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Investigation on Per- and Poly-Fluoroalkyl Substances Sources and Removal in a Municipal Wastewater Treatment Plant

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***Abstract* – PFAS (per- and poly-fluoroalkyl substances) is a complex family of manmade highly fluorinated aliphatic organic chemicals including thousands of chemical structures identified. A research was carried out at a municipal wastewater treatment plant (MWWTP) in Northern Italy to define the inflowing load of PFAS, the main load sources and the removal efficiency of the treatment processes. Then, the 100 industrial settlements served by the MWWTP were examined, and 8 of them were selected as potential PFAS sources. The calculated loads summed up to 98.5 % of the total load entering the plant. The removal in the WWTP was null and, in some cases, negative. As also reported in the literature some precursors can be transformed in PFAS during the biological process and some sludge accumulated compounds can be released.**

***Keywords* – Industrial contribution; PFAS load; PFAS removal; Wastewater Treatment Plant.**

Nomenclature		
PFAS	per- and poly-fluoroalkyl substances	–
AFFF	Aqueous Film Forming Foam	–
HPLC	High Performance Liquid chromatography	–
MS	Mass spectrometry	–
PFBA	Poly Fluoro Butanoic acid	
PFBS	Perfluorobutanesulfonic acid	
PFPeA	Perfluoropentanoic acid	
PFPeS	Perfluoropentanesulfonic acid	
PFHxA	Perfluorohexanoic acid	
PFHxS	Perfluorohexanesulfonic acid	
PFECHS	Perfluoroethylcyclohexane sulfonate	
PFHpA	Perfluoroheptanoic acid	
PFHpS	Perfluoroheptanesulfonic acid	
PFOA	Perfluorooctanoic acid	
PFOS	Perfluorooctanesulfonic acid	

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PFNA	Perfluorononanoic acid
PFNS	Perfluorononanesulfonic acid
PFDeA	Perfluorodecanoic acid
PFDS	Perfluorodecane sulfonic acid
PFTrDA	Perfluorotridecanoic acid
PFTrDS	Perfluorotridecanesulfonic acid.
PFTeDA	Perfluorotetradecanoic acid
PFUdA	Perfluoro-n-undecanoic acid
PFUdS	Perfluoroundecanesulfonic acid
PFDoA	Perfluorododecanoic acid
PFDoS	Perfluorododecanesulfonic acid·
4:2 – FTS	4:2 fluorotelomer sulfonic acid
6:2-FTS	6:2-fluorotelomersulfonic acid
8:2 – FTS	8:2 fluorotelomer sulfonic acid
LOQ	Limit of quantification

1. INTRODUCTION

PFAS is an acronym for per- and poly-fluoroalkyl substances, a large family of xenobiotics made of fluorinated aliphatic organic chemicals, including thousands of molecules known as "forever chemicals" due to their persistence in the environment. The first PFAS was polychlorotrifluoroethylene (PCTFE), a precursor of Polytetrafluoroethylene (PTFE), better known as Teflon, produced since 1934 and still largely used in many applications. In 1967 the US Food and Drug Administration (FDA) approved the first PFAS produced by DuPont for coating paper and cardboard.

PFASs are oil and water repellent, resistant to high temperatures and useful as lubricants, to reduce friction. So, they are used in extremely wide fields and can be found in most everyday objects [1].

Evidence of their accumulation in human body was first demonstrated in the 1970s when the presence of some PFAS in the blood of exposed workers was found [2]. Later studies showed the presence of PFASs in the blood of general human population not directly exposed for working reasons. The general population may be exposed to PFAS through diet, including drinking water and contaminated foods, which are key sources of human exposure, as well as from PFAS polluted environments [3]. The migration of PFAS from food packaging or cookware can contaminate foods and water can be contaminated not only by direct inputs, but also by the release of PFASs from textiles during washing.

The possible risks related to exposure to these substances have gained interest in recent years and is resulting in the inclusion of such compounds in the new Proposals for amending the European Water Framework Directive and the Directives on Urban Wastewater Treatment, on Groundwater and on the Water Quality Standards. PFAS and related compounds have been designated as target chemicals for regulation by the Stockholm Convention on Persistent Organic Pollutants, aimed at eliminating or restricting their production and use because they are mobile, persistent, and bio-accumulative.

The European Food Safety Authority (EFSA) has proposed to lower the current tolerable intake of specific PFAS (PFOS and PFOA) in foods, after examining new human epidemiological evidence [4].

