiboles in soil. The C peak (d = 4.75 Å) was thus more distinct in the sample after experiments due to a slight sample inhomogenity or a reduced I/S amount after the experiments.

Discrete illite (Id) is the dominant clay mineral in the SČS 5 soil horizon (see Fig. 2); chlorite and kaolinite (C > K) are less abundant. This sample shows also a small content of I/S with expandability around 30 % S. I/S content increased significantly after the experiments (expandability of ca. 40 % S) and the K/C ratio also changed. Samples SČS 5 and SČS 5k (after experiments) were treated by H_2O_2 (modified method of Jackson 1974) for removal of organic matter. Due to the removal of organic matter, lepidocrocite was identified, probably being produced from released Fe. Amorphous Fe-oxyhydroxides formed a gel, which crystallised on oriented preparations.

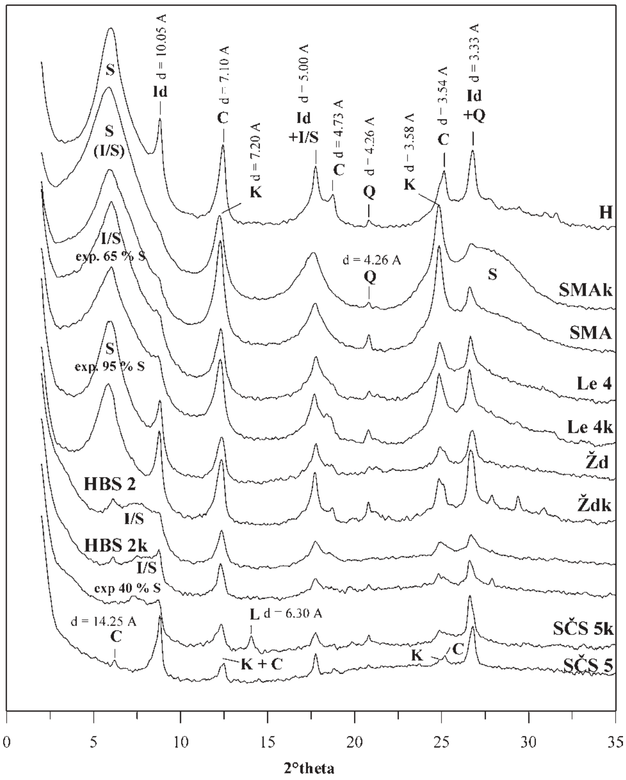

Fig. 2. X-ray diffraction patterns of air-dried oriented samples of the clay-sized fraction of < 2 μm (SMAk, Le 4k, HBS 2k, SČS 5k and Ždk are samples after sorption experiments), K = kaolinite, Id = discrete illite, I/S = mixed-layer illite/smectite, S = smectite, L = lepidocrocite, Q = quartz.

SMA sandstone sample (see Fig. 2) contains approximately equal amounts of kaolinite and smectite (expandability I/S ca 90 % S) and a negligible amount of Id.

Almost pure smectite (expandability between 95 and 100 % S) and chlorite were identified in H sample (see Fig. 2). A small amount of kaolinite was also detected (chlorite peak at d = 3.54 Å was slightly deformed). Kaolinite was later proved by the IR analysis.

Clay fraction of the Žd sandstone (see Fig. 2) consists mostly of smectite (expandability 95 to 100 % S). Kaolinite, chlorite (K > C) and Id are present in smaller amounts.

It can be generally stated that the content of mixed-layer illite/smectite in lake bottom sediments (HBS 2, HBS 4, HBS 6, HBV 4), in soil horizon Le 4 and sandstones Žd and H decreased after the experiments. The amount of chlorite (C) in HBS 2 sample was also reduced after experiments. On the other hand, the content of illite/smectite in the SČS 5 soil horizon and SMA sandstone increased.

A pp index (proportion of peak area) was introduced for a better comparison of tested samples. This index can be calculated as the ratio between the K + C (d = 7.15 Å) peak area and I/S (d = ca. 12 Å) peak area. These peak areas were determined using the ClayLab program (Mystkowski 2000).

