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VARIATION IN THE CONTENT OF DIFFERENT FORMS OF MERCURY IN RIVER CATCHMENTS OF THE SOUTHERN BALTIC SEA – CASE STUDY

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ABSTRACT

Mercury (Hg) is recognised as a global environmental pollutant. Despite numerous studies being conducted around the world, the transformation of mercury in natural environments is still not fully understood. In addition, increasing droughts and heavy rains are currently observed to contribute to changes in the circulation of Hg. The purpose of this study was to recognise the influence of extreme meteorological and hydrological conditions on the inflow of various forms of mercury to the coastal zone of the Bay of Puck. The studies were carried out at estuarial stations of four rivers belonging to the southern Baltic Sea catchment: Reda, Zagórska Struga, Gizdepka and Płutnica. The results showed that meteorological and hydrological parameters affect mercury speciation in river catchments, which translates into inflow of labile mercury to the southern Baltic Sea.
Keywords: Hg, Hg transformations, river catchments, Baltic Sea.

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INTRODUCTION

Mercury (Hg) is a neurotoxin present in the environment that negatively affects the human nervous system and has been linked to many diseases such as Parkinson's disease and Alzheimer's. In addition, mercury easily penetrates the placental barrier, causing fetal damage and miscarriage. In view of the fact that this metal causes a number of health problems already at low concentrations [1,2,3], research conducted to understand the circulation of Hg in the environment is essential. This is particularly important because mercury has been found to easily bioaccumulate and then biomagnify in the trophic chain in which humans stand at the top [4,5,6].

Mercury has been used for many years in a wide range of industrial as well as economic sectors, which has translated into increased Hg emissions to the environment [7]. Owing to the lack of positive effects of mercury on living organisms, many legal restrictions were introduced to reduce Hg emissions to air, water and soil. These changes have been implemented, however, no proportionate decrease in mercury concentrations in various environmental components has been observed. One reason is the continued deposition of mercury on land, where the metal accumulates in the surface layer of soils [8-11]. Studies conducted by scientists indicate that elevated concentrations of the metal are found near individual household furnaces as well as in larger urban areas where mercury is emitted into the environment during energy and heat production [12-16]. This indicates a significant impact of coal burning on environmental mercury concentrations. Moreover, despite the introduction of legal restrictions that have led to a reduction in anthropogenic sources of mercury, the metal that has been deposited over decades is now being remobilised from land into river water [13,16,17,18]. The process of Hg leaching into rivers is stimulated by surface runoff, which in turn is amplified by heavy rains. In addition, the type of catchment development plays a major role in the process of mercury transport between land and water systems [19,20]. A study by [15] showed that continuous sealing of natural surfaces, through e.g. concreting, promotes the intensity of mercury leaching from land. The mobility of Hg in the environment is also dependent on the speciation of mercury i.e. the form in which it occurs [16,21,22]. Mercury that is bioavailable to organisms is easily transformed and transported in the environment, which directly affects the form of mercury delivered by rivers to the sea [15,16,18]. This is particularly important because rivers are the main source of mercury to the Baltic coastal zone [23]. Thus, it is of utmost importance to recognise the processes that promote the transformation of mercury in the environment.

MATERIAL AND METHOD

SAMPLING LOCATION

Samples for the study were collected from the estuary stations of four rivers draining into the Puck Bay: Reda, Zagórska Struga, Gizdepka and Płutnica. Each of the selected rivers was characterised by different land use type, catchment area, length and flow of river water (Table 1). Additionally, research material was collected from stations located in the coastal zone of the sea (ca. (surface layer, 0-20cm) of the Reda and Gizdepka catchments and river and seawater from the four rivers mentioned above were collected.

Tab. 1

Characteristics of the studied rivers [24].

River	Length (km)	Area (km^2)	Average flow (m^3/s)	Dominant type of estuary station development
Reda	49.3	485.2	5.9	agricultural land and pastures
Zagórska Struga	26.0	144.5	1.5	agricultural land, meadows, pastures
Płutnica	11.2	85.2	0.8	wetlands, pastures
Gizdepka	11.8	38.5	0.3	forests, agricultural land

Fig. 1 Sampling location.