Little information is available on the possible toxic effects of PFASs on wildlife, but they have been shown to affect cell−cell communication, the process of energy generation, membrane transport and peroxisome proliferation.

Due to the high number of molecules belonging to the PFAS group and to the high complexity and cost of target analyses, very few data are reported about the presence of such compounds in wastewater and about their main sources.

It is known that biological treatment is not effective in removing PFASs from wastewater, while adsorption on activated carbon is considered as a practicable technology, even if it is more effective on long chain molecules than on shorter ones (so, the performances vary according to the kind and age of PFAS contamination) and, especially, activated carbons used for PFAS removal get fastly exhausted and must be thermally regenerated at high temperatures (700–900 °C) [5], [6].

An analytical campaign was carried out to define the PFAS load entering a municipal wastewater treatment plant (MWWTP) in Northern Italy and to assess the efficiency of the treatment train in removing such molecules. A detailed analysis was carried out in the basin deserved by the MWWTP to detect the main sources of PFASs, considering the industrial activities, the quality of the supplied water and the contribution of Aqueous Film Forming Foams (AFFF) used to extinguish fires in industrial sites and also within the WWTP area.

2. MATERIALS AND METHODS

2.1. Source Investigation

Over 100 industrial settlements connected to the wastewater treatment plant were examined and classified as follows, according to their activities and the kind of wastewaters they produce:

a) wastewaters from continuous operations, having a relatively constant composition,
b) wastewaters from discontinuous processes, having a variable composition,
c) occasional discharges from road accidents or fires extinguished with Aqueous Film Forming Foams (AFFF) containing PFASs.

Eight settlements were selected and grab samples of their wastewaters were collected and analysed. The specific contribution to the load entering the WWTP was calculated multiplying the sum of the concentrations of the different PFAS in each industrial wastewater by the discharged flow. The obtained data were compared with the total load entering the WWTP, calculated at the same way (concentration × inlet flow).

In the end, a fire extinguishing event was simulated and the water contained in the tank for firefighting system was sampled and analysed.

2.2. Evaluation of WWTP Removal Efficiency

To evaluate the removal of PFAS in the wastewater treatment plant 24-h average samples were collected from the inlet and the outlet.

2.3. Sampling and Analyses

Samples were stored at $T \leq 6$ °C in polypropylene bottles and analyses were carried out within 14 days from sampling.

Target analysis of PFAS were performed by HPLC. The analysis was carried out according to the standard method UNI CEN/TS 15968:2010 (British Standards Institute Staff, 2010) by a certified (UNI CEI EN ISO/IEC 17025) external laboratory. (UltiMate 3000 Basic LC System, Orbitraptm) and high resolution mass spectrometry (Q Exactive Plus Hybrid Quadrupole Orbitrap Mass Spectrometer, Orbitraptm). 25 compounds were searched: PFBA, PFDeA, PFDoS, PFDS, PFPeA, PFBS, PFECHS, PFHxA, PFHpA, PFPeS, PFHpS, PFHxS, PFNS, PFOA, 4:2 – FTS, 6:2-FTS, 8:2 – FTS, PFNA, PFUdA, PFTrDA, PFTrDS, PFOS, PFUdS, PFDoA, PFTeDA. The LOQ was 0.2 ng/L.

3. RESULTS AND DISCUSSION

Five out of the eight selected settlements appeared as relevant for the PFAS load they contribute to the WWTP. The analyses gave the results reported in Table 1 as sum of the concentrations of the detected PFASs (PFOS, PFOA, PFBA, PFPeA, PFHxA, and PFBS). The discharge flows and the resulting loads are also reported in Table 1. The number of detected molecules is limited. The concentrations were higher than LOQ only for 6 out of the 25 analysed compounds: PFOS, PFOA, PFBA, PFPeA, PFHxA, and PFBS. The maximum number of carbon atoms in the detected compounds was 6.

TABLE 1. DISCHARGED FLOWS AND PFAS CONCENTRATION (SUM OF PFOS, PFOA, PFBA, PFPEA, PFHXA AND PFBS) AND LOADS IN THE SELECTED INDUSTRIAL EFFLUENTS

Industrial activity	Discharged flow, m^3/day	PFAS concentration, sum, µg/L	Load to WWTP, mg/day
Textile dyeing	829	2.10	61
Processing of slaughterhouse wastes	683	4.97	14
Production of chemicals for agriculture	661	5.23	13
Treatment and disposal of solid wastes	291	0.51	6
Treatment and disposal of liquid wastes	191	0.47	47
Production of detergents	99	0.24	5

The results of the analyses of samples collected from the tank for fire-fighting system are reported in Table 2.

