Determination of Inorganic Elements in Sediment Samples from the Curonian Lagoon, Lithuania, by X-ray Fluorescent Measurements

This study reports on an X-Ray Fluorescence analysis of 30 determinants from sediment samples taken from the Curonian Lagoon, (CL) Lithuania, influenced by transboundary pollution. Data on number of less well-known elements, e.g. scandium, yttrium, niobium, and gallium are included. The heavy metals and metaloids (zinc (from 16 - 96), copper (up to 19), lead (2.45 - 2.7), arsenic (1.8 – 4.5), and sulphur (> 45) times) were found accumulated in clay-type fine sediments, differing from all the other sediments of mostly fine sand origin. Clay sediments were polluted higher than proposed in situ Belgium Sediment Quality Guidelines: Cr (5.3 times), Zn (1.46 times), Pb (1.93 times), and Cu (2.38 times). Lithuania, as well as other countries, discharging to Nemunas River system, is a signatory to the 2007 UNIDO Ministerial Declaration on "Zero Discharges". Enactment of this Declaration should lead to the reduction of some of these elements in CL fine sediments.

1. Introduction

The Curonian Lagoon, (CL), is a large 1584 km^2 area water body, separated from the Baltic Sea by a narrow sandy spit [1]. It is a largely fresh water Lagoon shared between Lithuania (1/3 part of CL) and Kaliningrad (2/3 parts of CL). It is fed by a major river, Nemunas, which rises in Belarus with some minor tributaries, flowing from Poland, and passes through 2/3 of Lithuanian territory. Additionally, the largest tributary of the River Nemunas, the river Neris rises in Belarus [1]. CL discharge waters to the Baltic Sea at the North side through the Klaipėda city harbour gates; its annual outflow is approximately 22 km3 [1]. The lower reaches of Nemunas River has settlements, cities, located on both Lithuanian and Russian opposite shores, hence the pollution has major trans-boundary (international) implications. Such trans-boundary location of water bodies also creates difficulties in integrated CL and Nemunas river monitoring.

Data from the studies undertaken by Lithuanian conjoint teams from universities and research institutes in 2001-2004 and later (NATO/CCMS Field Studies) in CL stimulated concerns related to pollution by industrial pollutants, i.e. accumulation of heavy metals, and POPs in sediments of Western and Central parts of the Lagoon [2]. The analysis of elements in these studies was performed using Atomic Absorption Spectrometry, (AAS), in Vilnius, or by Ionization Coupled Plasma Mass Spectrometry (ICP-MS), undertaken at the University of Lund, Sweden. The data showed, that sediment samples from sites, containing fine silty mud (aleurite), of 0.075-0.003 mm diameter, had a higher (for Zn, As, Cd) or lower (for Cr (Cr^{6+}+Cr^{3+}), Ni, Pb, Cu) values (mg/kg), comparing to US EPA established Sediment Quality Guidelines (SQGs) values, namely Threshold Effect Level (or Effect Range Low). It was also realized that the quantity (mg/kg) of metals and metalloids (Zn, As, Cd, Cr, Cu, Ni, Pb, Zn) in all these samples exceeds 1999 the Belgian in situ SQGs for these elements [2,3]. This study indicates the levels of elements (metals and metalloids) found by X-Ray Fluorescent (XRF) examination in other different sites of CL. As the sampling sites for the AAS and ICP-MS were different to those for the XRF analyses, no data for the former are included in this article.

As industrial pollution on the river basin and estuaries scale, and especially, its accumulation in sediments, is under considerable focus / attention of scientific organizations of European countries (i.e. SedNet organization) and United Nations Industrial Development Organization, UNIDO [4,5], it is important to have a periodic data review of independent analysis of pollutants quantities at non-standard state monitoring CL sites in order to have ongoing understanding of clean/ reference sites, levels, gradients and possible sources of pollution

Fig 1. Location of Curonian Lagoon (Lithuania) and sediment sampling sites

2. Materials and methods

2.1. Sampling

Ten sediment samples were taken from the Curonian Lagoon (CL), Lithuania. The coordinates of the sediment sampling sites are shown in *Table 1*. It should be noted that the water flow rate, sedimentation and sediment particle sizes, especially on the east and central areas are regulated largely by the Nemunas river, which flows into the CL on the east side. These groups of sampling sites are indicated in *Tables 1 - 3*. The sediment sampling was undertaken using a Peterson dredge, combined with sample layers separation platforms. Surface sediment layers up to 3 cm depth were collected from each site.

