Quantitative analysis of PFAS distribution in aqueous environmental samples of the Hungarian section of the Danube River.

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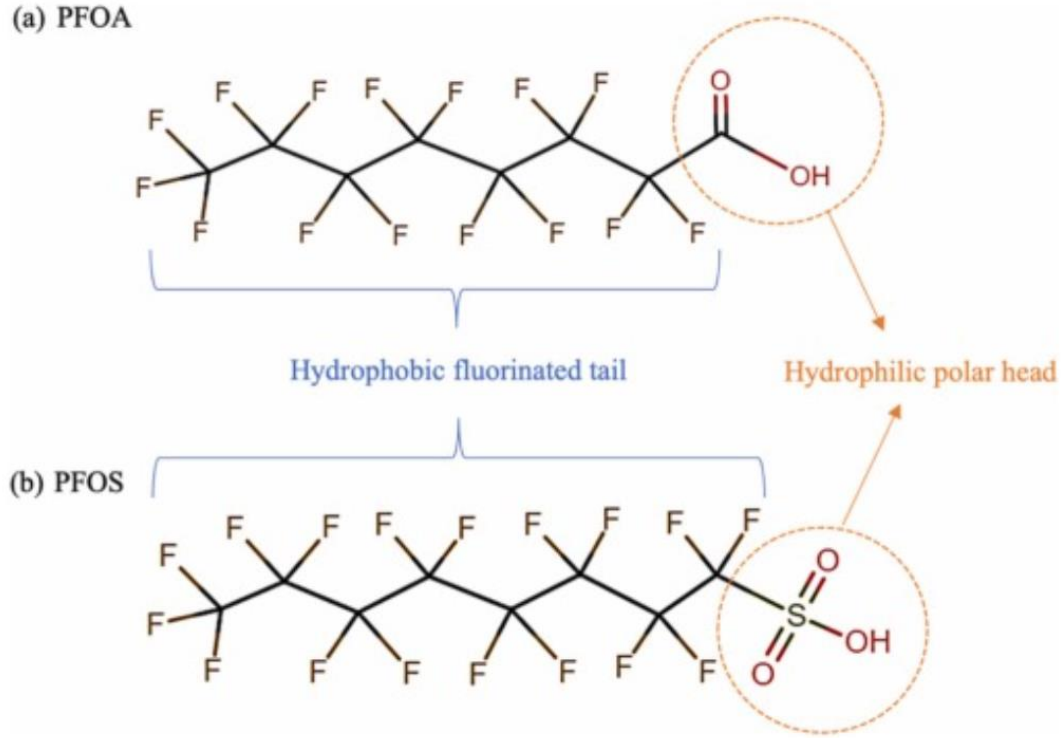
What are PFAS?

Per- and Polyfluoroalkyl substances (PFAS) is any substance that contains at least one fully fluorinated methyl group ($-CF_3$) or fully fluorinated methylene group ($-CF_2$) without any hydrogen, chlorine, bromine or iodine atom attached to it.

~OECD 2021

They are also know as “forever chemicals” and are anthropogenic highly stable chemical compounds that have been in use since the 1940s



These chemicals are characterised by:

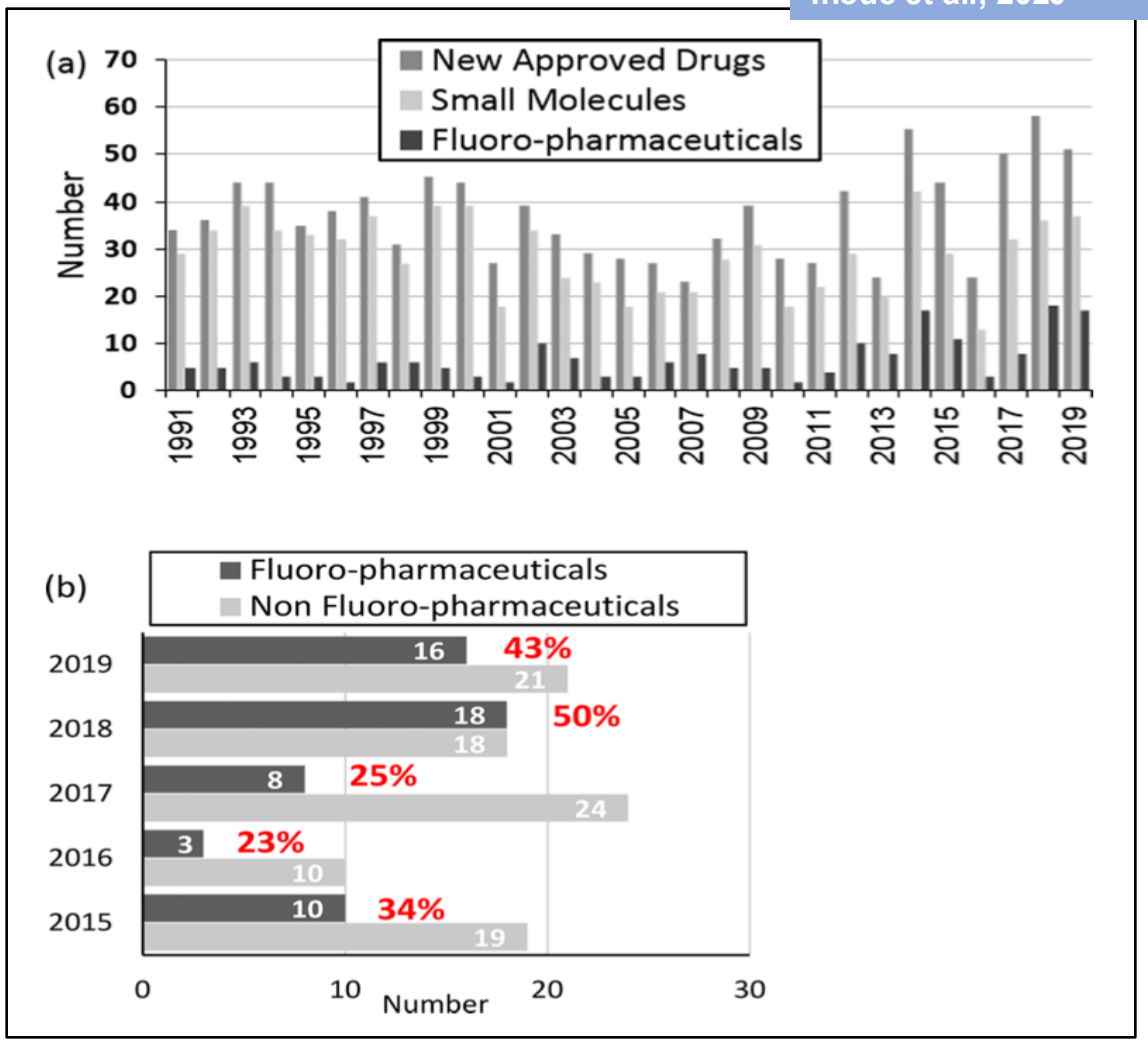
- Strong C-F bond
- Hydrophilic head group
- Hydrophobic and lipophobic tails
- Repels oil, grease, water and heat
- Persistent, resist degradation, bioaccumulative and toxic

