CHEMISTRY JOURNAL OF MOLDOVA. General, Industrial and Ecological Chemistry. 2025, 20(2), 7-14
ISSN (p) 1857-1727 ISSN (e) 2345-1688
http://cjm.ichem.md
https://doi.org/10.19261/cjm.2025.1341

FLUORIDE TRENDS IN THE DNIESTER RIVER AND DUBASARI RESERVOIR OVER 2011–2024 PERIOD

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Abstract. The paper presents data on fluoride concentrations in the Dniester River and Dubasari reservoir, the waters of which are used for multiple purposes, including use as a source of drinking water. Water samples were taken seasonally from 2011 to 2024. The concentration of fluoride ions was determined photometrically using an acidic solution of zirconyl chloride and alizarin red S. Fluorides ranged from 0.05 to 1.07 mg/L in the Dniester sector from Naslavcea (entry point of the river into the territory of the Republic of Moldova) to Dubasari reservoir, and from 0.05 to 0.93 mg/L downstream of the Dubasari dam. They varied from 0.02 to 0.95 mg/L in the reservoir waters. Mean annual concentrations of fluorides in the entire Dniester River, including the Dubasari reservoir, oscillated from 0.15±0.07 to 0.81±0.10 mg/L during the study period. Concentrations below 0.5 mg/L were recorded in 76.6% of analysed water samples. Higher concentrations were observed during periods of low waters, when the river was predominantly supplied by groundwater. In most cases, due to the modified regime of the Dniester resulting from the river regulation, no clear seasonal dynamics were recorded.

Keywords: fluoride ion, surface water, Dniester River, Dubasari reservoir.

Received: 8 July 2025/ Revised final: 3 October 2025/ Accepted: 6 September 2025

Introduction

Fluorine, like many other elements, is beneficial to human health in trace amounts, but can be toxic in excess. Elemental fluorine does not occur in nature, due to its extremely high chemical reactivity; instead, it is found as fluoride mineral complexes, mostly in combination with calcium and aluminium. The fluoride ion (F^-) accounts for over 95% of the total fluoride present in freshwaters (with total dissolved solids less than 500 mg/L), with the magnesium-fluoride complex (MgF^+) typically being the next most prevalent form [1]. Natural sources of fluorides in freshwaters are the weathering of fluoride-containing rocks and soils through which groundwater flows, emissions from volcanoes, and marine aerosols [2]. Calcium fluoride (CaF_2), also known as fluorite or fluorspar, is used in the production of aluminum, steel, glass, enamel and bricks, as well as in the chemical industry as a raw material for the manufacture of hydrofluoric acid and anhydrous hydrogen fluoride. Fluorapatite ($Ca_3(PO_4)_3F$), another important calcium- and fluoride-containing mineral, is used as a source of phosphates in the fertiliser industry [3]. The industrial effluents, application of phosphate fertilisers and fluoride-containing pesticides are the anthropogenic sources of inorganic fluorides in surface waters.

Fluorides are among the few chemicals for which the contribution from drinking water to overall intake is an important factor in preventing health problems. World Health Organisation has established a guideline value of 1.5 mg/L as the upper limit for fluorides, as extensive research revealed that concentrations above this level increase the risk of dental fluorosis and, at much higher concentrations - of skeletal fluorosis [4]. Drinking waters with higher concentrations of fluorides require defluoridation. In the case of low concentrations of fluorides in drinking water, different ways of ensuring the necessary intake of fluorides are used - from dental preparations enriched with fluorides (*e.g.*, toothpaste, tablets *etc.*) to artificial fluoridation. In the latter case the final concentrations of fluorides shall range between 0.5 and 1 mg/L [4]. The national legislative framework of the Republic of Moldova also sets the concentration of fluorides of 1.5 mg/L as a maximum allowable one for drinking water [5].