2.2. Measurements

The analysis of sediment particles sizes (30 to 50 g of sample) was undertaken by wet sieving, because of expected substantial amount of silt and clay size sediment. Samples were passed through the set screens of the decreasing mesh (Φ), covering all fractions from fine sand- coarse silt through to clay. Particle size was expressed in mm, as Φ, = -$\log_2$ (sieve mesh/interval in mm). The analysis of 20 fractions in total from the highest 1.0 – 0.5 mm up to the lowest of 0.005-0.001 mm interval was performed. The dry weight of the sediment particles in each size fraction was determined.

Samples for X-Ray Fluorescence (XRF) analysis were oven dried (~105 ^{0}C) to constant weight, then very finely ground and finally pelletised under a pressure of 25 tonnes. The elemental analyses of samples were undertaken by XRF measurement at the National Oceanographic Center, Southampton, England, using Magix-PRO WD-X-ray Fluorescence Spectrometer, Philips (Einhoven, Netherlands).

It is emphasized that due to very limited funds both for sampling and measurements, that the data shown are 'on-off' and hence no statistic analyses or standard deviations, etc.

Curonian Spit – National Park and UNESCO Protected Area. In the year 2000, the Curonian Spit was included in the UNESCO World Heritage List as one of the most beautiful and unique landscapes of Europe. The Curonian Spit is a narrow (up to 3 km width) sand and pine forest peninsula of 98 km of length, which divides the Curonian Lagoon from the Baltic Sea. In the South, Lithuanian part of the Spit borders with Kaliningrad region of the Russian Federation. The borderline marks the external borders of the European Union.

Fig 2. Sampling site No 1 in Nagliu Bay of Curonian Lagoon North of the Horses Horn and at the South end of Old Dunes.

3. Results and discussion

Table 2 indicates the types of sediment: the average of sieved particles diameter (in mm) and the percentage of dry weight of the particle fraction. The sediments from sites 2 - 8 (Central and Eastern side of CL) consisted mainly (up to 95 %) of fine sand (particle diameter 0.357-0.1 mm); they could be considered to be of terrigenous origin. Site 1 sediments consisted mainly (up to 96 %) of fine silty mud (i.e. aleurite or aleuritic pelite) of 0.075-0.001 mm diameter (*Table 2*).

This is the first time that a survey of the Northern part (Lithuanian territory) of the CL for metal / metalloid impurities by XRF has been undertaken (*Table 3*). Some geochemical data, e.g. concentrations of aluminum (Al_2O_3), calcium (CaO), iron (Fe_2O_3) indicate, that site 1 sediments (3.5 m depth) of CL contained from 2-3 times (for Al_2O_3), up to 10 times (for CaO) and up to 4.6-14 times (for Fe_2O_3) higher concentrations, than sediments from sandy bottom sites 2-8 (depth up to 3.0 m depth), RD1 (0.5 m depth) and Littoral (0.05 m depth).

In general, together with the smallest particle size this could indicate that clay minerals have highest adsorption capacities (this is well known in thin layer chromatography) for different chemicals including organics in site 1 sediment particles. The geochemical data for individual heavy metals and metaloids showed that the accumulation was higher in site 1 sediments than from all the other sediments for: zinc (from 16 to 96), copper (up to 19), lead (2.45 - 2.7), arsenic (1.8 – 4.5), and sulphur (> 45) times. The lower accumulation of cobalt (Co) and chromium (Cr^{6+} and Cr^{3+}) was found in site 1 sediment.