Introduction-II

Lithium Ion Battery (LIB) production

https://www.sshhzlaw.com/blog/2022/03/the-rising-problem-of-forever-chemicals-in-u-s-drinking-water/

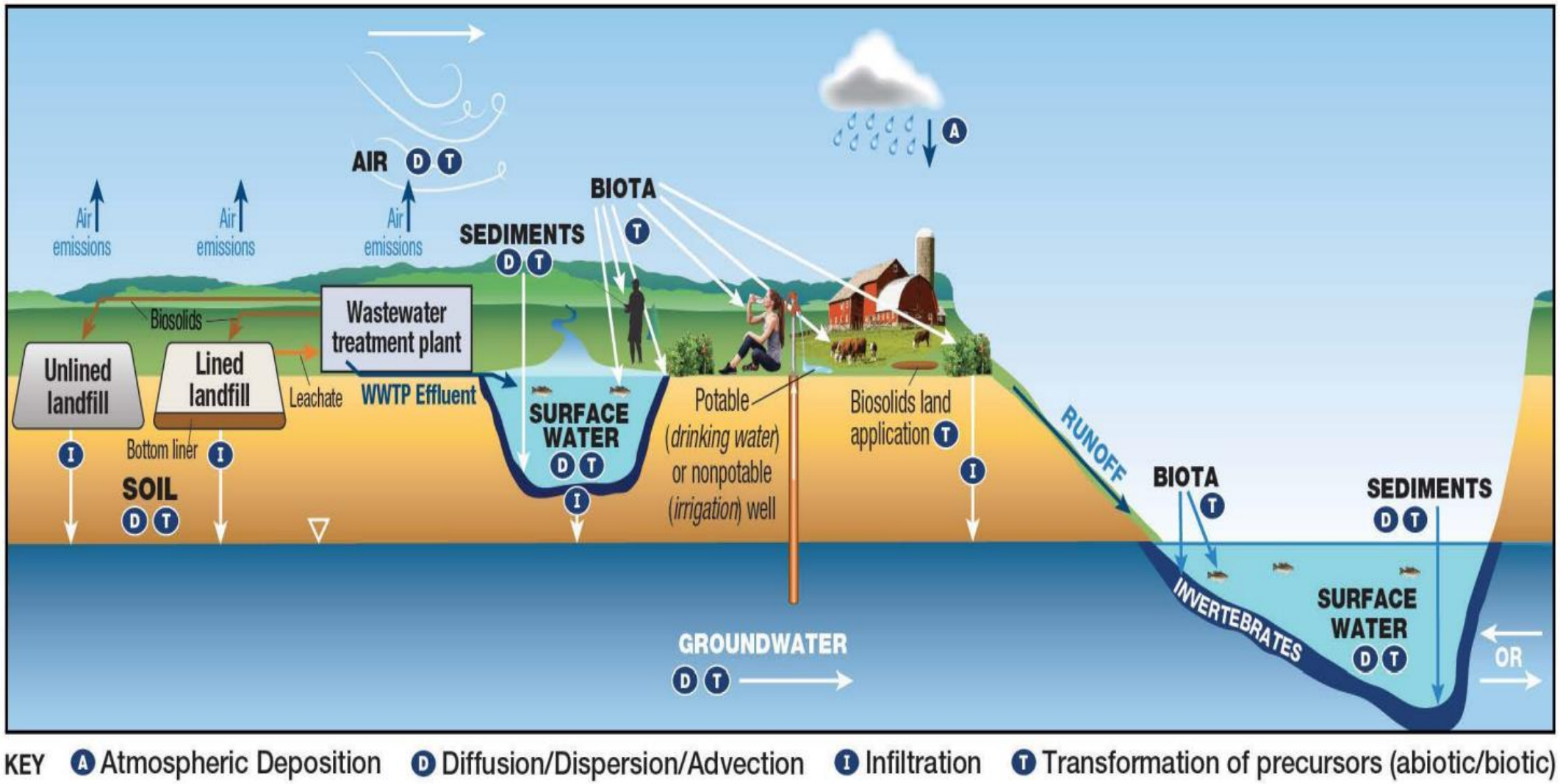
Inoue et al., 2020



(a) Prevalence of fluoro-pharmaceuticals among globally registered drugs. **All pharmaceuticals = 1072 compounds, small-molecule drugs = 839 compounds, and fluoro-pharmaceuticals = 191 compounds**

(b) Data for small-molecule drugs over the past five years.