SAMPLE COLLECTION

The study was conducted in 2015-2017. Samples were taken in spring, summer, autumn and winter during the average flow of the Reda River water (3.3-4.3 m^3/s) and after extreme meteorological and hydrological conditions such as low (<3.2 m^3/s) and high-water flow (>4.3 m^3/s).

Soil samples were placed in plastic bags and then frozen at -20°C. Before analysis, the collected material was freeze-dried and homogenised using a ball mill.

Sea and river water was collected into borosilicate bottles (1.2 dm^3). Immediately upon return to the laboratory, the water was filtered through pre-treated Whatman GF/F filters with a pore diameter of 0.7µm. The samples were then frozen at -20°C. As with the soil samples, the material was freeze-dried prior to analysis.

Both sampling and preparation of the material for analysis were carried out in accordance with the principles in force for the determination of trace metals in environmental materials, which minimised the possibility of contamination of the samples [25,26].

CHEMICAL ANALYSES

To determine the concentration of total mercury (Htot) and individual forms of Hg in soil and in river and marine suspension (SPM), the thermo-desorption method was used using atomic absorption spectrometry with an automatic analyser DMA-80 (Milestone, Italy) [17] Thanks to this method, a total of five mercury fractions belonging to two groups were separated (Fig. 2): labile mercury, i.e. such forms of Hg that are easily transformed in the environment:

- Hg_{ads1} (mainly halides: $HgCl_2$, $HgBr_2$, HgI_2, $Hg(CN)_2$ oraz Hg^0), mercury adsorbed on organic matter/fine fraction, released at 175°C,
- Hg_{abs} (mainly organic forms: MeHg, $Hg(SCN)_2$, $(CH3COO)_2Hg$, $Hg(NO_3)_2$), $Hg(ClO_4)$ and Hg bound to organic matter), mercury absorbed in organic matter, released at 225°C,
- Hg_{ads2} ($HgSO_4$, HgO), Mercury adsorbed on organic matter/fine sediment fraction, released at 475°C

and stable mercury, i.e. forms of Hg that are not readily transformed in the environment:

- HgS (released at 375°C),
- Hg_{res} (residua fraction), mercury embedded in minerals, released at 750°C.

The limit of detection (LOD) for both the suspension and soil was: total mercury 0.05 ng/g d.m.; mercury fractions 0.06 ng/g d.m. The analytical recovery representing the difference between total mercury and the sum of the individual Hg fractions was approximately 90%.

STATISTICAL ANALYSES

Statistical analyses were performed using STATISTICA 12. Data distribution was checked using the Kolmogorov-Smirnov test. Additionally, in order to verify the significance of differences in the content of particular Hg forms in soil, the Kruskal-Wallis ANOVA test was used, *$p < 0.05$.*

RESULTS AND DISCUSSION

SEASONAL VARIATIONS IN THE CONTENT OF HG FORMS IN THE SOIL OF RIVER CATCHMENTS

Mercury concentration in soil depends on its content of fine fraction and organic matter [28] as well as on the location of anthropogenic sources of the metal. The highest concentrations are recorded in Silesia, where Hg was introduced into the environment from various industrial sectors [29]. In the region of northern Poland, where the studies were carried out, median total mercury concentrations (Hg_{tot}) were 20.2 ng/g and 34.3 ng/g in the Reda and Gizdepka drainage basins, respectively. Although the Gizdepka catchment had higher Hgtot concentrations, the organic matter and fine fraction of the Reda catchment was more enriched in mercury. This was probably due to the significant urbanisation of the Reda catchment, where fossil fuel combustion and road transport play a key role and are significant sources of Hg to the environment.