Table 1. WGS 84 Coordinates of the Sediment Samples from Western, Central and Eastern sides of Curonian Lagoon

Sampling site	N and E Coordinates	
Western side, bay, 3.5 m depth		
1	55° 27' 51'' N	21° 06' 58'' E
Central and Eastern side (high water flow rate) 1.0 m to 3.0 depth		
2	55° 27' 51'' N	21° 09' 15'' E
3	55° 27' 54'' N	21° 11' 11'' E
4	55° 27' 52'' N	21° 13' 07'' E
5	55° 30' 03'' N	21° 08' 42'' E
6	55° 28' 57'' N	21° 11' 05'' E
7	55° 26' 46'' N	21° 10' 41'' E
8	55° 25' 43'' N	21° 08' 41'' E
Western side, Near-shore, 0.5 & 0.05 m depth		
RD1	55° 33' 54'' N	21° 07' 28'' E
Littoral	55° 33' 54'' N	21° 07' 28'' E

*Table 2. Quantitative Distribution of Particle Size in the Sediment Samples from Western, Central and Eastern sides of Curonian Lagoon: Data of Granulometric (Sieving) Analysis**

Particle dia-meter, mm (average of fraction interval)	Curonian Lagoon, Lithuania									
	Western side			Central and Eastern side (high water flow rate) 1.0 m to 3.0 depth						
	Bay, 3.5 m depth	Near-shore, 0.05 & 0.5 m depth								
	1**	RD1	Littoral	2	3	4	5	6	7	8
0.75**	0.1	like 8	like 8							
0.56				1.42	1.01	0.56	0.30	0.60	0.51	1.13
0.45				0.69	0.49	0.34	0.14	0.45	0.50	0.49
0.375**	0.51									
0.357				1.17	1.10	0.75	0.21	0.92	0.93	1.18
0.282				2.10	3.31	3.20	0.51	2.09	2.42	3.84
0.225				6.31	13.07	13.18	0.88	9.09	11.91	20.88
0.18 – 0.175**	3.06			38.98	49.70	52.84	11.77	49.21	51.68	59.09
0.142				42.72	27.12	25.09	47.52	34.56	29.58	11.87
0.107				4.65	2.57	2.37	27.50	1.94	1.57	0.90
0.09				1.41	1.20	1.20	9.13	0.76	0.62	0.39
0.075** - 0.072	27.49			0.29	0.31	0.34	1.17	0.23	0.15	0.12
0.056				0.15	0.07	0.07	0.50	0.09	0.08	0.06
< 0.05 – 0.03**	56.5			0.11	0.05	0.06	0.38	0.07	0.06	0.06
0.0075**	4.16									
0.0025**	2.61									
0.001**	3.02									

* - *all units are % of dry weight*
** - *only for sampling site No 1*

Table 3. Minerals and Elements in the Sediment Samples of Western and Central and Eastern sides of Curonian Lagoon: Data of XRF Analysis