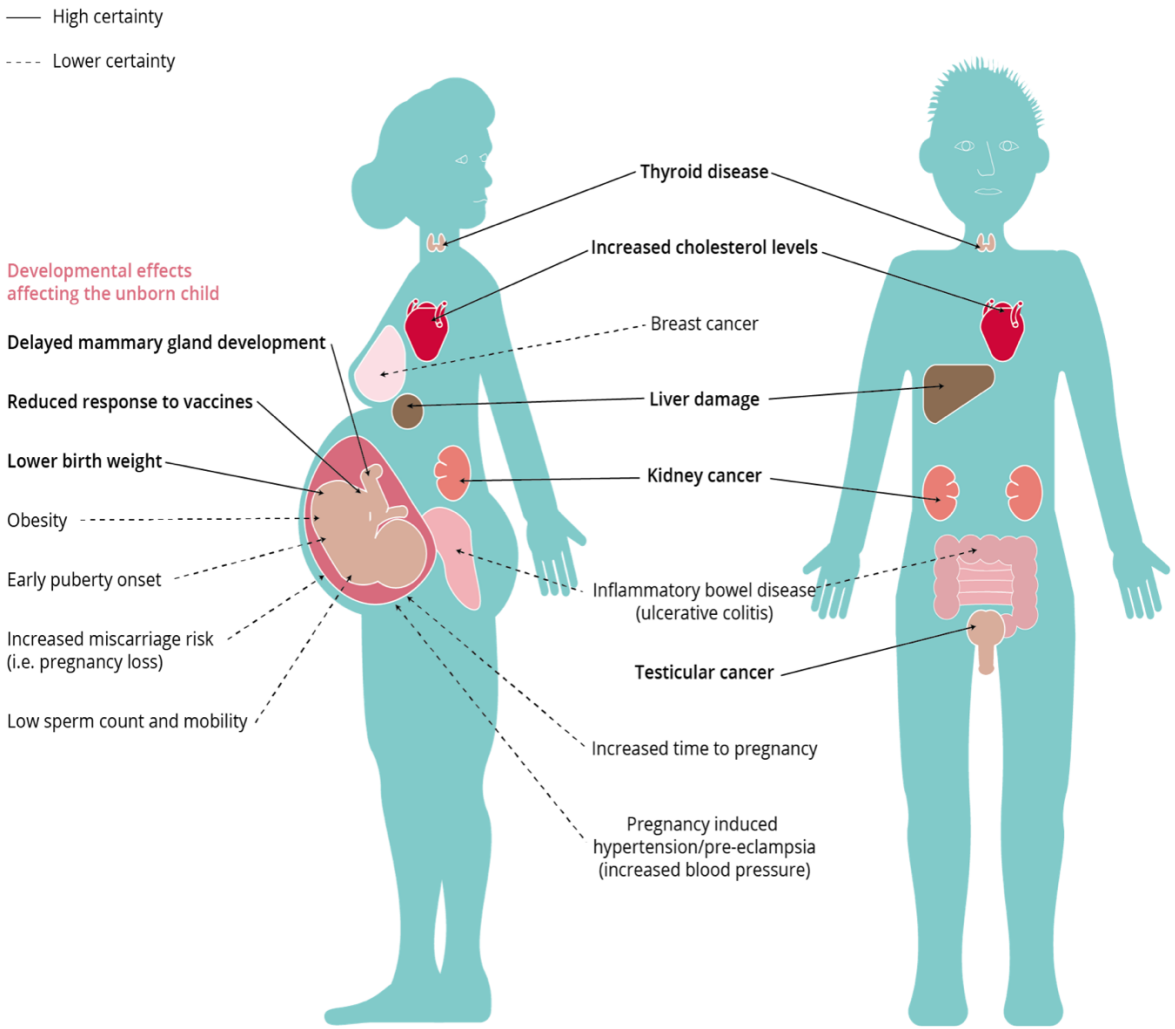
PFAS cycle and sources of exposure

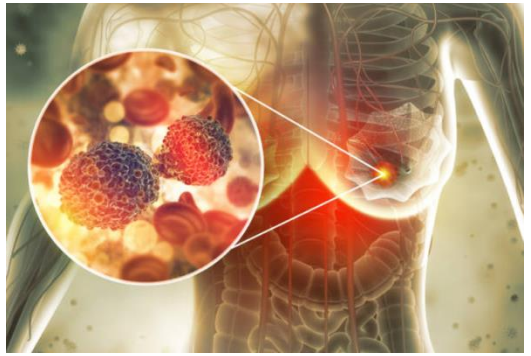


ITRC Fact sheet "Environmental Fate and Transport of Per - and Polyfluoroalkyl Substances", Oct. 20, 2022.

