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Features of Pesticide-Contaminated Surface Water

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Abstract: This work aims to investigate the concentration indices of 14 pesticides in the major water bodies of the Southern Federal District of the Russian Federation. The study was conducted in 2019 using samples of bottom sediments and surface waters collected from three reservoirs (Krasnodar, Proletarian, and Veselovsky) and two rivers (Don and Sal). Concentrations of active pesticide substances were studied, and the results were benchmarked against 2018. In 2019, 14 active pesticide substances were detected in water and sediment samples from the three reservoirs under study. Maximum pesticide concentrations were observed in the spring of 2019 and autumn 2018. Total concentration indices in these time frames were similar, but autumn 2019 is 20 times lower than 2018 (0.7 versus 6.5, $p \leq 0.001$). Total pesticide concentrations for rivers and reservoirs averaged about the same (0.15-0.80, $p \geq 0.05$). There are different levels of pesticides in rivers and reservoirs. The detected pesticide concentrations did not exceed the maximum allowable concentrations, but the cumulative effect can have a harmful impact on hydrobionts in the future.

Key words: Concentration, pesticides, pollution, reservoirs, rivers, the Southern Federal District, water bodies.

Introduction

Currently, approximately 55,000 different chemical compounds are found in the external environment as a result of human activity (Akhtar et al., 2014). Many of these chemicals are unsafe for living organisms and human health. Based on the number of chemicals entering the environment following human activity, approximately 3% fall under the category of pesticides (Satiroff et al., 2021). Consequently, pesticides are constantly accumulated in the environment, participating in the metabolic processes of the ecosystem. These trends led to the accumulation of pesticides as one of the main problems brought to light in the Stockholm Convention on Organic Pollutants (Zeng et al., 2018).

The pathways through which pesticides, as well as other pollutants and toxic substances, enter water bodies and rivers vary. These include atmospheric precipitation, soil deposits, surface or groundwater leaching or drainage processes, or direct inputs from on-farm treatments (generally as particles or droplets). Some pesticides can enter rivers from products of animal or human origin (Tang et al., 2018).

The toxic effects of pesticides are related to their solubility in water, reagent activity, and the degree of pesticide uptake by bottom sediments, plants, and

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animals (Li et al., 2021). Climate plays an important role as well. The factors that have a direct influence on pesticides include temperature and solar radiation. Especially, when temperature indicators increase or decrease, the solubility of pesticides in water may also change, depending on the chemical structure of the compound itself. Half of the samples contained pesticides, such as hexachlorocyclohexane, but at acceptable levels as per regulations of the European Union (Sanchez-Palencia et al., 2017).

It is well known that pesticide concentrations in sediments at the bottom of water bodies decrease much slower than in the water column (Pan et al., 2017).

Bottom sediments are also known to have unequal distribution of pollutants. The reason for this is the water movement system typical of each large enough water body (or water body with running water). This system determines water circulation in the inshore when cyclonic and anticyclonic structures are formed during maximum runoff indicators (Tang et al., 2018). Furthermore, the exact composition (in fractions) of bottom sediments is a way of controlling pesticide concentration indices. In particular, a cumulative effect is observed when the concentration of pesticides in the bottom sediment increases. According to some studies, the pH of the aquatic environment and the presence of iron salts (silicates) and heavy metal ions can also significantly impact pesticide sorption rates (Ullah et al., 2019).

Whereas soil cover plays a significant role in accumulating pesticides on the soil, it is the biophase for the hydrosphere. It is especially evident in the deeps of rivers, seas, and lakes. Pesticides accumulate in lower parts of food chains, e.g., plankton, invertebrates, and further down the chain – in fish and mammals. However, the principal quantity of pesticides accumulates in microorganisms (Qu et al., 2018). Most organochlorine pesticides break down rather rapidly in water. Therefore, products with much lower toxicity indicators are developed (Tang et al., 2018; Wang et al., 2021). Although the effects of individual pesticides are well studied, a comprehensive approach is required due to the increased use of pesticides in some regions recently. In most cases, the aquatic ecosystems consist of a number of pesticides. Usually, their concentrations vary according to the season and the level of surface water release. This shortcoming specifies the need for this study.