		Curonian Lagoon, Lithuania									
		Western side			Central and Eastern side						
Para-meter	Units (for dry weight)	Bay, 3.5 m depth	Near-shore, 0.5 & 0.05 m depth		1.0 to 3.0 m depth (high water flow rate)						
		1	RD1	Littoral	2	3	4	5	6	7	8
SiO_2	%	44.59	93.87	92.85	93.09	93.19	93.51	92.69	93.18	93.68	93.59
TiO_2	%	0.44	0.12	0.11	0.12	0.09	0.07	0.11	0.06	0.08	0.12
Al_2O_3	%	6.83	2.31	3.22	3.23	3.27	2.77	3.21	2.86	3.19	2.30
Fe_2O_3	%	5.05	1.10	0.62	0.63	0.49	0.57	0.62	0.36	0.38	1.10
MnO	%	0.12	0.04	0.03	0.03	0.02	0.02	0.03	0.02	0.02	0.04
MgO	%	2.42	0.04	0.24	0.24	0.15	0.09	0.24	0.11	0.08	0.04
CaO	%	24.74	2.07	2.03	2.04	1.10	1.05	2.03	1.56	0.64	2.07
K_2O	%	2.03	1.09	1.47	1.47	1.54	1.41	1.47	1.45	1.60	1.09
Na_2O	%	0.69	0.35	0.44	0.45	0.49	0.40	0.44	0.44	0.48	0.34
P_2O_5	%	0.26	0.05	0.05	0.05	0.05	0.03	0.05	0.03	0.04	0.05
Co	mg/kg	3	4	6	6	1	1	7	6	1	3
Cr	mg/kg	91	144	156	166	155	159	155	153	157	145
V	mg/kg	54	5	5	4	3	4	3	0	2	2
Sc	mg/kg	10	2	3	2	2	0	3	0	0	2
Cu	mg/kg	19	0	1	1	0	0	1	0	0	0
Zn	mg/kg	98	5	6	5	3	4	6	1	1	4
Pb	mg/kg	27	10	11	11	10	11	11	10	11	10
Ba	mg/kg	342	194	234	242	224	209	233	231	245	199
Rb	mg/kg	56	26	34	34	35	32	34	32	35	25
Sr	mg/kg	151	48	56	56	53	46	56	49	49	48
Y	mg/kg	20	12	12	11	11	10	12	10	10	12
Zr	mg/kg	135	220	182	179	128	90	181	97	118	219
Nb	mg/kg	11	7	7	7	7	6	7	6	7	7
U	mg/kg	2	1	0	1	1	0	1	0	0	0
La	mg/kg	24	6	9	8	8	7	6	5	6	9
Ce	mg/kg	63	15	17	3	23	11	19	3	11	10
Ga	mg/kg	7	2	3	3	3	2	3	2	2	2
As	mg/kg	9	5	2	3	2	3	2	2	2	5
Sn	mg/kg	9 (Sn)	6	7	9	7	8	6	7	8	9
S	%	0.948	< det.lim	0.063	0.067	0.012	0.022	0.065	0.029	0.02	< det.lim
Cl	%	0.081	0	0.002	0.001	0.003	0.003	0.001	0.005	0.001	0

Unfortunately, the high, but not characteristic to CL sediments, chromium levels is speculated to be due to the presence of either industrial discharges or to the presence of chromite. The results are in the range 91 - 166 mg/kg. The Belgium Sediment Guidelines indicate Cr^{6+} + Cr^{3+} to be 17 ppm, German < 100 mg/kg, and Netherlands 120 mg/kg.

In general, such increased element accumulation in site 1 sediments was confirmed by other data: the rarer elements, such as gallium, lanthanum, cerium, niobium, yttrium, vanadium were more accumulated (from 1.6 to 27 times) in site 1 sediment. Thus, sediment from the bay in the West region of Lagoon (site 1) is characterized by very fine particles of fine aleurite (or aleuritic pelite), (*Table 2*) sediment and relatively high accumulation of heavy metals (*Table 3*).

The use of Sediment Quality Guidelines (SQG) as criterion for sediment pollution evaluation is a very important current approach for many researchers, despite its accuracy and predictive capabilities, which were under in-depth discussions [3,6-8]

The SQG for heavy metals in river and marine sediments of three EU countries are presented in *Table 4*. The most strict and lowest values of SQG for heavy metals and metalloids or organo-metallic compounds are proposed in Belgium [3]. Comparison of CL sediments data with these SQG shows that only fine structure, clay containing sediments of site 1 were polluted higher than proposed for SQG for Belgium for *in situ* Cr (5.3 times), 2) Zn (1.46 times), 3) Pb (1.93 times), 4) Cu (2.38 times). Currently new European Environmental Quality Standards (EQS) for sediments are under development [9,10].

The relatively high tin content (up to 9 mg/kg) is of some concern, but this is assumed to be in the form of inorganic tin; if it were in the form of tri-butyl tin no living organism could exist in the CL. Major industrial uses of tin are cans for food, soldering and printed circuit boards in semi-conductor devices - such industrial discharges require further investigation.

Nickel – The results are below detection limit.

Cobalt - This is unlikely to be of environmental significance as the results are not > 7 ppm.

