Pol. J. Environ. Stud. Vol. 23, No. 2 (2014), 357-362

Original Research

Trends in Trace Element Concentrations in Holocene Bottom Sediments of a Lake Wielki Staw in the Karkonosze Mountains

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Received: 17 October 2013
Accepted: 17 February 2014

Abstract

Lake sediments and peat bogs are "natural archives" of long-term changes in the quality of the natural environment at local and regional scales. However, due to technical difficulties the sediments of mountain lakes are rarely subject of geochemical analyses. Also, the sediments of Wielki Staw (1,225 m a.s.l.), the largest lake in the Karkonosze and entire Sudeten Mountains, have never been analyzed for trace elements. The aim of this study was to determine total content of Pb, Cu, Zn, Cd, Ni, Cr, Mn, and their vertical variations on the background of basic physical and chemical properties of sediments of Wielki Staw. The 11 m-thick sediment profile, representing the entire Holocene period, was analyzed after being divided into 55 sections, each 20 cm thick. Bottom sediments are characterized by variable particle-size distribution (prevailing loamy textures), strongly acidic pH, and high content of organic matter and Fe (2,730-10,100 $mg \cdot kg^{-1}$), but low Ca (70-688 $mg \cdot kg^{-1}$) and Mg (567-2,450 $mg \cdot kg^{-1}$). Mean concentrations of trace elements are lower than in the sediments of Polish lowland lakes and close to the contents in mountain peatbogs and soils, and are generally correlated with the organic matter and fine earths. Pb, Mn, Zn, and Cu concentrations (4.65-81.8, 85.4-346, 11.9-50.3, and 3.05-19.5 $mg \cdot kg^{-1}$, respectively) are generally higher in the oldest (pre- and boreal) sediments and lower in younger sediments (postatlantic). Cr, Ni, and Cd (0.55-10.7, 1.40-7.35, and 0.25-1.15 $mg \cdot kg^{-1}$, respectively) contents do not express any trend throughout the profile. Increases of Cu, Zn, and especially Pb concentrations in the uppermost layer of sediments (down to 1.2-1.5 m) is not justified by the change of the physicochemical properties of sediments and may only be explained with atmosphere anthropogenic contamination in the Sudeten Mountains that began at the end of the first millennium AD and accelerated during the last three-four centuries.

Keywords: bottom sediments, heavy metals, organic matter, air pollution, principal component analysis

Introduction

Paleolimnological research is often used as a tool to determine short- and long-term climatic changes and the occurrence of specific environmental conditions in the past, as well as to document the human impact on the environment. Analysis of sediments allows tracking the trends of changes over the decades to tens of thousands of years back [1, 2]. Current development of investigation methods and analytical techniques also allows using bottom sediments in predicting future changes in the environment [3]. However, it is not possible to predict the development trends for existing lakes without insight into their history by determining

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the rate of the transformation taking place previously, due to both climate change and anthropogenic impacts. It has been shown that the transformation of the environment has significantly accelerated in recent centuries [4-6]. Identification of trends for lake sediments, similar to that done for peatland ecosystems, e.g. by Tobolski [7], is of great importance for understanding the current functioning of lakes [8].

In fact, peatlands are the most often used sites of geochemical reconstruction in mountain areas [9], as the geochemical data in the peat are often well correlated with palinological analysis, the other excellent source of information on environmental conditions. Similar data for mountain lake sediments don't exist or are very scarce and fragmented. Few works on lakes located in the Tatra and Karkonosze Mountains, published by Wicik [10, 11], Więckowski [12], and Chmal and Traczyk [13], related mainly to the morphology and sedimentological features of sediments, while geochemical issues were treated marginally (except for the contents of Si, Fe, and Al).

Lake beds and their immediate surroundings in the Karkonosze Mts. are built of "acidic" granites containing only small amounts of alkaline components that, combined with harsh climatic conditions, specifically influence water and sediment chemistry [11, 14], including heavy metal content and activity [15]. The aim of this study was to determine the levels of trace element (Pb, Cu, Zn, Cd, Ni, Cr, Mn) total contents, their vertical variation, and probable relations to the basic physical and chemical properties of sediments of Lake Wielki Staw in the Karkonosze Mountains to indicate both the metal's geochemical background and human influences imprinted in the mountain lake sediments.