Although Hg_{tot} concentration in the catchments of the studied rivers changed during the study period (Table 2), the differences were not statistically significant. However, significant differences were found in the case of the content of particular forms of Hg in soil (ANOVA, Kruskal-Wallis, $p < 0.05$), which indicates a significant role of the influence of meteorological-hydrological parameters in the speciation of mercury in the environment. The greatest disproportions between typical seasons: heating and non-heating were recorded in surface soil of the Reda drainage basin. During the heating season, the median mercury content associated primarily with halides (Hg_{ads1}) was 82% (Figure 2a). This was likely due to land deposition of Hg from fossil fuel combustion [9,10]. This was confirmed by [30], who demonstrated that during the heating period Hg_{ads1} was the dominant form in the aerosols. The non-heating period, on the other hand, in both catchments was characterised by high mercury mainly associated with organic matter (Hg_{abs}), which was mainly recorded in the catchment dominated by forests and agricultural land (Gizdepka). The dominance of Hg_{abs} during this period was likely related to the development of mercury-absorbing vegetation [27]. A study [31] showed that in catchments where reed cutting occurs, there is an increase in the proportion of organic mercury (Hg_{abs}). This suggests that reeds readily absorb bioavailable mercury. Stable forms of mercury can be transformed in the environment [14]. First of all, it depends on the aerobic conditions in the soil [21,32].

Previous studies have shown that the most intensive formation of HgS occurs at greater depths in soil profiles, where degradation of organic matter leading to anaerobic conditions is observed [16,21,22,32]. Considering the total study area, the highest median mercury sulfide content was found in the surface soil of the Gizdepka catchment after heavy rainfall (Figure 2b). In this case, it was probably due to the inflow of this form of mercury from nearby agricultural areas along with surface runoff. According to the Regulation of the European Parliament and of the EU Council of 5 June 2019 [33], fertilisers may contain mercury up to 1 mg per kg of dry mass of fertiliser, which is an additional source of metal to the environment. In the case of Reda, there

was no statistically significant difference in HgS content in surface soil between the seasons studied.

The conducted analyses have shown that catchments that are dominated by urbanised areas are characterised by a significant difference in the content of individual forms of mercury in soil during the heating and non-heating season, which is caused by intensive burning of fossil fuels [9,10,12].

In contrast, it is in more natural catchments, i.e., under minor influence of human activities, that variability in the content of individual forms of mercury in soil can be observed throughout the year. This means that natural catchments provide excellent testing grounds for recording mercury transformations.

Tab. 2

Median total mercury concentration (ng/g).

Season	**Hg_{tot} (ng/g)**	
	Reda	**Gizdepka**
Heating	23.1	35.4
Non-heating	19.5	36.4
Drought	24.0	11.4
Rainfall	27.0	38.8
Heavy rainfall	19.1	29.5
Snowmelt	17.8	25.7

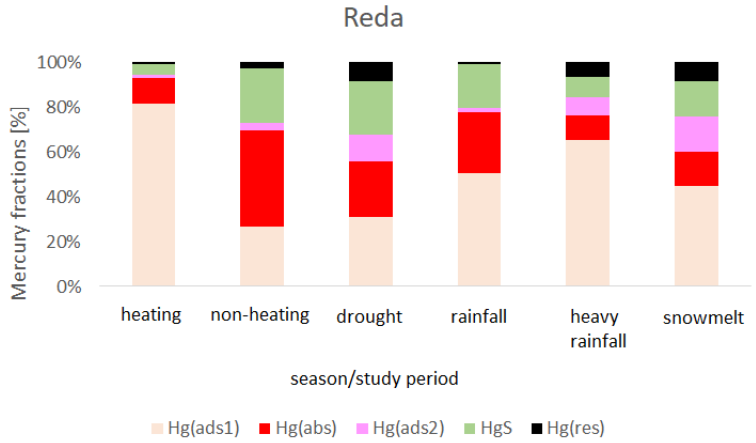

Fig. 2a Contents of particular Hg fractions (%) in the Reda catchment basin, divided into seasons/study periods.