By plotting the expandability against the pp index (see Fig. 3), all samples can be divided into three groups. Sandstones in group I (SMA, H, Žd) contain mostly smectite

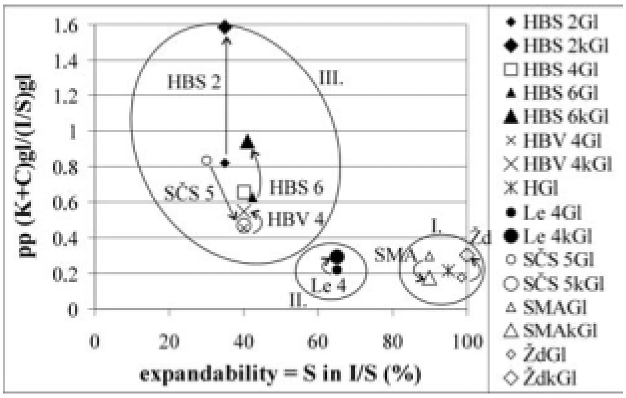

Fig. 3. Dependence of expandability of mixed-layer illite/smectite on the pp index: samples of group I are rich in smectite with expandability around 90 % S, sample of group II is rich in I/S with expandability of 65 % S. Samples in group III contain higher amounts of K + C and have the expandability of I/S around 40 % S. The arrows indicate samples after experiments.

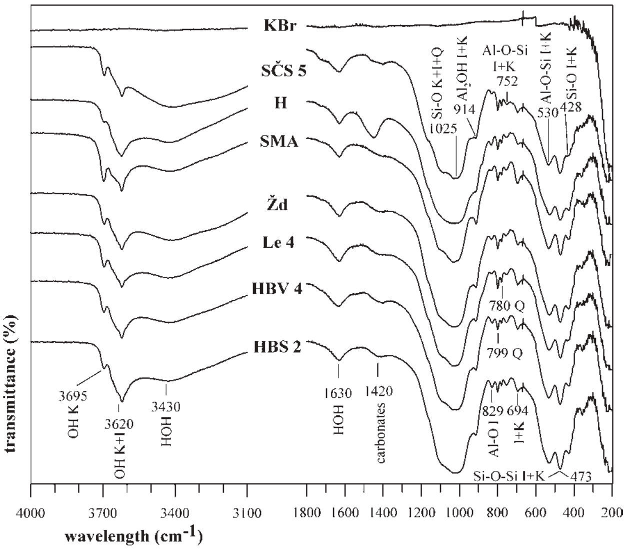

Fig. 4. Infrared spectroscopy of samples of the clay-sized fraction < 2 mm, K = kaolinite, I = discrete illite + mixed-layer illite/smectite + smectite, Q = quartz.

(expandability ca. 90 % S). Soil horizon Le 4 in group II comprises mostly smectite/illite (expandability ca 65 % S). Group III is formed by locally transported lake bottom sediments (HBV 4, HBS 2, HBS 4, HBS 6) and soil horizon SČS 5 with higher contents of kaolinite, chlorite and I/S with expandability of ca. 40 % S.

Infrared analyses

Kaolinite and chlorite have different sorption characteristics and chemical reactivity (Almon and Davies 1981). Infrared spectroscopy was used for their comparison. Kaolinite was identified in all samples, and a small amount of kaolinite was proved in the sandstone from Hevlín (see Fig. 4). Illite + smectite + mixed-layer illite/smectite were also detected in all samples. The proportion of illite and smectite can be judged from the absorption band of illite (829 cm^{-1}).

Discussion and conclusions

Bulk chemical analyses were recalculated to real minerals (see Tab. 1) in accordance with the results of bulk-rock X-ray diffraction analyses, X-ray analyses of < 2 μm fractions, infrared spectroscopy, EDS analyses, microscopic description and grain-size distribution.

The clay minerals were calculated first. The amounts of clay minerals were known from grain-size distribution. Composition and ratios of clay minerals were approximately determined on the basis of X-ray diffraction analyses of clay fraction. The proportion of chlorites was not preserved (within the scope of clay minerals) because chlorites are mostly formed by chloritised biotite, with aggregates larger than 2 μm.