Methods and Materials

Wielki Staw is located in Karkonosze Mountains National Park in the western Sudeten Mountains, in a postglacial circus on the northwestern slope of Mt. Smogornia, at 1,225 m a.s.l. (in the transitional zone between the spruce forest and subalpine climatic and vegetation belts). The lake has the shape of a tub elongated at the W-E axis. Lake length is 646 m with maximum and average width of 183 m and 138 m, respectively. Area of the water body is 8.32 hectares, maximum depth 24.4 m, and the average depth 9.5 m. Lake depth is asymmetrical; the western part is clearly shallower than the eastern one. Wielki Staw is supplied mainly by rainwater and snowmelt directly, or by seasonal streams flowing from the rubble and weathering granite covering the plateau of the main ridge of the Karkonosze Mountains [16]. The catchment area (about 65 hectares) is dominated by Albic Podzols and Stagnic Podzols on the plateau, and Hyperskeletic Leptosols and Lithic Leptosols in the direct surroundings of the lake [17]. The area is presently under the influence of suboceanic climate with significant influences of continental air masses. Mean annual air temperature at 1,300 m a.s.l. is about 2.0ºC, mean annual precipitation is 1,500 mm, and the mean annual snow cover length is 170 days [18].

The soil sampling was conducted from the ice surface by Więckowski and team [12] using the geological probe in winter 1983. Initial testing indicated the sediment thickness varying in the range from 3 to 11 m. Three sediment cores were excavated, but only the longest, reaching granite regolith under the undisturbed lake sediments, was classified for laboratory analyses. The core was 11 m thick starting at a depth of 18.5 m below the water table and was collected from the eastern, deeper part of Wielki Staw. The core was divided into 20 cm-long sections (55 samples), which were dried and stored in double polyethylene bags, packed together in an additional polyethylene bag and carton box. The drying conditions are not known; however, sample weight loss at 105ºC checked in 2012 was less than 1%, indicating oven drying before storage.

Two samples of wood were collected within the core that allowed sediment dating by C-14 method. The first piece of wood was found at a depth 6.7 m and dated to 5,400 years BC, and the other was at a depth 9.8 m and dated to 7,880 years BC [12]. However, due to the sediment liquidity allowing wood subsidence, the primary depth of wood sedimentation was estimated to 5.4 and 9.5 m. Based on the age of woods, the age of the oldest deposits was defined on at least 10,000 years and the sedimentation rate was calculated to be 1.7 mm per year in the lower part, and 1 mm per year in the upper part of the core.

Physicochemical and chemical analyzes of the sediments were conducted in 2012 in the laboratories of the Institute of Soil Science and Environmental Protection, Wrocław University of Environmental and Life Sciences[1]. In the samples we determined: particle-size distribution by sieving (sand fraction) and by hydrometer method (silt and clay fractions), pH in distilled water (soil to solution ratio of 1:2.5) – potentiometrically, total organic carbon (TOC) by the dry combustion method with CO_2 absorption (Ströhlein, CS-mat 5500), and total concentrations of Zn, Cu, Pb, Cr, Cd, Ni, Mn, Fe, Mg, Ca, and K by atomic absorption spectrophotometry (AAS) or inductively coupled plasma (ICP) in the extracts obtained by sample digestion in 70% perchloric acid (soil to acid ratio 1:10). The quality of determinations has been monitored using soil reference materials (SRM 2709, SRM 2711, RTH 912, RTH 953) with certified total concentration of trace elements being analyzed.

Basic statistical measurements, e.g. arithmetic means, medians, and standard deviations, have been extended by multivariate analysis (principal component analysis, PCA) supported by Pearson's correlations to understand the relationships between variables. All statistical calculations were performed using the Statistica 9.0 (StatSoft Inc.) software package.