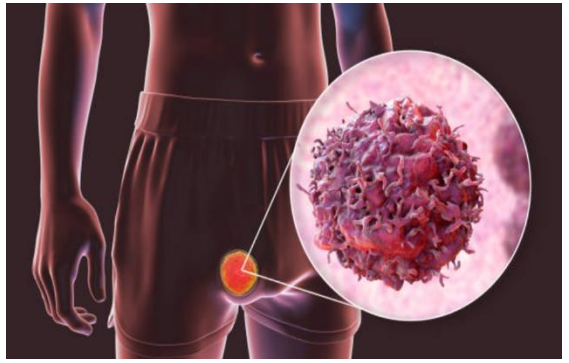
Introduction-IV

PFAS Detrimental Health Effects

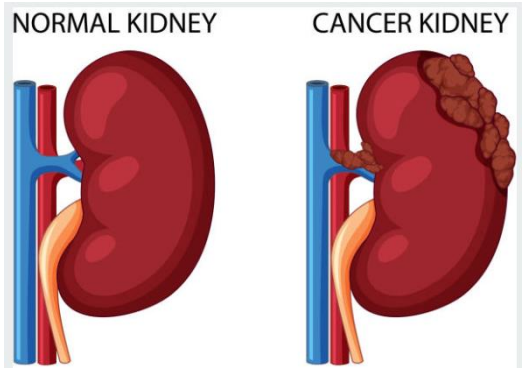




Breast cancer



Testicular cancer

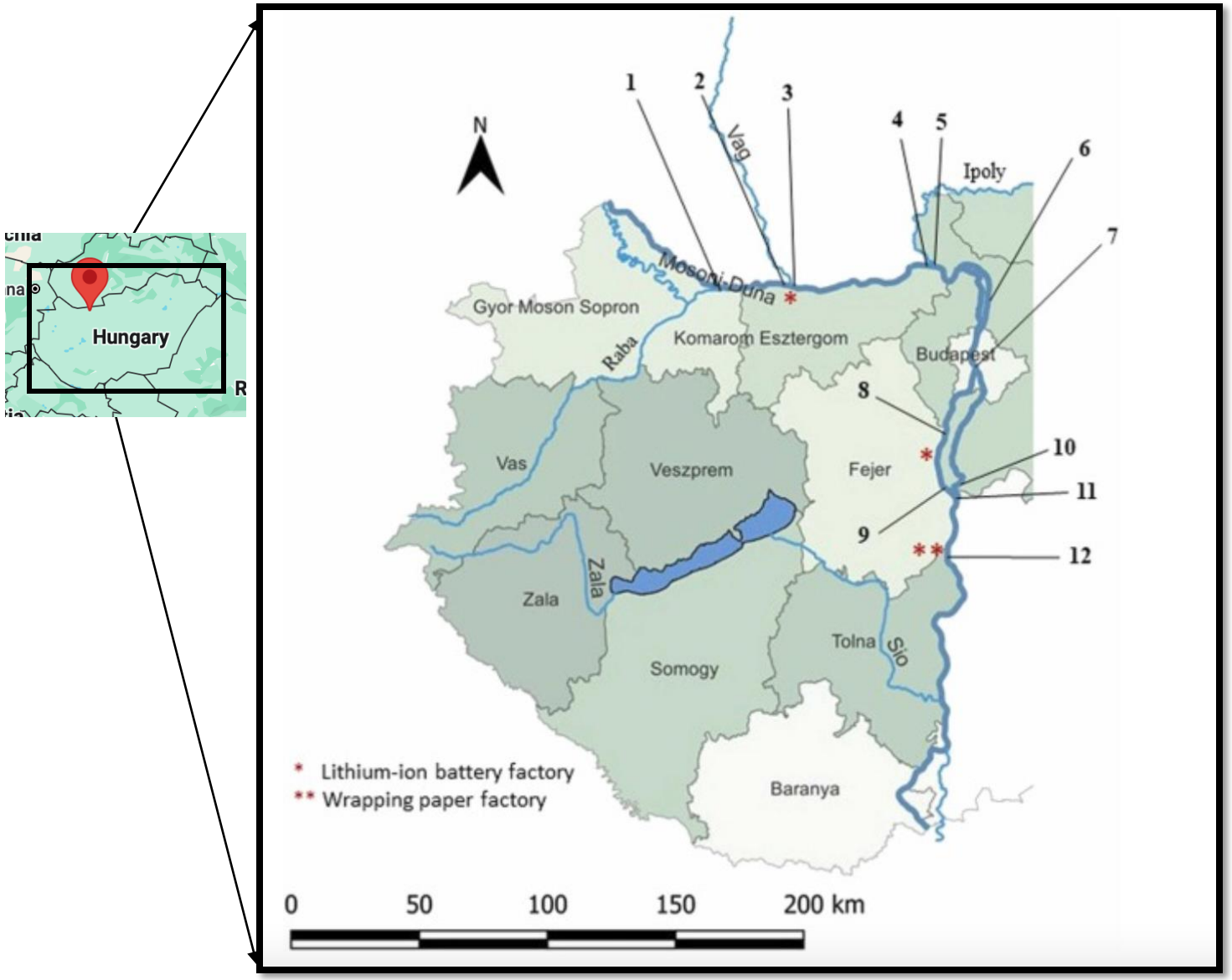


Thyroid disease

Regulatory Alignments

Country	Regulations
Europe	• In 2009, the European Union (EU), through the European Chemicals Agency (ECHA), regulated PFOS as a POP. • In 2017, the EU banned the sale, use, and import of PFOA, its salts and PFOA-related substances through Annex XVII of the European Chemicals Regulation (REACH), with phase-outs occurring through 2032 and certain allowed uses. • In 2020, EU banned the use of PFOA and PFOS and added them along with other PFAS compounds to the Toxic Release Inventory list (TRI)
Canada	• Embarked on an Environmental Performance Agreement with four major manufacturers to phase out PFOA and related compounds from 2010 to 2015 (CEPA 2006). • By 2016, Canada prohibited the import, manufacture, use and sale of PFOS, PFOA, and other long-chain PFCAs (and salts and precursors), with limited exemptions (CEPA 2018), and subsequently • In 2017, the South Australia state government took initial steps to develop legislation banning environmentally harmful foams, such as Class B firefighting foams containing PFAS (SA EPA 2017).
Japan	• There are restrictions on the manufacture, import, export, and use of PFOS and its salts (OECD 2015).
South Australia	• In 2017, the South Australia state government took initial steps to develop legislation banning environmentally harmful foams, such as Class B firefighting foams containing PFAS (SA EPA 2017).

HUNGARY



Location of sampling sites along the Danube River

Research goal : Investigate the occurrence of PFAS in the water and sediment phase of the Hungarian section of the Danube river.

Analytical techniques : To achieve the goal of the study, we observed:

- The temporal and spatial exposure of selected PFAS from 12 sampling points of the Danube river using the **UHPLC-QTOF-MS**
- Ascertain the adsorbable organofluoride compounds (enrichment with GAC before combustion) using the **Combustion Ion Chromatography (C-IC)**
- Determined the Inorganic Fluoride content using the **Ion Chromatography (IC)**

Methodology

1. Lab work & Analysis

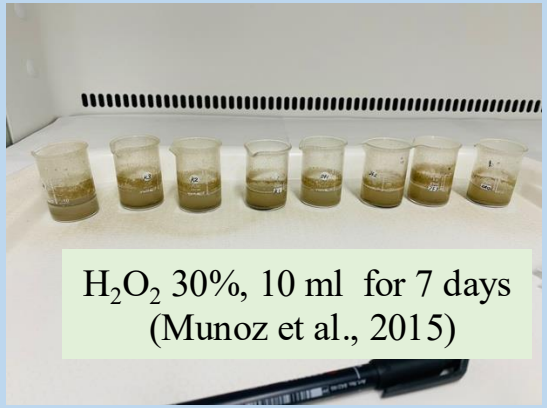
Water samples were drawn from the river's upper layer (10–50 cm) into 1 L HDPE bottles, while 1kg surface sediment samples were collected using a Van Veen grab sampler and stored in borosilicate glass bottles sealed and transported to the lab at 4 °C

2. Sample preparation

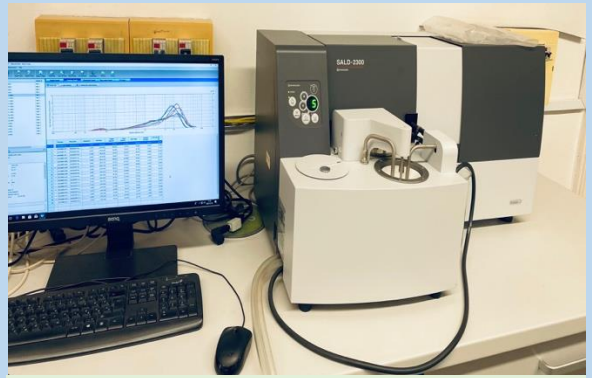
Drying

Homogenisation

3. Grain size analysis



H_2O_2 30%, 10 ml for 7 days
(Munoz et al., 2015)