This study provides data on the state of aquatic ecosystems as of 2019. The authors suggest that in the cold period (spring and winter), active substances used a year before but with a half-life of 6–9 months can be found in silt sediments and water columns. In summer and autumn, pesticide contamination, which occurred in the current year, may be observed. The purpose of the study was to determine the concentrations of 14 active pesticide substances by investigating water bodies in the SFD. The study's objectives included: a) determining the concentrations of active substances in each of the selected water bodies in the Republic of Kalmykia, Adygea, Karachay-Tcherkessia, Rostov and Stavropol regions, as well as Krasnodar Krai b) comparing indicators obtained from different locations between each other.

Material and Methods

Region of Study

The studies were conducted in 2019 on water bodies located in the following regions: Republic of Adygea, Republic of Kalmykia, Karachay-Cherkess Republic, Rostov region, Stavropol region, and Krasnodar Krai, which are part of the Southern Federal District of the Russian Federation (Figure 1A). The examined water bodies include two reservoirs of Manych water economic system (Veselovsky, Proletarian), Krasnodar Reservoir (Figure 1B), and two rivers (Don and Sal, Figure 1B).

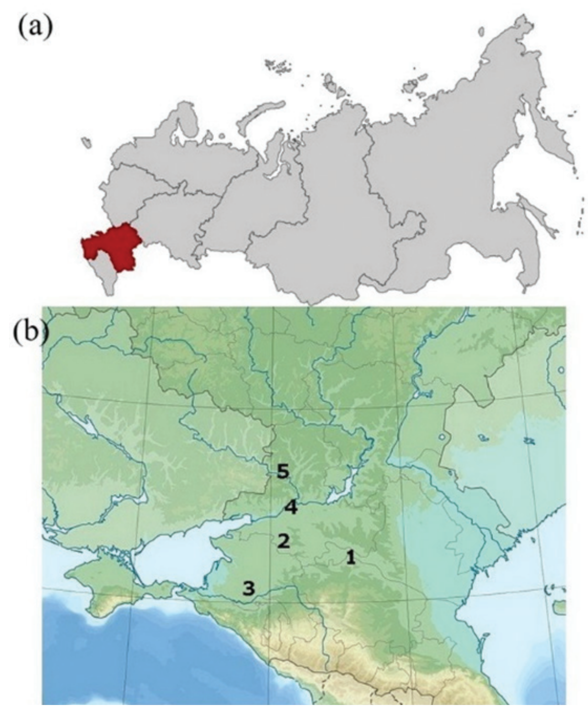

Figure 1: A – Southern Federal District of the Russian Federation; B – sampling points: 1 – the Krasnodar reservoir, 2 – the Proletarian reservoir, 3 – the Veselovsky reservoir, 4 – the Don river, 5 – the Sal river.

Due to its geographical location, climatic conditions, geomorphological and geological-tectonic structure, Krasnodar Krai is frequently exposed to natural hazards and disasters caused mainly by dangerous meteorological, hydrological, geological processes and phenomena, such as mudflows, avalanches, hail, and heavy downpours.

This may lead to an increased flow of pesticides from fields to rivers and reservoirs. The average annual discharge of the river in Krasnodar Krai is 22.05 km³. Specific resources amount to 292,000 m³/year per 1 km², which is higher than the average in the Russian Federation (237,000 m³/year), and 4,300 m³/year per inhabitant, which is five times lower than in the Russian Federation (27,800 m³/year). The distribution of runoff on the rivers during the year is uneven. Thus, spring run off typical for rivers in the middle-altitude mountains of the Black Sea chain of the Caucasus (Tuapse, Sochi).

Pesticide Active Ingredients

Table 1 presents the key properties of 14 pesticides under examination.

As shown in Table 1, most pesticides under study are fungicides and herbicides actively used to control fungi and weeds. At the same time, the chemical structure of these compounds is quite different, i.e., they are derived from different substances.

Sampling

Water and sediment samples were collected from the hydrobiont habitats, i.e., rivers and reservoirs, to estimate pesticide levels. Sampling stations were chosen based on the location of the fishing zones.