As fluoride ions are not among the main anions in surface freshwaters, from quantitative point of view, their concentrations are usually not regulated. This issue has typically raised concern only in the areas with intense natural release of fluorides into environment or anthropogenic fluoride pollution. Thus, only few countries established national water quality criteria for the protection of freshwater biota. For example, Spain has set a maximum allowable concentration of 1.7 mg F^-/L, Philippines - 1.0 mg F^-/L, while Canada has the most restrictive water quality benchmark of 0.12 mg F^-/L [6].

Investigations of fluoride content in environmental components on the territory of the Republic of Moldova have mainly focused on groundwater, which is used for drinking purposes. Mapping of extensive data collected between 2015 and 2019 revealed that 12 administrative-territorial units of the country meet the criteria of endemic areas with a pronounced excess of fluoride in groundwater. Among them, Ungheni, Nisporeni, Straseni, Glodeni and Falesti counties, with average fluoride concentrations ranging from 3.04 to 9.2 mg/L, are the most affected [7]. An early study in the region, conducted at the beginning of the 21^{th} century, revealed that high and low fluoride waters from different aquifer horizons can be available in the same location, as rock geochemistry exerts a major control on fluoride concentration in groundwater [8].

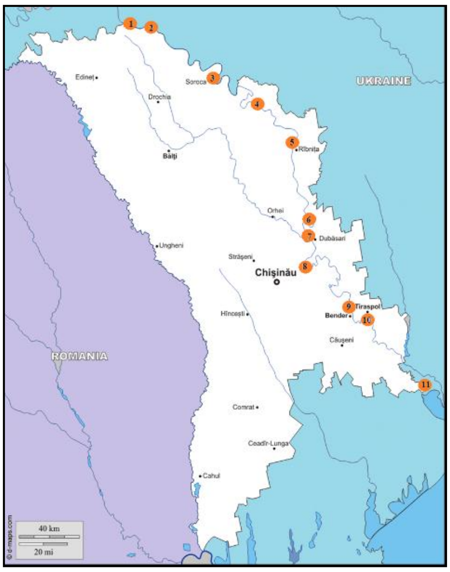
Statistics for the 2003–2022 period showed that the supply of drinking water to Chisinau municipality - capital of the Republic of Moldova - is ensured by surface waters, specifically the waters of the Dniester River, which accounted 94% of the total supplied volume in the given period [9]. The Dniester waters are captured at the Vadul lui Voda station, located in the Lower Dniester, immediately downstream of the Dubasari reservoir. The last is a relatively old semi-flowing water body, with a length of 128 km and a surface of 6570 ha, built on the Dniester between Camenca and Dubasari towns in 1954 [10].

The purpose of this study was to analyse the fluoride concentrations in the water of Dniester River and Dubasari reservoir over the last decade, by taking into consideration the influence of other hydrochemical parameters, such as pH, mineralisation, major ions and water hardness. Analysis of the spatio-temporal dynamics of fluorides provide support for the prediction of environmental risks and optimisation of water management in a region where the reservoir serves as a key source of both water supply and energy.

Experimental

Research of the chemical composition of the Dniester River and Dubasari reservoir waters was carried out over the 2011–2024 period.

Within the study, water samples were collected from the Dniester River within the territory of the Republic of Moldova near settlements located upstream the Dubasari reservoir (Naslavcea, Valcinet, Soroca and Camenca), from the reservoir itself (Erjovo, Goieni and Cocieri) and downstream the Dubasari HPP dam (Vadul lui Voda, Varnita, Suclea and Palanca). This sampling strategy provided a comprehensive picture of the state of water resources in various stretches of the Dniester and in the reservoir (Figure 1).