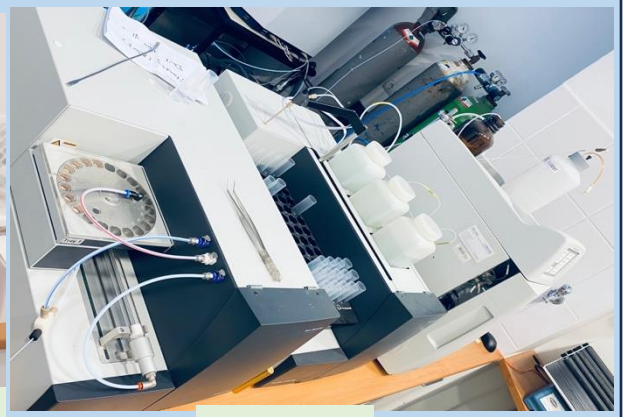


Laser diffraction PSA

4. AOF analysis of water and sediment

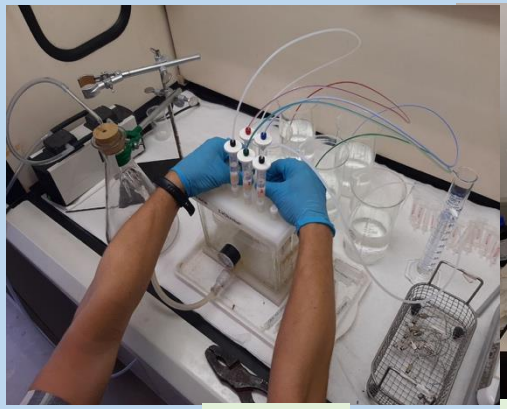
Filtration with Glass Microfiber filter

50 mL sediment extract
+ 5 mL of nitrate stock
+ 50 mg GAC



CIC

5. SPE and PFAS analysis

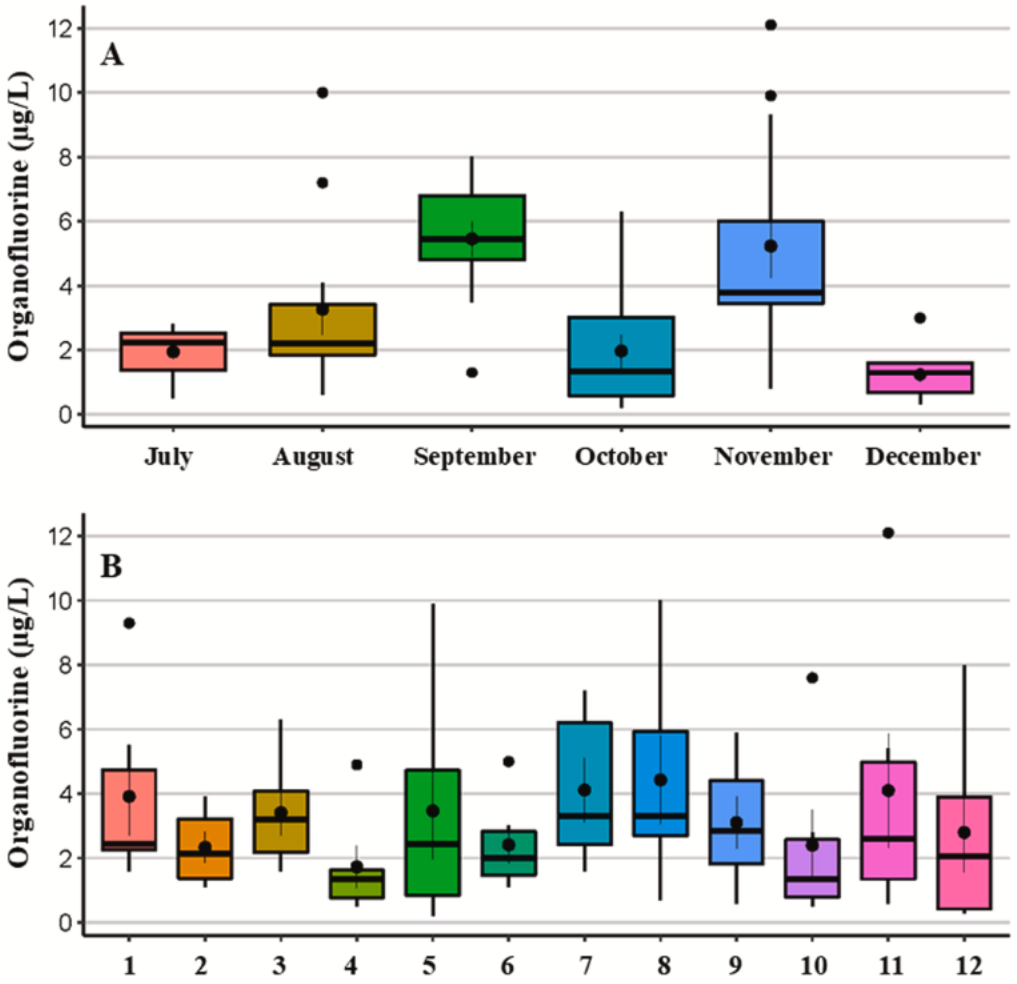


SPE

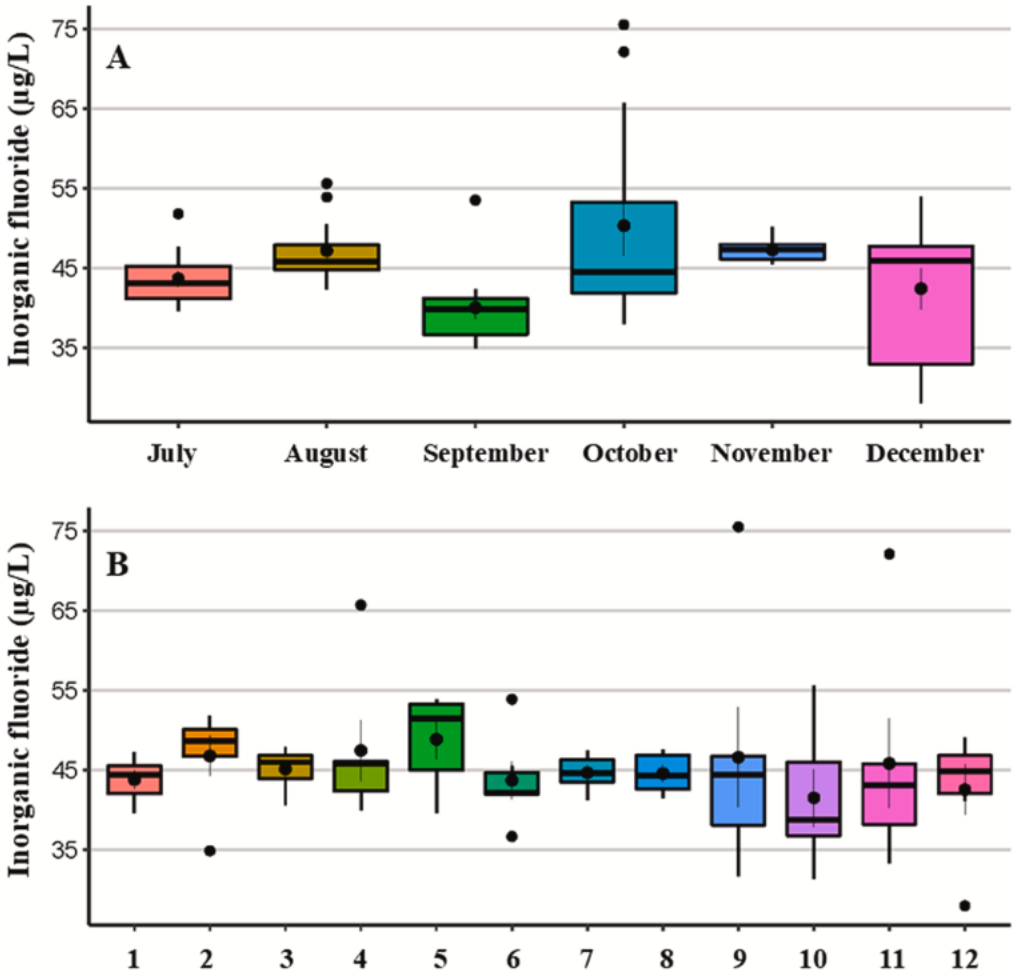
UHPLC-Q-TOF-MS

Results- I

(a) Average concentration of **AOF** measured in time and space within a 6 months timeframe