The water sampling method complied with the "Uniform Rules for the Sampling of Agricultural Products, Foodstuffs and Environmental Objects to Determine Microquantities of Pesticides" (Methodological instructions of the Deputy Chief State Sanitary Inspector of the USSR No. 2051-79 dated 21.08.79). Samples were taken in the water layer close to the surface to a depth of 0.5 m. Sampling was performed using a glass barometer in a 10 dm^3 volume dark glass container with a hermetically sealed lid. Prior to the sample's intake, the water bottle and reservoir were rinsed with the same water, and the sample was then taken.

The extracts were examined by liquid chromatography (Applied Biosystems, USA) using an ultraviolet detector with a degassing column and thermostat. The Reprosil-PUR ODS column template with a size of 4150 mm and a working wavelength of 230 nm was produced by Elsico, the Russian Federation. The column was thermostated at 40°C. The mobile phase consisted of a molar solution with 0.01 acetonitrile and H_3PO_4 in a ratio of 4 to 6 per volume. The solution was isocratic, with flow parameters of 0.4 mL per minute. Only 10 microlitres (µl) of the extract obtained from the sample was injected into the chromatograph. The retention time parameter was used to determine the active ingredient of the pesticide. For quantification, an absolute calibration method was applied.

Table 1: Key properties of 14 pesticides

Active substance name	*Substance derivative*	*Substance intended use*	*Maximum allowable concentration values (in mg/dm^3)*
1	Nicotinic acid	H	0.1000
2	Benzimidazole	H	0.4000
3	Benzimidazole	I	0.1000
4	Dicarboxamide	F	0.1250
5	1,2,4-Triazinone	H	0.5000
6	Urea	F	0.0100
7	Oxazolidinedione	F	0.0500
8	Carbamata	H	0.0001
9	Phthalimide	I-A	0.1000
10	Phthalimide and benzoxazine	H	0.0400
11	Oxyacetamide	H	0.5000
12	Aryloxypropionic acid and quinoxaline	H	0.0010
13	Methoxybenzamide	A	0.0100
14	Furana	H	0.0070

Note: 1 – Diflufenican, 2 – Imazetapyr, 3 – Imidacloprid, 4 – Proprodione, 5 – Metribuzin, 6 – Penticuron, 7 – Famoxadone, 8 – Fenmedipham, 9 – Flubendiamid, 10 – Flumioxazin, 11 – Flufenacet, 12 – Quizalofop-p-ethyl, 13 – Cyprosulfamide, 14 – Ethofumesate, H – Herbicide, A – Antidote, I-A – Insectoacaricide, F –Fungicide.

The risk associated with the pesticide under investigation was determined based on the following formula:

$$C_{tot} = \sum \frac{C_i}{MAC_i} \qquad (1)$$

where C_{tot} is the total relative toxicity of water from the sample, C_i is the detected concentration of a toxic compound, and MAC_i is the maximum allowable concentration value of the studied pesticide. In the case when $C_{tot} < 1$ the aquatic environment or samples from bottom sediments are considered safe. The method used makes it possible to determine the toxicity of the pesticide to the whole of the studied ecosystem.

Statistical analysis was performed using Statistica v. 7.0 software. The difference in concentration of an active substance or different substances between seasons and water bodies was determined using the Student's *t*-test. The minimum level of significance was $p \leq 0.05$.

Results

The 14 active pesticide substances were found in the water samples and bottom sediments of the three reservoirs studied in 2019. At the same time, they were significantly different in terms of detection frequency. Substances 2, 3, 5, 10, and 13 (see notes in Table 1) were present in all samples throughout the 2019 seasons, while substances 1, 4, and 6 were occasionally observed.

Total toxicity values calculated by the proposed formula did not exceed 0.7 in spring 2019, compared with an average of less than 0.1 in autumn, summer, and winter. During the previous year, this indicator exceeded 0.8 and remained above 0.15 for the rest of the year.

For the Sal and Don rivers, 12 pesticides were detected in 2019, except for substances 9 and 12. Results from the studies carried out for these rivers are presented in Table 2.