	Sampling station	*N*	*E*
1	Naslavcea	48˚29'21"	27˚34'54"
2	Valcinet	48˚26'75"	27˚41'85"
3	Soroca	48˚08'35"	28˚18'13"
4	Camenca	48˚00'45"	28˚42'17"
5	Erjovo	47˚50'10"	29˚01'17"
6	Goieni	47˚22'28"	29˚09'12"
7	Cocieri	47°16'58"	29°07'31"
8	Vadul lui Voda	47°05'19"	29°05'22"
9	Varnita	46°52'53"	29°28'59"
10	Suclea	46°48'06"	29°40'04"
11	Palanca	46°24'62"	30°07'92"

Figure 1. Coordinates of sampling stations.

Water samples were collected from the surface layer (0.25–0.50 m) and analysed within 24 hours. A total of 444 samples were collected during the study period, excluding repeated samples at some stations. Collection and primary processing of water samples were carried out according to SM EN ISO 5667-6:2017 [11].

Fluoride concentration in water was determined photometrically by using an acidic solution of zirconyl chloride and alizarin red S. The method is based on the ability of fluoride ions to form a colourless zirconium complex $[ZrF_6]^{2-}$. The released alizarin sulfonic acid contributes to the appearance of yellow colour. Depending on the fluoride ions concentration, the colour of the solution changes from light pink to yellow. Due to its simplicity and high sensitivity (0.05 mgF^-/L), this method is suitable for determining fluorides at low concentrations in water [12]. The colour intensity was determined using a Specord 210 plus spectrophotometer (Analytik Jena).

Water temperature and pH were determined in situ using a portable pH meter (Consort C5030), in accordance with the national standard SM SR EN ISO 10523:2014 [13]. The concentrations of dissolved oxygen, major ions, biogenic elements, soluble silicates, and suspended matter were analysed under laboratory conditions [14].

The Pearson correlation coefficient (r) between the fluoride ions concentration and other hydrochemical parameters was calculated using Excel 2007. This dimensionless index ranges from -1.0 to 1.0 and measures the linear relationship between two data sets.

Results and discussion

The gathered data set on the concentrations of fluorides in the Dniester River and Dubasari reservoir allowed identification of their spatio-temporal variations, including seasonal dynamics. Concentrations of fluorides ranged from 0.05 to 1.07 mg/L in the Dniester sector from Naslavcea to Dubasari reservoir, and from 0.05 to 0.93 mg/L downstream of the Dubasari dam. The frequency of recorded concentrations of fluorides was as follows (n= 444): less than 0.5 mg/L in 76.6% of cases, between 0.5 and 1.0 mg/L in 22.5%, and above 1.0 mg/L in 0.9% of cases, with the highest recorded value being 1.07 mg/L (Table 1).

Analysis of the average annual concentration of fluorides in both the Dniester River and Dubasari reservoir over the last fourteen years put in evidence three periods. During 2011–2015, the annual mean concentration of fluorides remained within narrow limits (0.15–0.25 mg/L). A clear upward trend was observed (0.71–0.81 mg/L) in the second period, 2016–2018. It is worth to mention that concentrations exceeding 1.0 mg/L were recorded, about four times higher than in the first period. Since 2019, the average concentration of fluorides has sharply decreased - by 5.4 times -, remaining below 0.30 mg/L in the Dniester and 0.33 mg/L in the Dubasari reservoir (Table 1, Figure 2*(a)*).

Table 1

Annual dynamics of fluorides in the Dniester River, including Dubasari reservoir, mg/L.

Year	*Minimum (mg/L)*	*Maximum (mg/L)*	*Mean (mg/L)*	*St. dev.**
2011	0.05	0.43	0.25	0.11
2012	0.05	0.26	0.15	0.07
2013	0.05	0.31	0.15	0.08
2014	0.05	0.43	0.24	0.13
2015	0.08	0.55	0.25	0.14
2016	0.43	1.07	0.76	0.18
2017	0.57	1.04	0.71	0.13
2018	0.63	0.98	0.81	0.10
2019	0.03	0.94	0.26	0.24
2020	0.06	0.49	0.25	0.13
2021	0.06	0.46	0.25	0.09
2022	0.13	0.40	0.25	0.08
2023	0.09	0.42	0.23	0.07
2024	0.02	0.67	0.30	0.18