Vanadium - With the exception of site 1, (the highly calcareous sample) the results are of negligible environmental significance. However, vanadium at 54 mg/kg may well be due to very fine sediment particles, of geological background (unlikely in view of the other results), or perhaps, an industrial enterprise is discharging excessive quantities, in which case it should be controlled at source.

Scandium - all the results except of site 1 are of no (little) environmental significance, scandium is a rare element, not widely used in industry and is not a good element for XRF measurement. Scandium occurs in the earth's crust at approximately 5 mg/kg and its sulphate is very insoluble. Hence 10 mg/kg (site 1) is unlikely to be of environmental significance.

Copper - The only sample of any concern is site No1 at 19 mg/kg; this is greater than the Belgium SQG of 8 mg/kg. Again this can only be conjectured to have originated from industrial discharge(s), which should be controlled at source.

Zinc - The only sample of environmental significance, the site 1 result of 98 mg/kg is greater than the Belgium SQG of 67 mg/kg, and the industrial sources should be sought and discharges reduced/ eliminated.

Lead - All the samples except of site 1 are lower than the Belgium Guidance of 14 mg/kg. Any industrial discharges should be identified and discharge(s) reduced / eliminated at source.

Barium - Even the highest value of site 1 is unlikely to be of environmental significance especially in view of the very low solubility of barium sulphate.

Rubidium, Strontium and Yttrium - These elements, whilst of interest by the XRF technique are unlikely to be of any environmental significance, strontium sulfate is of low solubility.

Zirconium - The results indicate considerable variability sampling site to sampling site 90-220 mg/kg (219 mg/kg site 8) situated in the center of the lagoon is of interest, but little logical explanation can be offered. However, it is noted that zirconium salts are used in cosmetics.

Niobium and Uranium - Niobium is a comparatively little used element (except in speciality steels), and with the low values of uranium are of negligible environmental significance.

Lanthanum and Cerium - As rare earth elements they are of very little environmental significance.

Gallium - Again of site 1 is higher than the other samples; but 7 mg/kg is unlikely to be of environmental significance. The presence of gallium raises some conjecture - does it originate from a geological background or from industrial discharges, especially in view of its use (with arsenic) in semi-conductor devices?

Arsenic - As above); is the arsenic from site 1 of significance? But, it is emphasized that the result from site 1 is lower than the Belgium SQG.

Sulphur - see reference [11].

Chloride - No significance.

Table 4. Examples of some frequently used freshwater and marine Sediment Quality Guidelines , SQG, in EU and other countries*

Parameter (chemcal and ecotoxico-logical assessment criteria)	Belgian [3] *in situ;* 1999 to present	Germany [8] River sed. *(in situ),* II Quality Goal	Netherlands [3, 7] Marine sediment, dredged *(ex situ)*
Arsenic (As)	11	< 20	29
Chromium (Cr^{6+}+Cr^{3+})	17	< 100	120
Zinc (Zn)	67	< 200	365
Mercury (Hg)	0.12	< 0.8	1.2
Cadmium (Cd)	0.38	< 1.2	4
Nickel (Ni)	11	< 50	45
Lead (Pb)	14	< 100	110
Copper (Cu)	8	< 60	60

** - all units are in mg/kg dry weight*

During the period 1985 – 1996, Jokšas et al. undertook mapping of the distribution of sediment trace elements (including heavy metals; AAS analysis) and hydrocarbons in many CL bottom areas [12]. They showed that pollution of bottom areas by different pollutants is overlaping (i.e. at similar locations), and such areas are located in relatively deep places of West and South/ Souh-West CL. The distribution of the maximal concentrations of hydrocarbons and heavy metals in these places occured in fine-grained sediments (pelite, aleurite mud) [12]. Hence, our study by using a different analysis method (XRF) confirms that even nine years after their study [12], that the accumulation of pollution by heavy metals and areas of their locations in CL central part sediments remain similar.

Lithuania and the other three Nemunas River Basin countries (Belarus, Russia, Poland) are signatories to the United Nations Industrial Development Organization (UNIDO) Ministerial Declaration [5] "Zero discharges", and its enactment is awaited with interest.