The most commonly observed compounds in the water samples and bottom sediments of both rivers were 3, 5, 10, and 13. Other compounds were recorded far less frequently, either once (1, 4, and 7) or exclusively in one sample type: water (5) or bottom sediments (8 and 14).

In general, approximately the same pesticide concentrations were noted in the water column and bottom sediment samples from reservoirs ($p \geq 0.05$). The values of total pesticide concentrations for rivers and reservoirs were approximately consistent with each other ($p \geq 0.05$). However, there were differences between the rivers. Thus, the total concentration values in the Don River coincided with those of the reservoirs, while for the Sal River, these values were lower ($p \leq 0.05$ with the Don).

In the case of the Krasnodar Reservoir located in another region, all 14 pesticides were not detected simultaneously. Among the pesticide concentrations, active substances 2, 3, 5, 9, and 10 predominated. Another two substances (1 and 14) were identified only once.

Therefore, the Krasnodar Reservoir demonstrated, on average, lower levels of pesticide pollution than other study objects (Table 3).

Total toxicity values did not exceed 0.13. In the Krasnodar Reservoir, substances No. 2, 3, and 5 were found in some samples. Other pesticides have been identified at even lower levels.

Table 2: Concentrations of active pesticide substances for bottom sediments (II) and water layer (I) of the Don and Sal rivers in 2019

Active substance number	*A*	*B*		*C*		*D*	
	I	*I*	*II*	*I*	*II*	*I*	*II*
Don River							
	1.351*	no	0.459*	no	0.119*	no	No
	6.243*	8.309*	0.056*	2.488*	0.009*	0.223*	no
	0.018*	2.415*	1.049*	2.013*	0.907*	0.965*	0.103*
	no	0.820*	no	0.049*	no	no	no
	no	no	0.077*	no	0.041*	no	no
10	0.011*	0.111*	no	no	no	no	no
13	0.068**	2.711**	0.015**	1.357**	0.008	0.231**	no
Sal River							
	0.958*	0.819*	0.311*	0.510*	0.089*	no	no
	3.137**	6.039**	0.197**	5.198**	0.149	3.311	0.084**
	no	no	2.187*	no	1.980	no	0.020*
	no	0.101**	0.011**	no	0.007	no	no
13	0.005**	0.422**	0.113**	no	0.009	0.006	no
14	no	no	no	0.131*	no	no	0.007*

Table 3: Concentrations of active pesticide substances for bottom sediments (II) and water layer (I) of the Krasnodar Reservoir in 2019

Active substance number	*A*	*B*		*C*	
	I	*I*	*II*	*I*	*II*
	1.150**	3.210**	0.204**	1.150	0.053**
	2.423**	3.296**	0.026	1.035**	0.018**
	no	0.736*	1.759*	no	0.027*
	no	no	3.311*	no	1.200*
	no	no	0.119*	no	0.004*
	no	no	1.343*	no	0.011*
12	no	no	0.233*	no	0.122*
13	no	0.112*	0.059*	no	no

As a result, pesticide levels vary within rivers and reservoirs, but the most favourable situation was noted for the Krasnodar Reservoir.

Discussion

At present, there are several hundred pesticides in the range of agricultural businesses and farms, representing various classes of chemicals (Sarker et al., 2021). Various toxic substances, including herbicides, insecticides, and acaricides, can enter water bodies through the surface water, drainage and groundwater runoff, causing irreparable damage to aquatic ecosystems (Ahad et al., 2010). Despite the widespread consideration of the adverse effects caused by pesticides in scientific publications, most of them focus on organochlorine pesticides, which are prohibited in most countries nowadays (Emoyan et al., 2021). In this work, a large variety of pesticides were taken into consideration. Some are widely distributed in all water bodies, while others are present in the water column and bottom sediments throughout all seasons. These substances comprise imazethapyr, imidacloprid, and metribuzin. Organochlorine pesticides were characterized by widespread application, stable environmental status, and quite significant methodological developments (Taufeeq et al., 2021). In the present situation, with the chemical structure of pesticides applied to be generally different, it is necessary to continue these studies over large areas.