* *St. dev. – standard deviation*

Considering the entire 2011–2024 period, the highest concentrations of fluorides in the upper section of the Dniester River were recorded at Valcinet station (1.07 mg/L) and in Dubasari reservoir at Erjovo station (0.95 mg/L) during the spring of 2016. In the lower section of the Dniester (Vadul lui Voda - Palanca), the highest concentration of fluorides (0.93 mg/L) was observed in the winter of 2019. The data obtained emphasizes the importance of continuous monitoring of water quality along the entire length of the water bodies from the Dniester basin.

Retrospective analysis of the data shows that the dynamics of fluorides in the Dubasari reservoir has undergone significant changes over the past decades. Thus, if before 1980 fluoride concentrations varied in the range of 0.05–0.35 mg/L [15], then in the 1980–1986 period an increase to 0.40–0.90 mg/L was observed [16]. In the 2011–2024 period, concentrations of fluorides in this water body fluctuated even more - ranging from 0.02 to 0.95 mg/L.

Comparative analysis of fluorides during the 2011–2014 period in the upper (Erjovo) and lower (Cocieri) sections of the reservoir showed similar ranges of values: 0.03–0.91 mg/L and

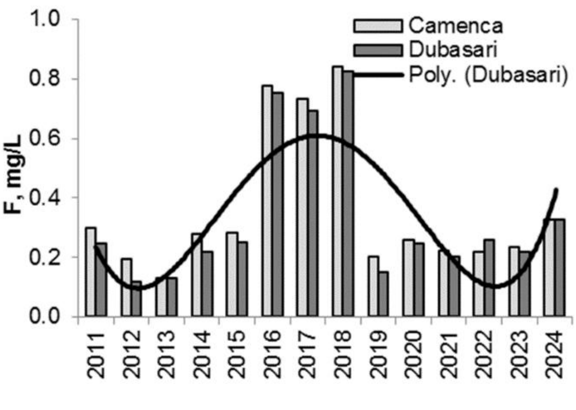
0.02–0.89 mg/L, respectively. This fact indicates the absence of significant changes along the reservoir and can be explained by the homogeneity of fluorine sources. The hydrochemical regime of Dubasari reservoir is primarily determined by the chemical composition of its main source of water supply - the Dniester River. Concentrations of fluorides in water at the Camenca sampling point, located at the entrance of Dubasari reservoir, were similar to those within the reservoir itself. However, the average concentrations of fluorides in the reservoir were slightly lower than at the point of direct Dniester inflow (Figure 2*(a)*). For example, the concentration of fluorides at Camenca station in 2016 was equal to 0.78 mg/L, compared to 0.75 mg/L in the Dubasari reservoir; in 2018, it was 0.84 mg/L and 0.82 mg/L, respectively. An exception occurred in 2022, when 0.26 mg/L of fluorides were recorded in the reservoir, with 0.04 mg/L higher than at Camenca sampling station (0.22 mg/L). The average difference between the stations for the 2011–2024 period was of +0.03 mg/L.

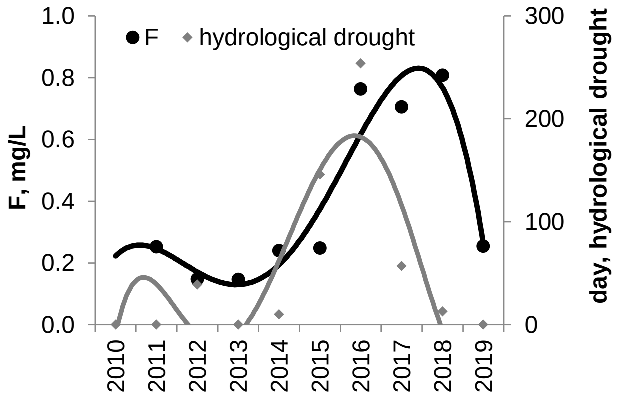
In general, higher concentrations of fluorides have been observed during the 2016–2018 hydrological drought (Figure 2*(a)* and *(b)*), when, according to the Hydrological Characteristics Yearbook 2019 [17], the highest flow of water in the Dniester did not exceed 160 m^3/s. This indicates an increased contribution of groundwater to the fluoride load in the Dniester and the reservoir.