TABLE 2. MEASURED CONCENTRATIONS OF PFAS IN THE TANK FOR FIREFIGHTING SYSTEM

Compound	Concentration, µg/L
PFBA	2.494
PFPeA	1.060
PFHxA	15.836
PFHpA	0.604
PFOA	0.336
PFNA	0.143
PFDeA	0.080
PFDoA	0.020

In this case also a low number of compounds (8) was present in concentrations over the LOQ, but besides the short chain ones found in industrial effluents, also some larger

molecules were detected. Firefighting products are known as important sources of PFASs but it is actually hard to quantify their role due to the diversity of available products, but, especially, to the importance of the frequency of use and of the amount of diluting water per event.

Fig. 1 reports the average concentration of PFASs in the influent and in the effluent of the WWTP and the percent removal efficiencies.

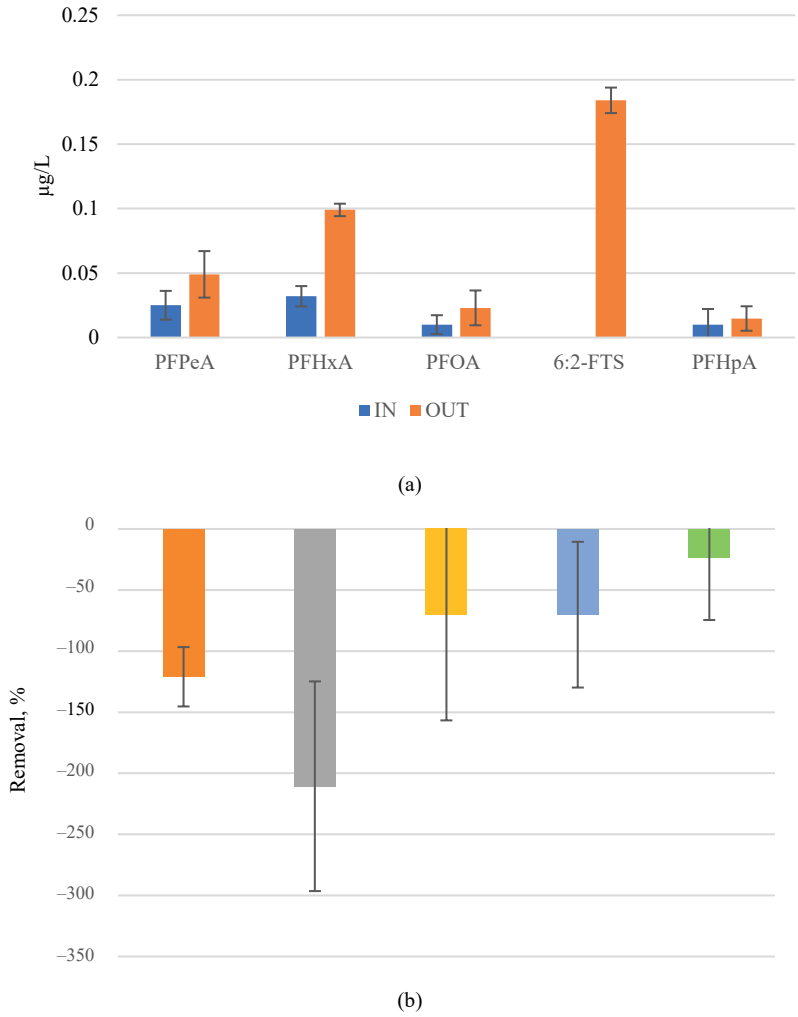

Fig. 1. Concentration of PFAS in the influent and in the effluent of the WWTP (a) and percent removal efficiency for the detected congeners (b). The error bars represent standard deviations.