Results and Discussion

Bottom sediments of Lake Wielki Staw were classified by Więckowski [12] to clay-detritus gyttja with numerous

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[1] Sediment samples for analysis were provided by Dr. A. Wicik from the University of Warsaw, whom the authors sincerely thank.

Table 1. Physicochemical properties and element concentrations in the bottom sediments of Lake Wielki Staw (n=55).

Variable	Minimum	Maximum	Median	Mean	Standard deviation
TOC [%]	0.23	30.2	7.89	7.81	4.38
pH	4.0	5.6	4.6	4.6	0.26
Clay [%]	4	26	17	16	7.2
Silt [%]	1	35	24	23	7.8
Ca [mg·kg^{-1}]	70.0	688	463	455	147
Mg [mg·kg^{-1}]	567	2450	1530	1520	344
K [mg·kg^{-1}]	848	3380	2620	2520	476
Fe [mg·kg^{-1}]	2730	10100	8230	7920	1550
Mn [mg·kg^{-1}]	85.4	346	237	244	57.2
Zn [mg·kg^{-1}]	11.9	50.3	26.2	29.3	8.9
Pb [mg·kg^{-1}]	4.65	81.8	36.9	37.5	13.5
Cu [mg·kg^{-1}]	3.05	19.5	7.40	8.95	4.43
Cr [mg·kg^{-1}]	0.55	10.7	6.90	6.85	2.19
Ni [mg·kg^{-1}]	1.40	7.35	5.80	5.57	1.29
Cd [mg·kg^{-1}]	0.25	1.15	0.90	0.82	0.19

(up to 90) thin sandy strata. The samples for analysis were, however, collected as regular 20-cm long sections and always involve both more organic and more sandy strata. Total organic carbon (TOC) content ranged from 0.23 to 30%, mean 7.82% (Table 1). TOC reached the level 6-7% just in the deepest section of deposits, and the highest TOC content was recorded at a depth of 6.7 m, where the probe cut buried a tree stump. In the upper sediment layers a clear decreasing TOC trend was observed. Sediments did not contain carbonates, therefore their low pH varied in a relatively narrow range between 4.0 and 5.6 (mean 4.6). The loamy textures (sandy loam and sandy clay loam [19]) dominated throughout the core with an average content of clay and silt fraction of 16% and 23%, respectively. There was no clear gradient in sediment texture throughout the core. The clay content was higher (20-25%) in the top and bottom sections and significantly lower (7-11%) in the central part of the core. Lower clay content in this part was accompanied by significantly higher content of silt - up to 35%. As stated before, the sand strata were very thin (0.5-2 mm); the only exception was a thick homogeneous sandy layer, with the silt and clay contents not exceeding 1-4%, present at the depth 8.6-9.2 m. This sand layer is a record of the sudden but short-term climate deterioration at around 8,000 years BC, recorded in the lake sediments and bogs all over Europe [20]. Climate fluctuations in the Karkonosze Mountains entailed increased soil erosion of weathering products and the delivery of large amounts of sand material to the lake [13]. This change was also reflected in the other properties of sand deposits. TOC content was reduced here to 0.27% and the pH reached a value of 5.6. TOC in the sediments was positively correlated with the amount of silt (strongly) and clay (poorly), resulting in a clear negative relationship with content of the sand fraction (Table 2). The pH value was strongly negatively correlated with the fine fraction (clay and silt) and TOC.

The concentration of Mg, K, and in particular of Ca in sediments of Wielki Staw was even tenfold lower than in the sediments of the Tatra lakes and postglacial lakes in the Polish lowland [5, 21, 22]. The highest content of K and Mg was observed in the lower layers, while the lowest – in a thick sandy stratum (Fig. 1A). From a depth of 8.5 m up to the top of sediments, Mg and K content was stable at the levels of 1,600 and 2,600 mg·kg^{-1}, respectively. Ca content was much more variable, but clearly higher values were recorded in the bottom (> 500 mg·kg^{-1}) than in the upper parts of deposits (about 400 mg·kg^{-1}), excluding low content in the sandy layer. Ca and Mg were not dependent on the organic matter (Table 2), but were positively correlated (especially Mg) with the fine mineral fractions (silt and clay). The negative correlation between Ca and Mg contents and pH proves the dependence of sediment pH of the organic acids mainly. Ca and Mg occur in the sediments in the primary, presumably weakly weathered minerals and do not influence the sediment reaction. This statement is in line with very low Ca ion concentrations and low water hardness in the Karkonosze Mts. [28].