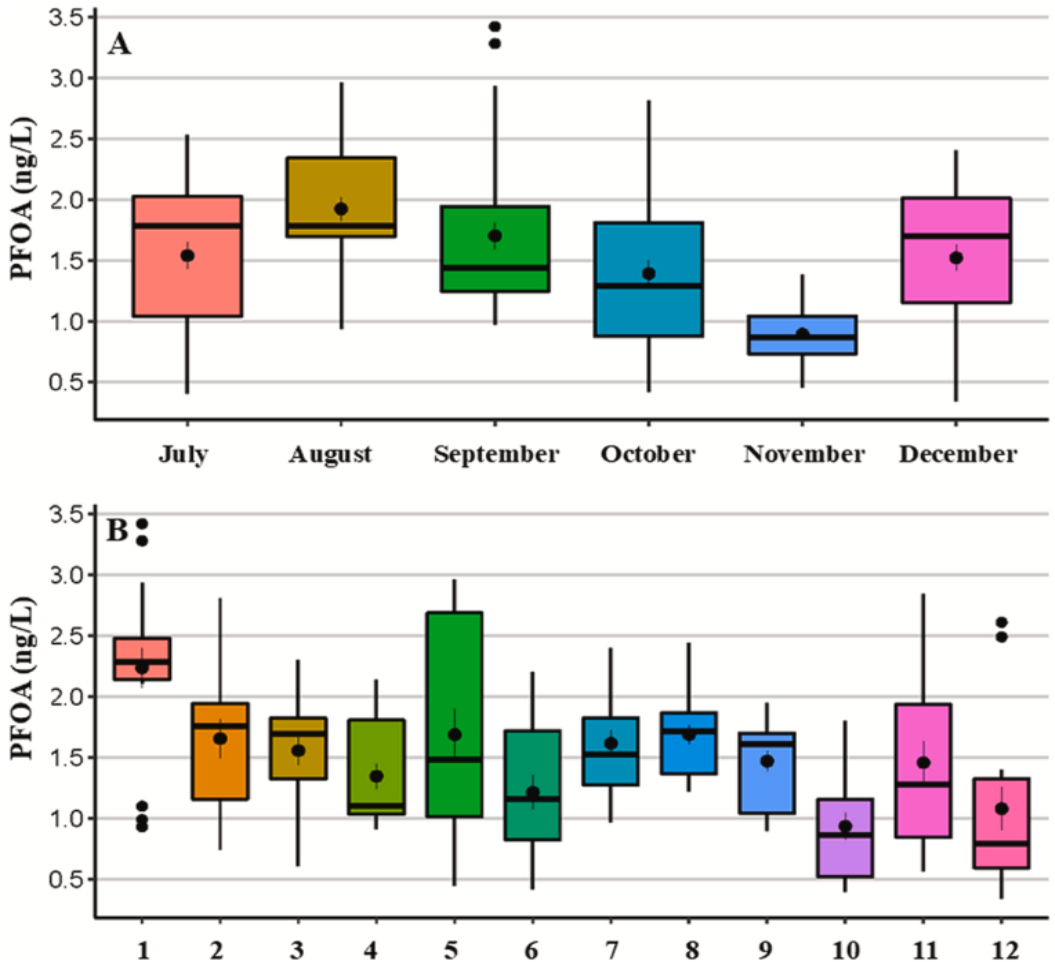
(b) Average concentration of **IF** compounds measured in time and space within a 6 months timeframe



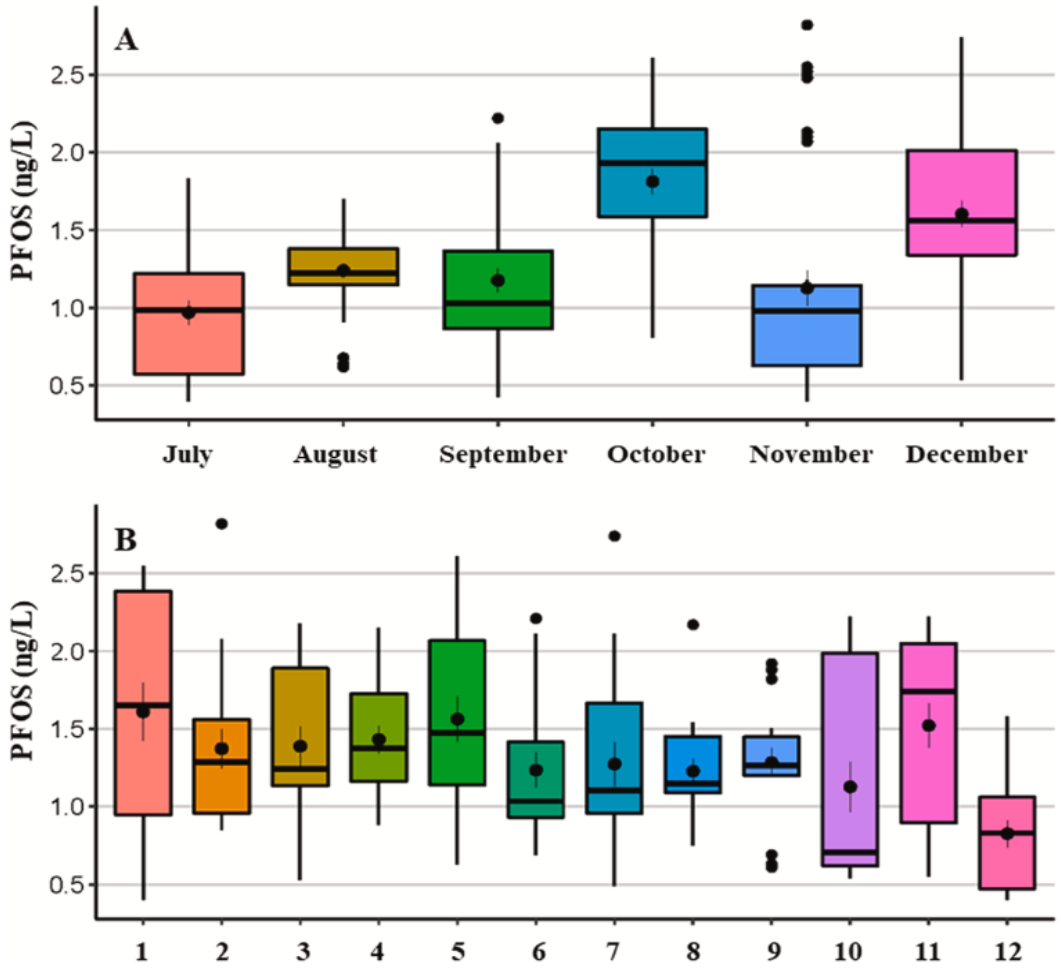
- It can be seen that both the temporal and spatial distribution of the AOF show high deviations while the inorganic fluoride concentration is relatively stable (40-60 µg/L) and more homogenous with less fluctuation. USEPA recommendation : 700 µg/L . Remediation : Fluoridization
- The AOF content is typically 3-10% of the IF.