It is interesting to observe that only 5 out of 25 compounds were found and that the standard deviations of analytical data were small. Except for PFOA, the detected compounds are all defined as short chain ones ($C < 8$), as expected due to their higher solubility and longer persistence in water [7]. However, the higher risk level is generally attributed to compounds having carbon chains longer than 6 atoms [2] as PFHxA, and PFHpA, regularly found during

monitoring. PFPeA, PFHxA and PFOA are among the 6 congeners for which, in Italy, a quality standard has been fixed for surface waters (D.Lgs.172/2015), according to the European Directives and to the recommendations by Stockholm Convention and ECHA. 6:2 FTS was found only once in the influent and twice in the effluent. The fate of 6:2 FTS in WWTP is still not clear. fluorotelomer precursor in biological SBR pilot plants and according to Coggan *et al*. [9], 6:2 FTS is an intermediate degradation product from C6 based precursors. On the contrary, some other articles report the degradation of FTS based molecules to some perfluoroalkyl carboxylic acids, such as PFOA [10]. Lab-scale experiments carried out by Wang *et al*. [11] showed that the major products derived from 6:2 FTS were PFPeA and PFHxA, whose summed concentrations after 90 days corresponded to only 2.6 % of the initial 6:2 FTS one. The same authors reported the biodegradation of 6:2 fluorotelomer sulfonic acid (6:2 FTSA) using the activated sludge from one WWTP, but not using the sludge from another one. At present, many different evidences are reported about both the formation and degradation and the ecotoxicological and toxicological effects of 6:2 FTS, which, at present, is largely used in industry as a less hazardous alternative to other compounds [10], [12].

In the effluent from the WWTP the detected PFAS were the same as in the influent, and a slight removal was observed only for PFHpA and 6:2 FTS, which could be considered as negligible taking into account the low starting concentrations, the standard deviations and, in the latter case, the presence of just one value in the influent. For the remaining three compounds the concentration increased by 77–194 %. Such increase might be attributed to the adsorption and subsequent release on primary and secondary sludge as well as on transformation processes from non-defined or unknown precursor and was reported also by other authors. Loganathan *et al*. [13] also attributed to the degradation of unidentified precursors as fluorotelomer alcohols or fluorotelomer sulfonates the increase of PFOA and PFOS concentrations in the effluent. Among others, Zhang *et al* [14] reported increases in PFAS concentrations up to 233 % after the activated sludge system and observed an increase for 8 compounds and a decrease for 10. Schultz *et al*. [15] attributed the increase of PFCAs, PFSAs, and PPOSA concentrations to the biodegradation of their precursors during activated sludge process.

Anyway, the maximum of the average concentrations in the effluent was 0.191 μg/L for 6:2 FTS, while for the other detected congeners the concentrations ranged between 0.015 and 0.098 μg/L, nearly complying with the indications for drinking water.

In spite of PFAS contamination, the overall performances of the WWTP have always been satisfying, and nitrification also, which is the most sensitive process in activated sludge, proceeded very well.

In the end, the load of PFAS from the selected industrial activities was compared to the total PFAS load entering the WWTP in order to define the % of their contribution. As shown in Fig. 2, the total load due to 5 industrial sites contributes about 70 % of the total PFAS load to the WWTP. Among the 5 sites, two seem nearly negligible, while the sum of the loads from the three others has a 69 % incidence on the total load. The about 30 % missing to the balance could be attributed to drinking water. Assuming that the concentration in the supplied water is 0.1 μg/L, corresponding to the lower limit set by the Italian rules, a “background load” can be estimated by multiplying such value by the discharged flow (39 000 m^3/day). The result shows a total background load of 3900 mg/day, corresponding to 31 % of the total load reaching the WWTP.

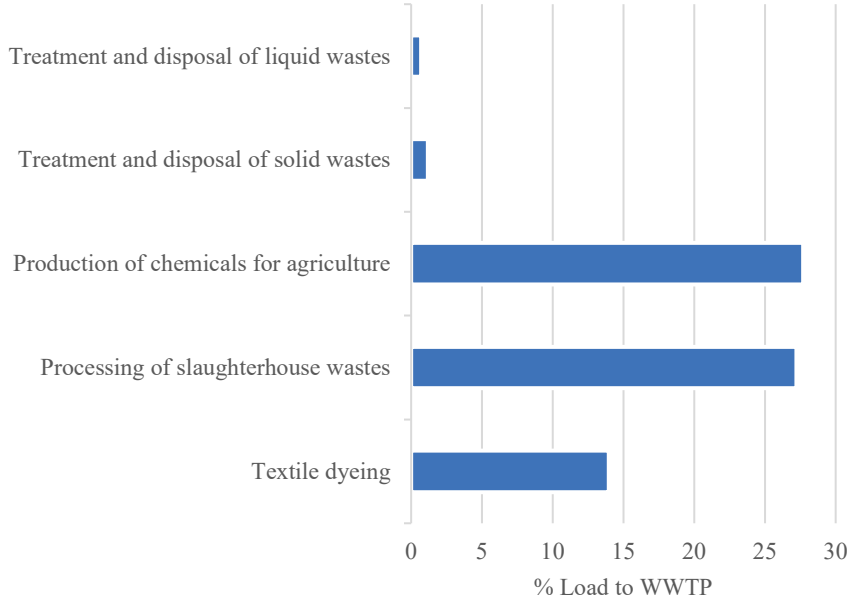

Fig. 2. Percent incidence of the PFAS loads form industry on the total PFAS load entering the WWTP.