Fe concentration (excluding sand layer) ranged from 5,540 to 10,100 mg·kg^{-1} (mean – 7,920 mg·kg^{-1}) and was the highest in the sediment's lowest part. A noticeable decreasing trend occurred from the bottom up to a depth of 5-5.5 m, above which started a weak increasing trend to the top of deposits. Fe correlated with the fine mineral fractions, but the strong correlation between Fe and TOC indicates

Table 2. Coefficients of Pearson's correlation.

	TOC	pH	Ca	Mg	K	Fe	Mn	Pb	Zn	Cu	Cr	Cd	Ni
TOC		-0.57*	0.11	0.14	0.46*	0.41*	0.49*	0.37*	0.06	-0.18	0.57*	0.06	0.64*
clay	0.27	-0.45*	0.35*	0.37*	0.34*	0.56*	0.29*	0.01	0.37*	0.35*	0.17	0.57*	0.20
silt	0.62*	-0.62*	0.27	0.56*	0.65*	0.50*	0.79*	0.35*	0.43*	0.34*	0.43*	0.08	0.58*
sand	-0.63*	0.75*	-0.42*	-0.65*	-0.70*	-0.73*	-0.76*	-0.25	-0.55*	-0.48*	-0.43*	-0.44*	-0.56*
pH			-0.50*	-0.75*	-0.79*	-0.79*	-0.64*	-0.40*	-0.45*	-0.44*	-0.49*	-0.44*	-0.53*
Ca				0.69*	0.55*	0.56*	0.49*	0.37*	0.61*	0.57*	0.27	0.39*	0.15
Mg					0.86*	0.83*	0.75*	0.45*	0.76*	0.77*	0.49*	0.53*	0.35*
K						0.92*	0.73*	0.60*	0.60*	0.49*	0.73*	0.50*	0.66*
Fe							0.63*	0.57*	0.64*	0.49*	0.70*	0.63*	0.62*
Mn								0.36*	0.71*	0.61*	0.43*	0.19	0.46*
Pb									0.55*	0.25	0.66*	0.32*	0.49*
Zn										0.85*	0.33*	0.46*	0.26
Cu											0.13	0.47*	0.03
Cr												0.37*	0.62*
Cd													0.30*

*significant at $p<0.05$.

the supply of iron in the form of organic chelates formed in Podzols to be widespread in the lake's surroundings [10].

Mn concentration initially maintained at approximately 300 mg·kg^{-1} (including a large diversity), but from the depth of 5.5 m to the top of deposits a decreasing trend occurred, with much lower variability. The mean Mn content in the top layer of sediments was about 200 mg·kg^{-1} (Fig. 1A).

Mean concentrations of heavy metals (Zn, Cu, Pb, Cr, Cd, Ni) in the sediments of Wielki Staw were generally lower than these reported from the sediments of the lowland lakes [5, 23-26], and similar to the contents in bogs [9, 27] and humus horizons of soils [28, 29] in the Sudeten Mountains. Particular elements had different distribution in the vertical profile of sediments and the only common feature was the lowest amount of all elements in the sand strata at a depth of 8.6-9.0 m. Cu concentration was the highest (18-20 mg·kg^{-1}) in lower parts of sediments and gradually decreased to 4 mg·kg^{-1} at a depth of 1.5-3.5 m, and then slowly increased to the top of sediments, where it reached about 12 mg·kg^{-1}, which was only about 60% of the concentration in the oldest sediments (Fig. 1B). Also, Zn concentration was relatively high (up to 47 mg·kg^{-1}) in the oldest sediments and gradually decreased toward the top (including fluctuations) to a depth of 5.0 m, where it stabilized at 22-25 mg·kg^{-1}. Starting at a depth of 1.2 m, Zn content rose toward the top of sediments and reached 50 mg·kg^{-1}, similar to this in the oldest sediment. Pb concentration, initially slightly less than the amount of Zn (37-43 mg·kg^{-1}) increased in the older sediments up to 53-55 mg·kg^{-1} at a depth of 4.8-5.5 m, and then rapidly decreased and stabilized at about 30 mg·kg^{-1}. Starting from a depth of about 1.2 m, Pb concentration increased like zinc, but much more