Analysis of the seasonal dynamics of concentration of fluorides in the waters of the Dniester River reveals various patterns which are essential for understanding the anthropogenic impact on the aquatic ecosystem. Thus, in the 1980–1990 period the highest concentrations were recorded in summer, which was partly associated with the increase in water temperature, but mainly with the intensive use of phosphorus fertilisers, which often contain fluoride compounds as accompanying components. Concentrations of fluorides decreased during periods of flooding compared to low-waters periods, which is explained by a decrease in the share of groundwater supply [16].

A pronounced annual and seasonal variability of concentration of fluorides was demonstrated by the data recorded from 2011 to 2024. However, the classic seasonal dynamics of fluorides, characterized by the lowest concentrations during the flood period and the highest ones during the low-water period, was observed only in 2013 and 2023 (Figure 3). Higher concentrations of fluorides were observed during the autumn-winter seasons, especially in 2016–2018, when they varied within 0.78–0.90 mg/L. This may be attributed to two factors: the processes of accumulation of fluorine-containing compounds and the reduction of the river flow as a result of hydrological drought, leading to a concentration effect.

In the spring of 2016 and 2017, following the special release of water from the Novodnestrovsk Hydropower Plant reservoir (also called Dniester HPP-1), necessary for successful fish spawning in Dubasari reservoir and the Lower Dniester, fluoride concentrations reached 0.95 mg/L. At the same time, the lowest concentrations were observed during the summer. It is important to mention that the water released from the Novodnestrovsk HHP reservoir is being pumped from depths of 9–10 m [10].





(a) ***(b)***

Figure 2. Average concentrations of fluorides in the Dniester river at Camenca station and in Dubasari reservoir, mg/L *(a)*; changes in concentrations of fluorides in Dubasari reservoir as a function of the number of days of hydrological drought *(b)*.

Such strong regulation of the Dniester River flow by hydroelectric facilities in recent years has led to the disappearance of natural spring floods. The typical summer-autumn low waters have transformed into almost multi-month hydrological droughts, and winter (January–February) water levels in some years exceeded the typical spring floods of previous years. This undoubtedly affected the seasonal dynamics of the chemical composition of water, including the fluorine content.

An increase in concentrations of fluorides was observed from spring to autumn in 2015, when duration of droughts began to gradually increase in the Dniester basin. A similar trend was observed in 2019, when, during the flood period, water level in some locations rose to 3 m, while during the low-water period the runoff into the reservoir was 50–60% below normal [17]. It should also be noted that concentrations of fluorides sharply increased in the winter of 2019 (up to 0.91 mg/L), during intensive water discharge from the Dniester HPPs reservoirs. Later, since the spring of the same year, fluoride levels gradually stabilized at 0.14–0.45 mg/L (Figure 3).

The migration capacity of fluorine in natural waters largely depends on temperature, the content of calcium, magnesium, and sodium ions, alkalinity, mineralisation [18], and the presence of ion-exchange materials such as clays [3].

Increased concentrations of calcium and magnesium ions have a significant impact on the migration capacity of fluorine, as they form poorly water-soluble compounds such as CaF_2 and MgF_2, while an increase in sodium ions, mineralisation and the alkaline environment promote the fluorine migration, as noted by several authors from other regions. For example, Petrenko, D.B. *et al*. [19] found that in urbanized areas of the Moscow region, low-calcium hydrocarbonate-sodium surface waters contained higher concentrations of fluorides compared with waters with a relatively high content of calcium.