Surprisingly, the contribution from the platforms for treatment and disposal of solid and liquid wastes seems quite low. In general, landfill leachates are rich in PFAS even if their concentration varies within a wide range according to the biodegradation step: in the early phases, when pH is acidic, the release of PFAS is much lower than during the methanogenic phase, when pH raises over 7 and especially larger perfluoroalkyl acids are released [16]. On the other hand, studies based on different age landfills have shown that the concentrations of many PFASs decreased significantly with increasing landfill age [17]–[19] and many other factors affect the kind and concentrations of PFASs in landfill leachate. The PFAS market is also rapidly changing, and this leads to the presence of different congeners also in wastes and in landfill leachates, even if some compounds are released from wastes for many years [18].

Very few data exist about slaughterhouse wastes, but a study by Fernandes *et al.* [20] reported that the use of contaminated recycled materials in farming lead to the accumulation of contaminants, including PFAS, in the tissues and eggs of animals, especially of laying chickens. Clarke & Smith [21] indicate the application of biosolids to soil a possible pathway for contaminating the food chain with several emerging pollutants, among which PFASs. Hlouskova *et al.* [22] found different degrees of PFAS contamination in food samples of animal origin from different European countries. PFOS and C8-C14 PFCAs were detected in approximately 50 % and 20 % of the analysed samples, respectively. The contamination concerned, in decreasing order of concentrations: seafood > pig/bovine liver >> freshwater/marine fish > hen egg > meat >> butter. The kind of materials processed from the analysed site is not defined but, based on data on animal sourced food and slaughter waste contamination, the presence of PFAS seems justified.

The use and discharge of PFASs from the production of chemicals for agriculture is also expected, as the formulation of many pesticides includes them as active ingredients as they make the pesticide more effective and stable, as reported by the American Environmental

Working Group (www.ewg.org). Sulfluramid, for instance, is a conventional insecticide whose active ingredient is N-ethyl perfluorooctane sulfonamide (EtFOSA) which is known to be biotransformed in PFOA and other PFASs [23]. Further, many fertilizers are based on biosolids of compost which are potentially contaminated by PFASs.

In the end, the textile industry is well-known as a large user of PFASs [24].

Although our study detected only a limited number of different PFASs, understanding the fate of non-detects is critical for understanding the overall pollution landscape. Non-detected PFAS may indicate that these substances are absent or that their concentrations are below detection limits, complicating environmental assessments. Research by Herzke *et al.* [25], reveal that the targeted PFAS analyses explained 1 % of the organofluorine contamination. This difference between detected molecules and the real amount of PFAS in the samples raises concerns about long-term ecological impacts, especially since the pathways and transformations of these substances are not thoroughly understood. In addition, undetectable PFASs may include emerging PFASs that have not yet been studied, revealing potential gaps in current monitoring and regulatory efforts. Therefore, expanding the range of targeted compounds to include these undetectable PFASs is critical for a comprehensive assessment of PFAS pollution.

4. CONCLUSION

The obtained results allowed to draw a tentative mass balance of the PFAS loads to the wastewater treatment plant. First of all, the contribution of domestic waters, in spite of their deriving from supply water complying with the most restrictive limits, not yet enforced, in Italy, is not negligible and accounts for about 30 % of the total load input.

Five industrial sites out of the hundred analysed sites were responsible for 71 % of the load and within them 3 contributed 69 % of the load. The most relevant inputs derive from textile dyeing, processing of slaughterhouse wastes and production of chemicals for agriculture.

Only 5 congeners (PFPeA, PFHxA, PFOA, 6:2 FTS and PFHpA) were found in the influent and in the effluent and the longest chain one was PFOA. However, except for 6:2 FTS, the detected compounds are listed among the most hazardous. The concentrations in the analysed samples varied within a quite narrow range.

In agreement with other research, the PFAS removal efficiency of the wastewater treatment plant, which is a conventional activated sludge, was very low for 6:2 FTS and PFHpA and negative for the other detected compounds, but the final concentrations in the effluent were very low.

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