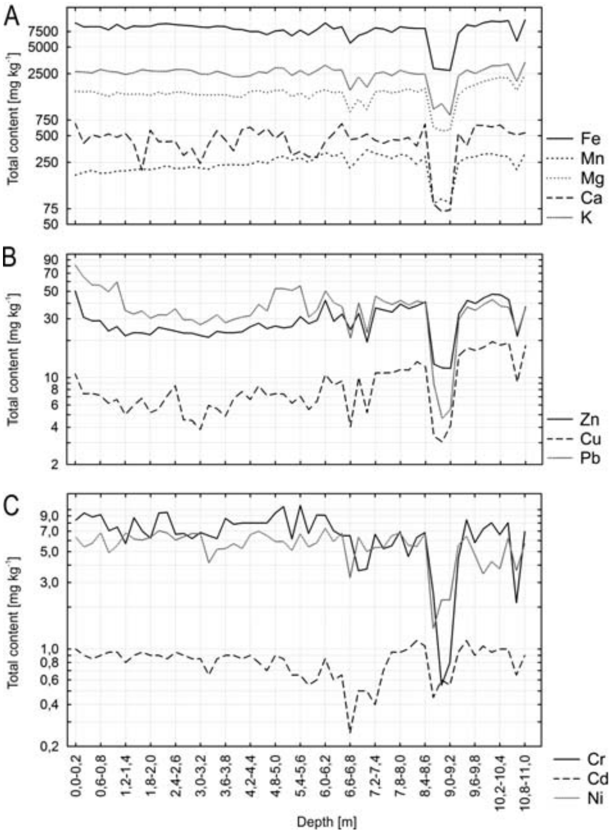

Fig. 1. Total concentrations of Ca, Mg, K, Fe, Mn (A), Cu, Zn, Pb (B), and Ni, Cr, Cd (C) in the bottom sediments of Lake Wielki Staw.

rapidly. In the uppermost part of deposits, Pb reached 82 mg·kg^{-1}, which was twice the content in the oldest sediments. The increase of Cu, Zn, and Pb concentrations, especially in the uppermost part of the sediment, was not accompanied by an increase of organic matter, silt, or clay contents, indicating anthropogenic origin of metals and supply from air pollution. Taking into account the rate of sediment accumulation calculated by Więckowski [12], it can be assumed that the lead concentration in the sediments began to grow as early as at the end of the first millennium AD.

The content of Ni and Cr oscillated around the mean values of 5.6 and 6.9 mg·kg^{-1}, respectively Ni and Cr, and did not exhibit a clear trend throughout the core (Fig. 1C). Also, in the case of Cd, no clear increase/decrease trend was observed. The amplitude of Cd concentration in the older sediments was several times higher than in the younger sediments, but the average amounts were similar and oscillated between 0.8 and 1.0 mg·kg^{-1} (the mean content of Cd in Table 1 seems underestimated due to a particularly low amount of this element in the sandy layer and in the samples containing wood, thus the median value better estimated the average concentrations of this metal).

Principal component analysis (PCA) indicated two main sources of variability of trace elements in the sediments of Lake Wielki Staw in the Karkonosze Mountains (Fig. 2). Factor 1, explaining 52% of the variation, is believed to be related to sediment texture, and factor 2, explaining ca. 13% of the variation, to the organic matter. All the analyzed elements can be distinguished into three groups:

(1) Ni and Cr – most strongly related to the organic matter and silt fractions in sediments (confirmed by high values of correlation coefficients in Table 2).