The intra-annual dynamics of fluorides are also influenced by their high tendency to form complexes, adsorption of fluoride compounds by suspended substances and bottom sediments, as well as their accumulation by hydrobionts, including higher aquatic vegetation. These processes can contribute to a decrease in concentration of fluorides during the summer.

The mineral composition of the Dniester water entering the Dubasari reservoir has remained largely unchanged over the 2011–2024 period. There always was a clear predominance of calcium ions (47.1–76.2 mg/L) among the cations, while magnesium ions did not exceed 17.6 mg/L, and the combined sodium and potassium concentrations reached up to 35.8 mg/L. Hydrocarbonate ions, which prevailed among the anions, varied within 137.3–234.9 mg/L. Content of sulphates and chlorides did not exceed 79.0 and 28.9 mg/L, respectively. Thus, according to the classification of Aleokin, O.A. *et al*. [20], the Dniester water at Camenca station belonged to the class of hydrocarbonate waters of the calcium group, the second-third type (C^{Ca}_{II-III}). Its highest mineralization reached 433.5 mg/L, and hardness - 5.25 mmol/L.

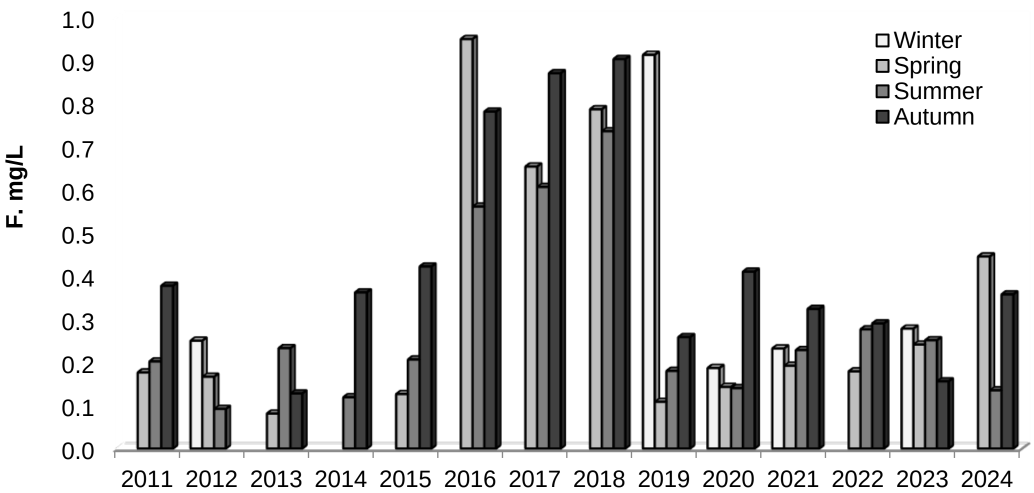


Figure 3. Seasonal dynamics of fluorides in the Dniester River, including Dubasari reservoir.

Analysis of average annual concentrations of ammonium nitrogen, suspended organic solids, and soluble silicates in the Dniester River waters within the territory of the Republic of Moldova over the 2011–2024 period, indicates significant changes in the aquatic ecosystem (Figure 4). Concentrations of N–NH_4^+ varied considerably, reaching the highest values in 2015 (0.46 mg/L) and 2018 (0.50 mg/L). Soluble silicate concentrations remained relatively stable, with noticeable peaks in 2015 and 2018. The data obtained indicate a complex impact on water quality, driven by increasing anthropogenic load, climatic and hydrological changes in the river regime.

Average multiannual concentrations of suspended organic matter did not exceed 0.92 mg/L. The most noticeable increase occurred in Dubasari reservoir in 2018–2019, when the concentration of the organic phase of suspended matter reached an abnormal value (2.0 g/L), as a result of intense algal bloom, especially in the middle section of the reservoir.