Various studies of toxicology aim primarily to describe the adverse effects of pesticides on biota (Oliveira et al., 2016). A key consideration is identifying the effects of pesticide pollution on biological systems, including aquatic organisms (Aamir et al., 2016). In such a case, the leading indicator developed is the pollutant threshold concentration. Below this value, the pollutant is not capable of causing substantial damage to living organisms of any organization level, from single-celled to multi-cellular. If the concentration of the pollutant is below the maximum allowable level, the pesticide is considered to have no effect on the organism (Robinson et al., 2016). Although pesticide levels in none of the water bodies studied significantly exceeded the MACs (according to the data obtained, the total pesticide count was 0.15-0.80 between seasons), prolonged exposure to the threshold values may result in their accumulation, followed by incorporation into the trophic chain and subsequent degradation of aquatic ecosystems.

In addition, organic matter contributes to the rapid development of microflora, oxygen concentration reduction, metal cations accumulation, and rapid growth of hydrogen sulphide concentration. This happened in 2018, when, as we found, the total number of pesticides was less than 6.5, which was almost 20 times higher than the average values for 2019. This may be due to the self-purification of water bodies. That is, they still have the capacity to reduce the concentration of pesticides relatively quickly. At the same time, most pesticides are removed from rivers and reservoirs in the Black Sea basin, which is rather closed and connected to the Mediterranean Sea by narrow straits. Therefore, pesticide levels in the Black Sea can be expected to increase significantly following these releases. This assumption was confirmed by previous data indicating that pesticide levels in samples taken off the coast of Russia exceeded MACs by dozens of times (Klenkin et al., 2006).

Consequently, a specific bottom sludge with hydrogen sulphide, metal cations, and ammonia is formed. As a result, various deleterious substances, including pesticides, accumulate in plant and animal organisms and negatively affect the growth rate, life expectancy of living organisms, and their species' diversity (Emoyan et al., 2021). Other substances replace particular plant and animal species, accelerating the growth rates of phytoplankton and zooplankton. The result of these processes is the eutrophication of water bodies. Although rivers and lakes are capable of self-cleaning under moderate loads, degradation processes are irresistible under severe emissions. Water in gaseous form (fog) can accumulate large amounts of pesticides, more than in a simple gas phase without water droplets by 3,000 times (Mulk et al., 2017).

Possible Ways to Reduce Pesticide Pollution in the Krasnodar Krai (Russia)

The quickest path for pesticides to enter the sea water zone is through surface water. As we have observed, the content in surface water changes most rapidly. Given the frequency of natural disasters in the region, the spread of moderate amounts of pesticides is expected to be associated with periods when the probability of such disasters is low. Otherwise, large quantities of pesticides can enter waterways and reservoirs at the same time and then enter the sea. Bottom sediments are more inert. However, when water bodies dry up, pesticides can also enter the sea rapidly with the increased dust levels. Therefore, it is also recommended to avoid drying water bodies (reservoirs) with already high levels of pesticides.

Conclusions

The results of this study allow stating that different amounts of pesticides representing modern classes of these compounds were found in the water column and bottom silt sediments. Bottom sediment can contain 10 to 20 times more pesticides than the water column, and the composition of these pesticides may differ from that of the water column. At the same time, pesticide concentrations detected did not exceed the maximum allowable levels. The highest levels were reported for diflufenican, imazetapyr, imidacloprid, iprodione, metribuzin, pencicuron, famoxadone, flubendiamid, flumioxazine, ciprosulfamide, etofumezate. In 2019, there was an increase in pesticide concentrations, increasing from winter to spring, followed by a decline to fall. Compared to 2018 data, pesticide levels decreased in terms of total quantity and total toxicity. Despite the increased prevalence of pesticides in 2019, some of these pesticides have only been encountered once. Consequently, pesticide pollution in the hydrosphere of the study areas has not yet had a negative effect on biota. Other studies should consider that pesticide concentrations are borderline and that long-term accumulation of pesticides can degrade aquatic ecosystems. Consequently, surface water pollution by SFD is chronic, although the hazard is currently low. The fact that pesticides have been detected in many samples collected indicates a potential threat for severe pollution in the future.

Conflict of Interests

The authors declare that they have no conflict of interests.

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