Then the carbonates were calculated from the results of the bulk-rock X-ray diffraction analyses. Finally, micas and feldspars were calculated on the basis of a microscopic study, and the bulk-rock X-ray diffraction analyses.

The presence of amphibole of actinolite composition in the Le 4 soil horizon was confirmed by EDS, and microscopic observations.

The interpretation of sorption experiments should consider factors such as pH, porosity, the content of Fe-oxyhydroxides, carbonates and also organic matter, on which heavy metals can adsorb (Zeman et al. 2002).

Higher amounts of carbonates were found in Žd and H sandstones and in Le 4 soil profile (see Fig. 5).

The highest content of Fe-oxyhydroxides (goethite) was detected in the SČS 5 soil horizon (see Fig. 5), smaller amounts were identified in the Le 4 soil horizon and HBV 4 lake bottom sediment.

The highest amount of organic carbon (Müller 1997, Bezděk et al. 2002) was recorded in the SČS 5 soil horizon, and higher amount was found in the HBV 4 lake bottom sediment and in the Le 4 soil horizon (see Fig. 5).

SMA sandstone contains the highest amount of clay fraction and shows the lowest porosity (20 vol%, Müller 1997, Zeman et al. 2002).

The pH of all aqueous solutions ranges from 7 to 8 (Müller 1996, Zeman et al. 2002) with the only exception of SČS 5 (pH = 4, Kruml 2002). This sample has the highest amount of organic matter.

Sediment sorption properties depend also on the content of clay fraction, thus on the amount of clay minerals. The highest content of clay fraction was found in SMA (31 wt%). The amount of clay further decreases in the succession: HBV 4 (18.5 wt%) > Le 4 (11 wt%) > HBS 6 and Žd (5 and 8.5 wt%). The lowest clay contents (less than 3.5 wt%) were determined in SČS 5, HBS 2 and H samples (see Fig. 5).

Sorption properties of sediments depend on the composition of clay minerals, especially illite and smectite. The most expansive samples include SMA, Žd and H. The prior mineral in these samples is smectite with expandability around 95 % S. Somewhat less expansive is Le 4, which comprises especially mixed-layer illite/smectite with expandability of 65 % S. Discrete illite is dominant in the

Tab. 1. Results of the recalculation of bulk chemical analyses into weight percentages of mineral phases

	HBV 4	HBS 2	HBS 6	Le 4	SČS 5	Žd	SMA	H
	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)	(wt%)
Apatite	0.55	0.14	0.22	0.26	0.60	0.12	0.14	0.36
Rutile	0.78	0.37	0.48	0.72	0.93	0.25	0.46	0.41
Goethite	4.57	1.84	2.66	4.70	7.49	0.77	2.21	2.05
Dolomite	0	0	0	3.30	0	14.99	0	7.79
Calcite	0.39	3.67	0.31	3.55	0.73	6.27	0.09	4.84
Albite	5.39	3.02	3.62	10.00	0.01	11.57	0	8.40
Pyrolusite	0.10	0.05	0.06	0.14	0.06	0.05	0.01	0.06
Pyrite	0.36	0.09	0.21	0	0.11	0	0	0.40
Mg-chlorites	3.45	1.79	2.61	0.12	0.19	0.29	0.94	2.68
Biotite	0	0	0	0	10.30	0	0	0
Actinolite	0	0	0	14.19	0	0	0	0
Fe-chlorites	3.63	2.17	2.92	0.22	0.73	1.36	0.26	4.40
Anortite	1.68	2.04	4.07	5.80	0.29	3.55	1.59	4.65
K-feldspar	3.43	5.97	4.25	3.83	0	8.79	5.89	4.04
Smectite	3.48	0.22	2.23	7.41	0.37	3.05	8.29	1.55
Muscovite	9.31	3.99	8.10	4.35	10.74	0.44	7.74	10.42
Paragonite	0	3.06	5.05	0	10.15	0	0	4.62
Illite	5.64	0.46	3.27	3.94	2.23	1.42	0.85	0.42
Diaspore	8.90	4.47	2.89	0	4.04	0	0	2.48
Kaolinite	6.85	0.40	3.22	5.80	0.41	1.33	9.54	0.12
Amorphous SiO_2	11.01	1.55	4.07	4.19	13.77	0,00	0.19	1.95
Quartz	30.49	67.99	49.75	27.47	36.86	45.75	61.79	38.36
Total	100	100	100	100	100	100	100	100

SČS 5 soil horizon and, to a certain degree, also in H and Žd sandstones. Chlorite is an important clay mineral in H sandstone. Kaolinite is the major clay mineral of SMA sandstone and also occurs in lake bottom sedimens (HBV 4, HBS 2, HBS 6) and Žd and Le 4 samples.