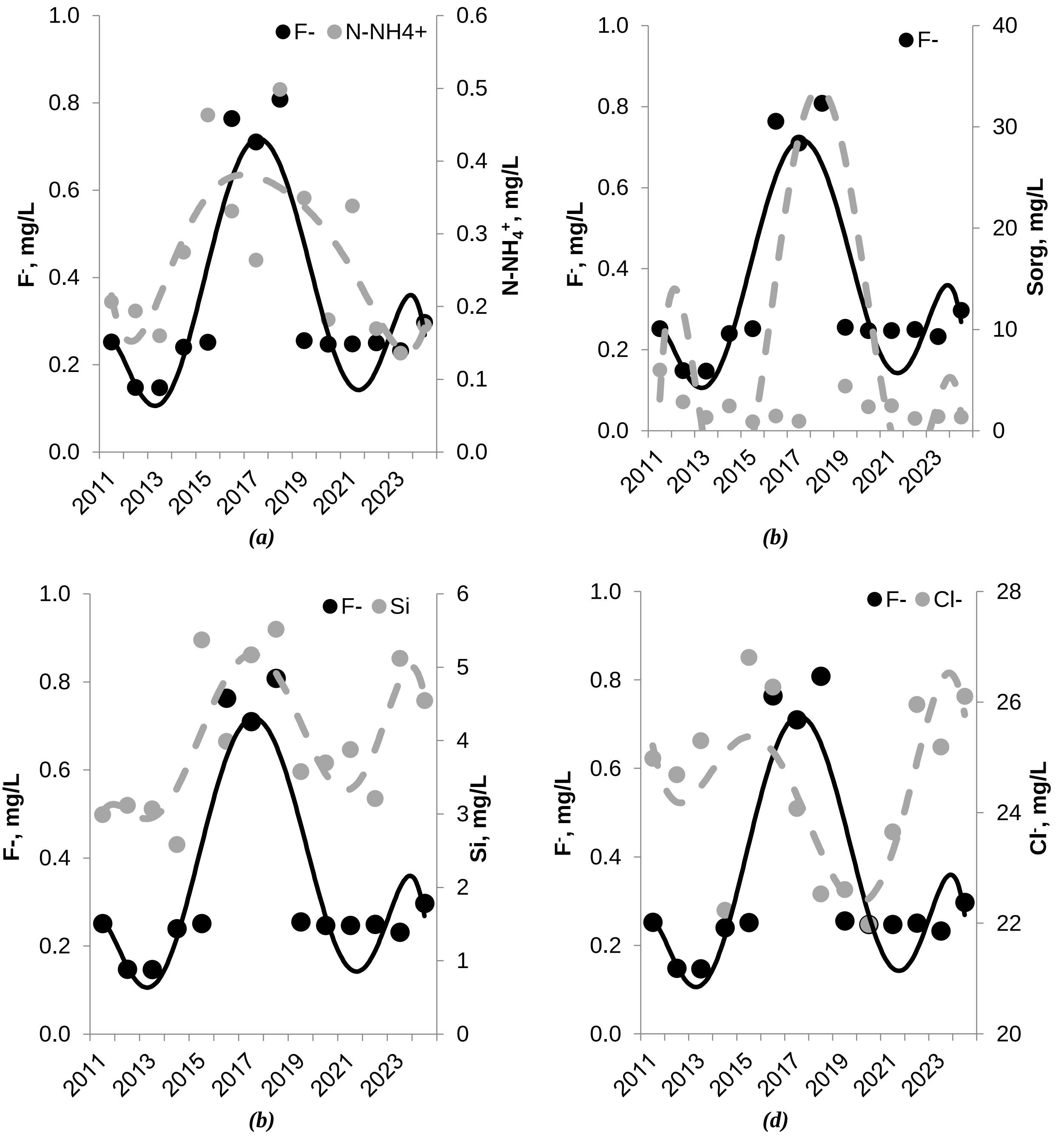


Figure 4. Dynamics of fluorides, ammonium nitrogen *(a)*, suspended organic solids *(b)*, silicates *(c)* and chlorides *(d)* in the Dniester River, including Dubasari reservoir.