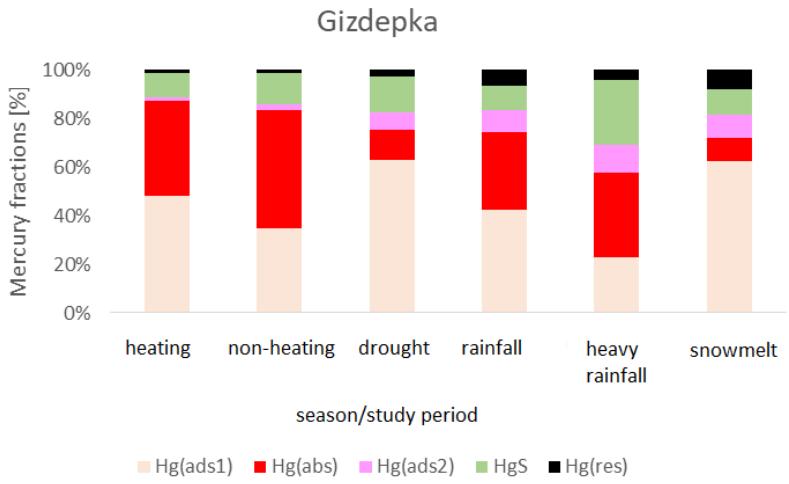

Fig. 2b Contents of particular Hg fractions (%) in the Reda catchment basin, divided into seasons/study periods.

THE LOAD OF PARTICULAR FORMS OF MERCURY ENTERING THE BAY OF PUCK

Rivers are the main source of mercury to the Baltic Sea environment, accounting for 85% of the introduced load [23]. Studies conducted by a number of scientists have shown that the highest Hg load is introduced during periods of heavy rainfall, when river water flow increases [13,16,18]. Furthermore, heavy rainfall and snowmelt, which determine surface runoff, contribute to the erosion and leaching of mercury deposited on land, which directly affects the load and form of Hg entering the southern Baltic Sea [15,34,35]. Mercury accumulated in the surface soil layer after leaching from soil profiles can either be deposited in bottom sediments or directly enrich the river suspension. Some of the mercury deposited in the sediment may be transported to the seashore zone with the movement of bedload of rivers. This process depends on both the hydrogeological parameters of the rivers as well as on the content of the fine fraction in the sediment [36,37]. It is the fine fraction, due to its large specific surface area, that shows a very high sorption capacity for heavy metals including mercury [28]. According to the studies conducted in relation to the load of total mercury entering the Baltic Sea with river water [13,14,38] the highest load of individual mercury fractions together with river bedload is introduced into the sea during intense rainfall and floods. Nevertheless, irrespective of the season and meteorological/hydrological conditions, the load, which mainly consists of labile forms of mercury, is carried into the sea along with the sediment. Moreover, during snowmelt, which is one of the shortest periods of the year, the load most enriched in labile mercury associated with halides is introduced to the Baltic Sea [18]. This is a direct response to the leaching of mercury from fossil fuel combustion with surface runoff caused by thaw. Much of the mercury associated with terrestrial organic matter due to remobilisation from land also enriches riverine suspended solids. Suspended solids (SPM) carry a variety of pollutants including mercury [18,21]. Although the highest loads of suspended Hg are carried into the Puck Bay by the main rivers i.e. the Reda and Zagórska Struga, the most enriched load in mobile mercury is carried into the Baltic Sea by small watercourses whose catchments are dominated by pastures and wetlands [15]. As in the case of river sediment, the load entering the sea with SPM mainly consists of labile mercury. Consistently also during melt, the load most enriched in labile mercury is introduced with the suspension. Nevertheless, fluvial suspended solids during the snowmelt period are characterised by a significant enrichment in organic mercury/associated organic matter (Hg_{abs}) in contrast to sediments [15].