Fig 3. Dead fish at the Western coast of North part of Curonian Lagoon due to eutrophication and toxicity during summer months (early August) 2002.

4. Conclusions

1) Geochemical data, e.g. concentrations of aluminum (Al_2O_3), calcium (CaO), iron (Fe_2O_3) indicate, that site 1 sediments of CL contained from 2-3 times (for Al_2O_3), up to 10 times (for CaO) and up to 4.6-14 times (for Fe_2O_3) higher concentrations, than sediments from sites 2-8, RD1 and Littoral. Sediment particles from site 1 had also the smallest size and highest quantity of adsorbed heavy metals, compared to all other sites. For observations on the other determinants see the Results and Discussion Section above.

2) The pollution of CL is a major transboundary matter: whilst the CL is situated in two countries, Kaliningrad Oblast of Russia and Lithuania. The major rivers flowing into CL rise in Belarus and Poland; some lesser rivers also discharge into the CL.

3) Lithuania and the other three countries (Belarus, Russia, Poland) are signatories to the United Nations Industrial Development Organization (UNIDO) Ministerial Declaration "Zero discharges"[5]. As Lithuania enacts this Declaration (equally as other CEE and NIS countries), the levels of elements (mostly heavy metals) discharged by industry and water transport should be reduced significantly in nearest feasible future.

Fig 4. Sediment sampling in Curonian Lagoon (A) and at the Nemunas River delta (B)

5. Recommendations

1) *International collaboration* and *strict local management* are required between transboundary pollution related countries: Belarus, the Kalinigrad Oblast of Russia and Lithuania [+ Poland], in order to reduce this pollution in Nemunas River Basin and CL, and thus reduce adverse environmental health effects.

2) The powers of the *inter-state* Nemunas River Catchment Council needs to be strengthened substantially. If this is unachievable, then the initiation of a suitable project is required by *independent international organization(s),* such as the United Nations Specialist Agencies. It is of global environmental importance, as the CL discharges to the Baltic Sea, the gateway to EU, CEE and NIS countries with different economic and political attitudes.

3) In order to pursue these important studies, further *international support* will be required for Lithuanian academics to fund further sampling expeditions and to undertake further elemental and other analyses. Funding will be required also for related personnel allocation and training to ensure successful management for the decrease of point and non-point discharges.

4) Both local municipalities and small /medium enterprises located on both sides of the Nemunas river suffer from poor economic situations. The appropriate Ministries, with UNIDO assistance, need to mobilize funding from international sources, to enact the principles of the 2007 "UNIDO Zero Discharges"

Abbreviations / Acronyms List

AAS	-	Atomic Absorption Spectrometry
CCMS	-	Committee on the Challenges of Modern Society
CEE	-	Central and Eastern Europe
CL	-	Curonian Lagoon
EPA	-	Environmental Protection Agency
EQS	-	Environmental Quality Standards
EU	-	European Union
ICP-MS	-	Ionization Coupled Plasma - Mass Spectrometry
NATO	-	North Atlantic Treaty Organization
NIS	-	Newly Independent States
SQG	-	Sediment Quality Guidelines
UNIDO	-	United Nations Industrial Development Organization
US	-	United States
WGS 84	-	World Geodetic System of year 1984
XRF	-	X-Ray Fluorescence

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AUTHOR DETAILS

Anolda Četkauskaitė*†, Saulius Gulbinskas‡, and Ričardas Paškauskas‡¶

*†Department of Biochemistry and Biophysics, Faculty of Natural Sciences, Vilnius University, Čiurlionio Street 21, Vilnius LT-03100, Lithuania,

‡Coastal Research and Planning Institute, Klaipėda University, H. Manto Str. 84, LT-92294, Klaipėda, Lithuania,

¶Laboratory of Hydrobotany, Institute of Botany, Žaliųjų Ežerų Str. 49, Vilnius LT-08406, Lithuania

*Corresponding author. Email: anolda.cetkauskaite@gf.vu.lt