Fluorine and chlorine, which belong to the group of halogens, demonstrate fundamentally different migration properties in aqueous systems, due to their electron configurations and reactivity. Fluorine, having a high electronegativity (4.0), forms strong bonds with calcium and aluminium (CaF_2, AlF_3), which limits its mobility in neutral waters [21]. Unlike fluorine, chlorine is present mainly as highly mobile Cl^- ions, whose concentrations (*e.g.*, 21.98–26.80 mg/L in the Dniester River) reflect the natural dissolution of halite (mineral form of sodium chloride).

Correlation analysis shows a moderate positive relationship between fluoride ions and ammonium nitrogen (r= +0.43), and a strong positive correlation (r= +0.72) between fluoride ions and suspended organic solids (Table 2). The moderately strong positive correlation between fluorides and silicon reflects their joint release into the water from aluminosilicates, as well as the stabilization of fluoride ions by soluble forms of silicon (H_4SiO_4), which can lead to the formation of complexes such as SiF_6^{2-}. The weak negative correlation between ionic forms of fluorine and chlorine emphasizes the need for separate monitoring of halogens when assessing the contribution of natural and anthropogenic factors to the pollution of water resources. To mention that Petrenko, D.B. *et al*. [19] reported no correlation between F^- and Ca^{2+}, Mg^{2+}or Cl^-, but found a moderate positive correlation with NH_4^+.

Table 2

The Pearson correlation coefficients (r) between fluorides and ammonium nitrogen, suspended organic solids, silicates, and chlorides in the Dniester River, including Dubasari reservoir.

Ions	F^-
N-NH$_4^+$	+0.43
Sorg	+0.72
Si	+0.65
Cl$^-$	−0.32

Currently, there is no evidence of industrial pollution of the Dniester River and Dubasari reservoir with fluorides. A potential source may be the long-term application of phosphate fertilizers on agricultural lands and further run-off of fluorides into the adjacent water bodies. According to official statistics of the Republic of Moldova, the amount of phosphoric fertilizers (active substance) used in agricultural enterprises varies significantly from year to year: for example, 23.8 thousand tonnes were applied in 2020 and 17.0 thousand tonnes - in 2023 [22]. Estimating the amount of fluorides released into the environment based on the quantity of fertilizers applied is difficult, as their chemical composition depends on the type of apatite used in their production.

Conclusions

The long-term dynamics of fluorides in the Dniester River and Dubasari reservoir has been assessed, by taking into account the intensive regulation of the river by the Dniester hydroelectric complex, built in Ukraine upstream of the river's entry into the territory of the Republic of Moldova. Overall, the concentrations of fluorides ranged from 0.05 to 1.07 mg/L throughout the Dniester River, including the Dubasari reservoir, with mean annual values varying from 0.15±0.07 to 0.81±0.10 mg/L over the investigated period.

Concentrations of fluorides recorded between 2011 and 2024 period never exceeded the maximum allowable concentration of 1.5 mg/L, established by the World Health Organisation for drinking water. In 76.6% of the analysed water samples, the concentration of fluorides was less than 0.5 mg/L, and in 99.1% of cases, it did not exceed 1.0 mg/L.

The analysis of the seasonal dynamics of fluorides proved that the fluctuation of their concentration are still influenced by the hydrological regime. A consistent relationship has been established between fluorides and silicates in the Dniester River waters, due to their geological relationship, namely, their origin from silicate rocks. In contrast, the weak correlations between fluorides and ammonium nitrogen, suspended organic solids and chlorides emphasize the independence of their sources and the need for their separate monitoring, in order to assess the contribution of natural and anthropogenic factors to aquatic ecosystem pollution.

Acknowledgments

The research was carried out in the framework of the AQUABIO project (2020–2023), research subprogram 010701 of the Moldova State University (2024–2027), and the international project BSB165 HydroEcoNex (2014–2020).

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