Target compounds: PFOA and PFOS

PFOA



PFOS



- These were the most frequently used PFAS compounds in the industries and commercial products during the last decades before their use was prohibited in EU countries (2020).
- Considering the SD values, both compounds show very similar variations in both the temporal and spatial distribution in the concentration range of 1-2 ng/L

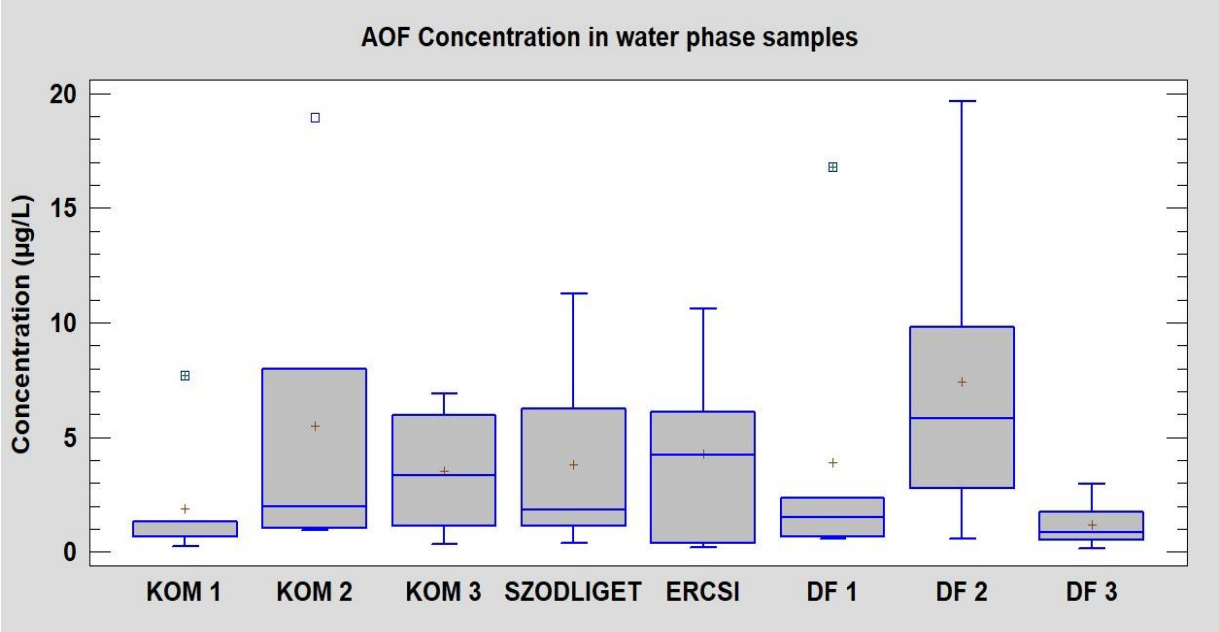
Results- II

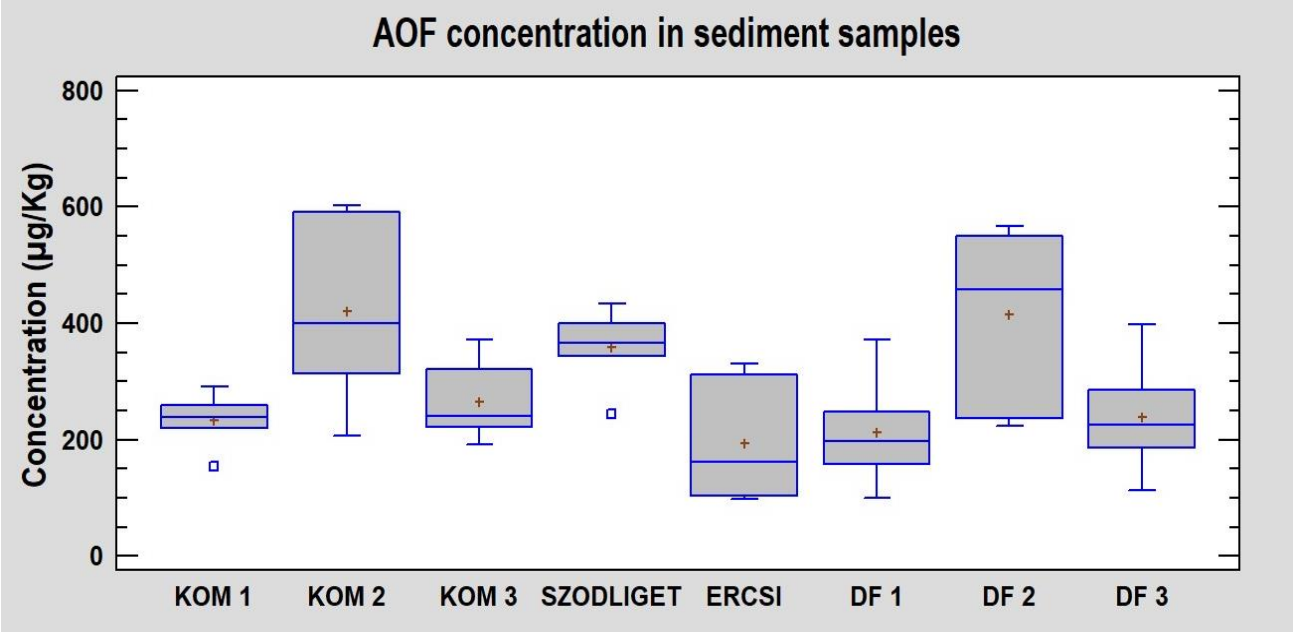
PFAS COMPOUNDS	Published average concentration values of PFOA and PFOS compounds in the water phase of the Danube River in the time period 2000 - 2023 (ng/L)							
	Sampling year							
	2000-2005	2005	2007	2012	2013	2019	2019	2023
	(Clara et al., 2009)	(McLachlan et al., 2007)	JDS 2 (Loos et al., 2010)	(Lindim et al., 2016)	JDS 3 (Loos et al., 2017)	JDS 4 (Liska et al., 2021)	(Beggs et al., 2023)	**Current study**
PFOA	18	16.4	14	8.51	8.1	2.1	2.77	1.54
PFOS	<4.3	-	~10	9.66	7.2	2.1	2.08	1.32

There is a clear decreasing tendency of the concentration of the banned PFAS compounds in the water phase of the Hungarian section of the Danube River

Results- IV

AOF Concentrations in Water (A) and Sediment (B) Samples Collected across three industrial regions of the Hungarian Section of the Danube River (July 2024 – January 2025).