(2) Mn – strongly correlated with the amounts of K and Fe and mineral fine fractions (both silt and clay).

(3) Cu, Zn, and Cd associated with the Ca, Mg, and Na content in the sediment.

All three groups showed the rather natural origins of trace elements: biogenic origin (group 1), related to micaceous/feldspars minerals rich in potassium, often preserved in the fine earth fractions (group 2), and related to plagioclase and other minerals, rich in calcium, magnesium, and sodium (group 3). It is important to note that the analysis was conducted in averaged samples of 20 cm thickness, often bearing several thin organic or loamy or sandy strata. Thus, sample averaging for analysis could reduce dependence of the elements on the organic matter and fine mineral fractions. Furthermore, the PCA model does not explain as much as 35% variation, especially in the case of lead. It is likely to note that the atmospheric pollution of the anthropogenic origin, resulting in the elevated concentration of Pb, but also Zn, Cu, and to a lesser extent of Fe in the uppermost part of the sediment, not correlated with changes in the physicochemical properties of sediments, influences the reduction of PCA model efficiency. The sources of sediment contamination with lead are not clear if started at the end of the first millennium AD, as the ore mining and smelting have begun in the Sudeten Mts. in the 14th century. However, the correlation between Pb and Fe proved the industrial origin of the metal, possibly from long-distance transport. The lack of further dating makes impossible more precise correlation of the sediment contamination increase with the industrial history of the region. However, the rapid rise of Pb, Zn, and Cu concentrations in the uppermost 40 cm-thick layer clearly documented rapidly increasing atmospheric pollution in the Sudeten Mts. and its surroundings during the last three centuries, well confirmed by similar data from peatbogs in various parts of the Sudeten Mts. [9, 27].

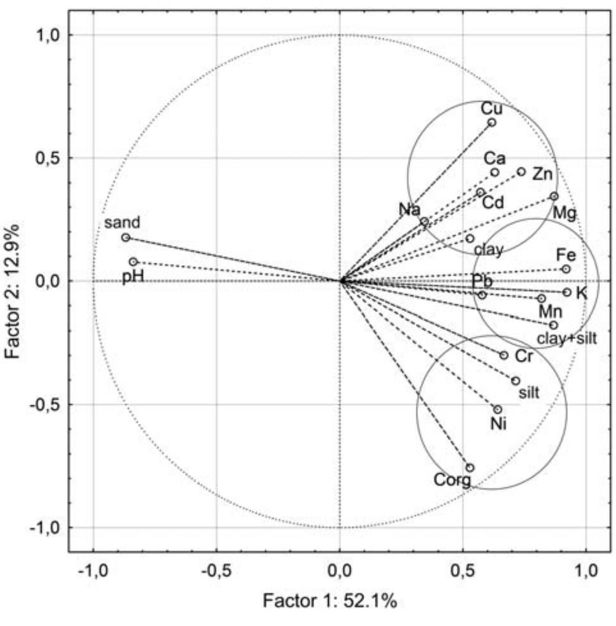

Fig. 2. Principal component analysis (PCA) for the relationship between trace elements and some physicochemical properties of the bottom sediments of Lake Wielki Staw.

Conclusions

1. Mean concentrations of trace elements are lower than in the sediments of Polish lowland lakes and close to the contents in the peat and humus horizons of soils in the Sudeten Mountains.
2. Pb, Mn, Zn, and Cu concentrations are relatively higher in the oldest (pre- and boreal) sediments and lower in the younger sediments (post-atlantic), whereas Cr, Ni, and Cd concentrations do not express any trend throughout the profile.
3. Increases of Cu, Zn, and especially Pb concentrations in the uppermost layer of sediments (1.2-1.5 m thick) is not justified by the change of the physicochemical properties of sediments and may only be explained with atmosphere anthropogenic contamination in the Sudeten Mountains that began at the end of the first millennium AD and accelerated during last three-four centuries.

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