Although a load of bioavailable mercury is delivered to the Puck Bay along with the dragged debris, this load represents only about 1% of the mercury that enters with suspended river matter. This confirms the significant role of suspended solids as an effective pollutant carrier in the terrestrial-marine system mercury cycle. This is particularly important as suspended matter provides food for coastal aquatic organisms. Analyses [5,6] show that organisms at the beginning of the trophic chain (e.g. macrozoobenthos) as well as those at higher levels can contain up to 90% organic mercury. This indicates a relationship between the content of individual forms of mercury in sediment and suspended solids and the proportion of Hg in aquatic organisms. This is particularly important because mercury undergoes biomagnification, which means that it eventually enters human organisms at the top of the trophic ladder.

DIFFERENCE IN THE PROPORTION OF HG FORMS IN SUSPENSION IN RIVER AND MARINE SYSTEMS

The Reda was the only river in which differences were noted between the contents of individual forms of Hg in suspension at the estuarine and marine stations (located about 200 m from the river mouth) during all designated periods. In every season except drought, a higher proportion of Hgabs was recorded in suspension at the marine station. In most cases, this was associated with significantly higher concentrations of suspended solids in the sea than in the river, suggesting that in this area there is an accumulation of riverine suspended solids that are transported through the Reda throughout the year.

The heating season, consistent with previous observations, had higher proportions of mercury from atmospheric deposition (Hg_{ads1}) relative to the non-heating season, where soil erosion and vegetation development (e.g., pollen) result in a predominance of Hg_{abs} in suspension. Such tendencies were recorded both at estuarine stations of the studied rivers and at sea stations, and the shares of contents of particular Hg fractions were equal at both stations of the remaining studied rivers. Additionally, it is worth emphasising that only in the period of drought significant differences were found between contents of particular Hg forms in suspension at estuarine and marine stations of all studied rivers. This was presumably due to low river water flow and low mercury loads entering the sea, resulting in no enrichment of marine suspended solids in terrestrial Hg. In spite of the fact that much higher river water flow and higher daily suspended mercury loads were recorded during rains and heavy rains, no differences in Hg fractions were observed at sea and estuarine stations of the Gizdepka and Zagórska Struga rivers. In the case of the Płutnica River, a lower content of Hgabs was recorded in the sea compared to the estuary, which may indicate transport of suspended matter with absorbed Hg deep into the Puck Bay. The most intensive enrichment of both riverine and marine suspended solids occurred during snowmelt, with the greatest differences between estuarine and marine stations recorded in the largest catchments (Zagórska Struga, Reda). Intensive surface runoff caused by snowmelt contributed to the enrichment of suspended matter in the Hg_{abs} fraction, the content of which increased to 81% in the suspension in the Zagórska Struga and to 82% in the suspension in the Reda, which was the highest Hg_{abs} content in suspension of all sea stations during the entire study period. This is particularly important for aquatic organisms living in the coastal zone, which are exposed to mercury. Because it is absorbed within organic matter, it is consumed by organisms lower down the trophic chain [5,40]. Due to the biomagnification process, this metal passes through the successive links of the trophic ladder and ultimately poses a threat to humans, for whom the consumption of

fish and seafood is the main source of metal entry into the body [41].

CONCLUSIONS

1. Since the end of the 20th century the inflow of Hg from anthropogenic sources has been limited, however, meteorological and hydrological phenomena influence the transformation and mobility of mercury in river catchments. These processes lead to an increase in inflow of labile forms of the metal to the southern Baltic Sea.
2. Mercury leaching from fossil fuel combustion has contributed to significant increases in river Hg concentrations.
3. The highest load of labile mercury with suspended solids was introduced to the southern Baltic Sea during heavy rainfall/floods. However, the load most enriched in bioavailable forms of Hg was introduced during snowmelt from small forest-agricultural rivers. The dry period, in turn, led to a reduction in the amount of introduced labile mercury to the coastal zone of the sea.
4. With river suspended solids, a much higher load of labile mercury is introduced into the sea compared to the amount of Hg contributed with river sediment. Nevertheless, both the load introduced with suspended and trailing river debris leads to enrichment of the marine coastal zone (sediment and marine suspension) with bioavailable mercury.

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