Lake bottom sediments (HBS 2, HBS 6 and HBV 4) are virtually free of carbonates and show a positive correlation of clay fraction content, organic matter and Fe-oxyhydroxides (goethite). This means that the sample containing the highest amount of clay (HBV 4, 18.5 wt%) contains the highest amounts of organic matter (C_{org} = 3.5 wt%, Bezděk et al. 2002) and Fe-oxyhydroxides (4.6 wt% goethite). Clay minerals are represented by kaolinite, chlorite and I/S with expandability around 40 % S.

SMA sandstone contains the highest amount of clay (31 wt%) and has therefore the lowest porosity (20 vol%, Müller 1997, Zeman et al. 2002). Žd and H sandstones encompass carbonates (dolomite > calcite: Žd 21 wt%,

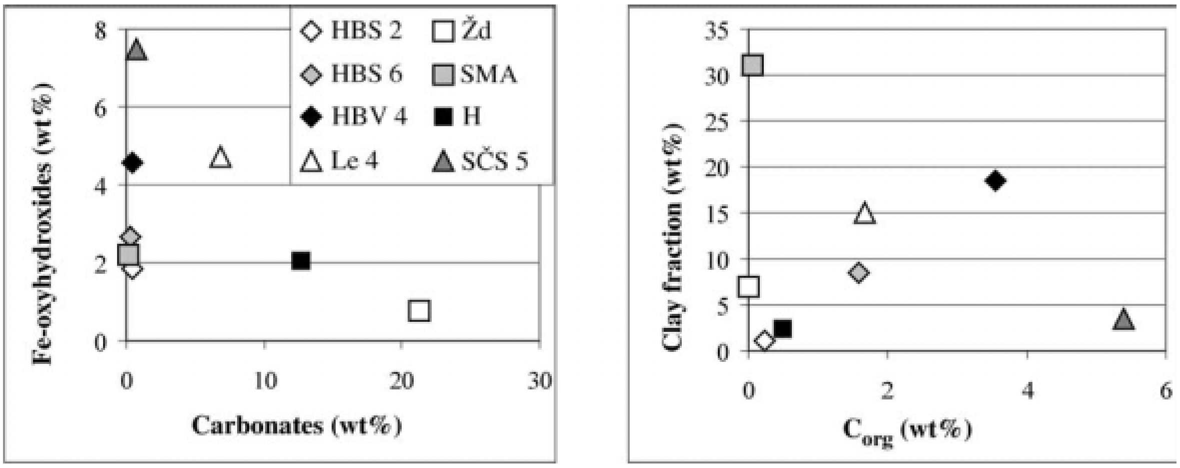

Fig. 5. Contents of Fe-oxyhydroxides (wt%), carbonates (wt%), clay fraction (wt%) and C_{org} (wt%, Müller 1997 and Bezděk et al. 2002) in samples used for sorption experiments.

H 12 wt%). Sandstones are almost free of organic matter and their prior mineral is smectite.

Le 4 soil horizon contains around 7 wt% of calcite and dolomite. This sample is also characterised by a considerable content of Fe-oxyhydroxides (4.7 wt% goethite) and clay content (15 wt%). The prior clay mineral is mixed-layer illite/smectite with expandability of around 65 % S.

SČS 5 soil horizon is virtually void of carbonates. It has the highest content of organic matter (C_{org} = 5.4 wt%, Bezděk et al. 2002) and Fe-oxyhydroxides (7.5 wt% goethite) of all sorbents. Aqueous solution of this sample is characterised by the lowest pH (pH = 4). Its prior clay mineral is discrete illite.

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