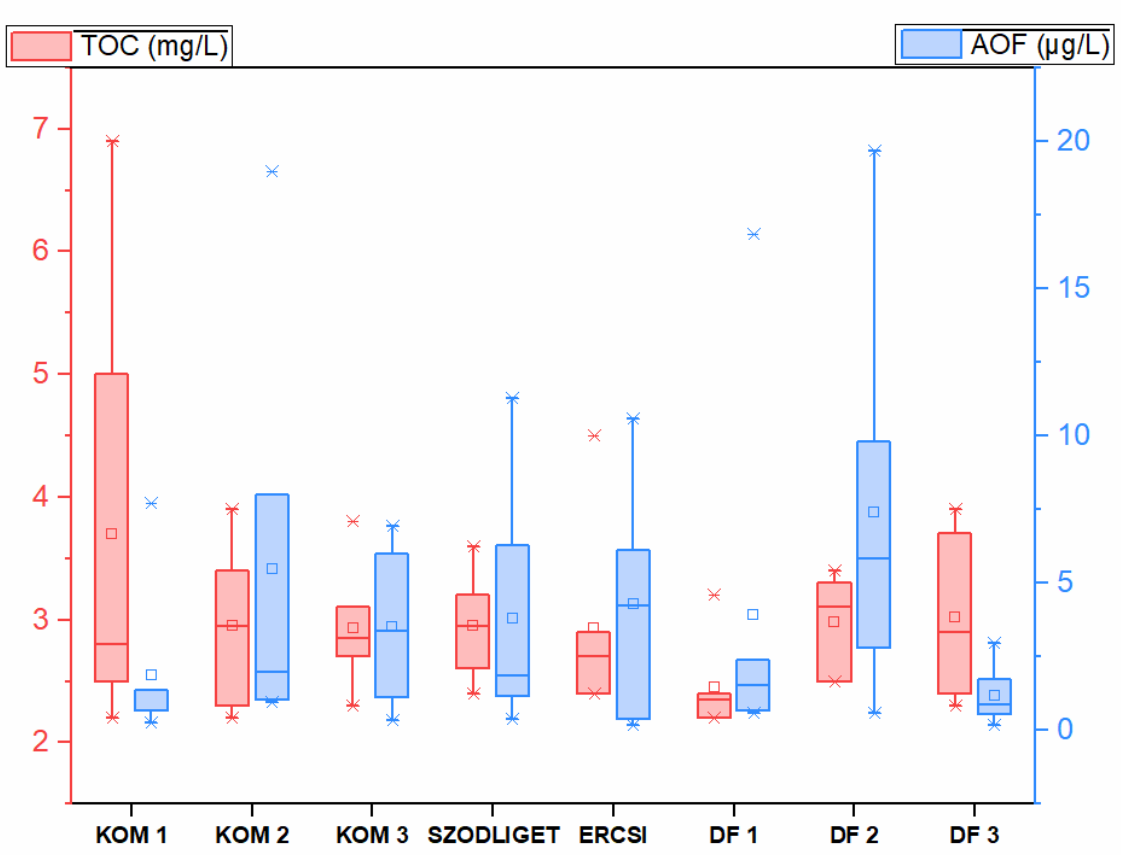


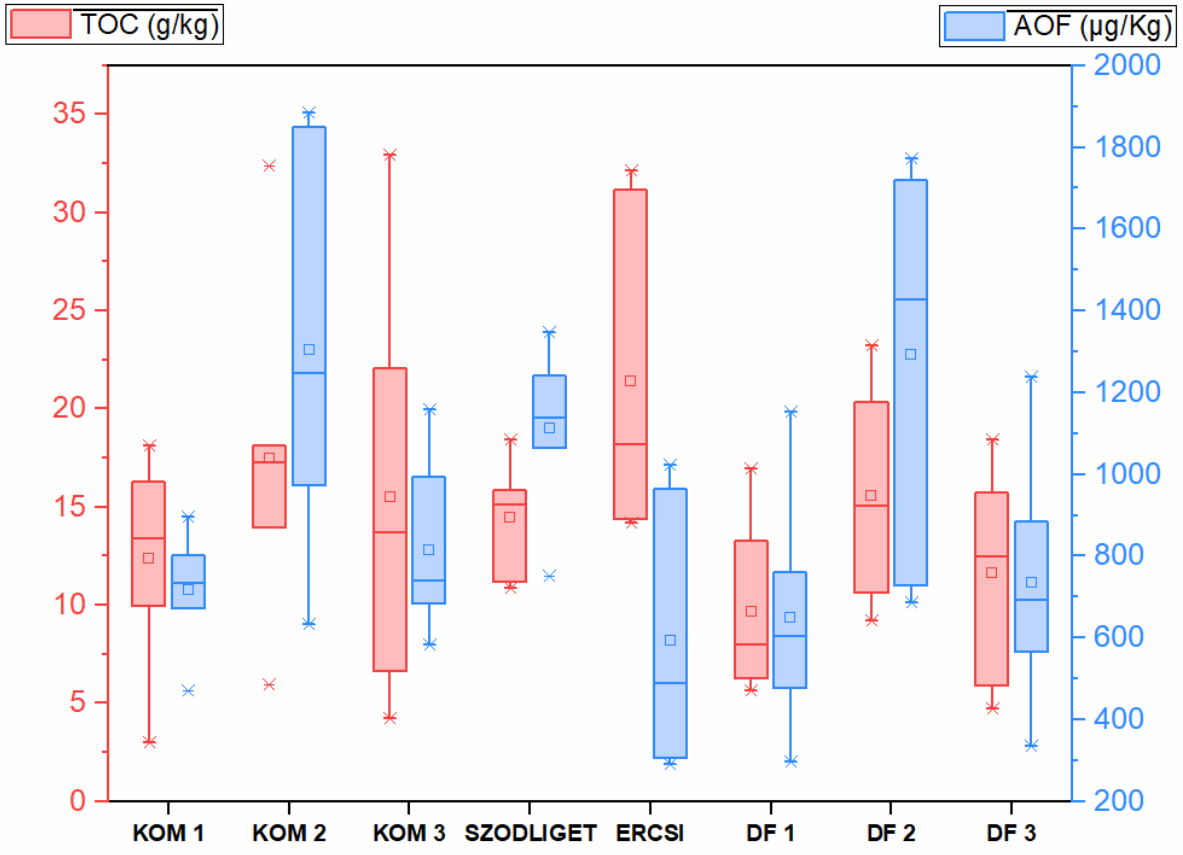


- The AOF concentration in sediment samples is ~1.86 orders of magnitude higher than the water concentration. Sediments often act as "sinks" for contaminants due to adsorption of organic molecules on biofilm covered particles.
- Higher concentrations were observed in samples close to the LIB and wrapping paper Factory (KOM 2 & DF2)
- Higher concentration in the sediment phase is very critical and dangerous for the filter feeding microorganisms living in the sediment area such as mussels and larvae zooplanktons

Results- II

Comparison of TOC and AOF average concentrations of sediments samples collected monthly from July 2024 to January 2025





- Low AOF and high TOC conc. were typical of surface sediments near oil refinery.
- Strong positive AOF-TOC correlation existed near LIB and wrapping paper factories.

Conclusion

Due to the high water yield of the Danube river in the Hungarian section (~2,350 m^3/s), both the spatial and temporal distribution of the **IF** content is practically homogeneous considering the standard deviation of our measurements

Concentration range : 40-60µg/L

The spatial and temporal distribution of the **AOF** in the Danube river shows higher deviations from the average values in areas of tributaries and industrial facilities

Concentration range : 1.5- 5.5µg/L

The AOF concentrations are approximately 10 times lower than the IF in spite of the high number of the organofluorines emitted into the aquatic environment.

The PFOA and PFOS show very similar variations in both the temporal and spatial distribution. As seen from the previous publications in the last decade down to this current research, there is a clear decreasing tendency of these compounds in the Danube River due to the regulations.

Concentration range : **PFOA** : 0.9-1.9ng/L,

PFOS: 0.8-1.8ng/L

The **AOF concentration in sediment samples** collected along the **industrial zone is 1.86 orders of magnitude** higher than the water phase concentration.

Acknowledgement

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Thank you for your attention! 🙂