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Levels of perfluorochemicals in water samples from Catalonia, Spain: is drinking water a significant contribution to human exposure?

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Abstract
Background, aim, and scope In recent years, due to a high persistence, biomagnification in food webs, presence in remote regions, and potential toxicity, perfluorochemicals (PFCs) have generated a considerable interest. The present study was aimed to determine the levels of perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA), and other PFCs in drinking water (tap and bottled) and river water samples from Tarragona Province (Catalonia, Spain).
Materials and methods Municipal drinking (tap) water samples were collected from the four most populated towns in the Tarragona Province, whereas samples of bottled waters were purchased from supermarkets. River water samples were collected from the Ebro (two samples), Cortiella, and Francolí Rivers. After pretreatment, PFC analyses were performed by HPLC-MS. Quantification was done using the internal standard method, with recoveries between 68% and 118%.
Results In tap water, PFOS and PFOA levels ranged between 0.39 and 0.87 ng/L (0.78 and 1.74 pmol/L) and between 0.32 and 6.28 ng/L (0.77 and 15.2 pmol/L), respectively. PFHpA, PFHxS, and PFNA were also other detected PFCs. PFC levels were notably lower in bottled water, where PFOS could not be detected in any sample. Moreover, PFHpA, PFHxS, PFOA, PFNA, PFOS, PFOSA, and PFDA could be detected in the river water samples. PFOS and PFOA concentrations were between <0.24 and 5.88 ng/L (<0.48 and 11.8 pmol/L) and between <0.22 and 24.9 ng/L (<0.53 and 60.1 pmol/L), respectively.
Discussion Assuming a human water consumption of 2 L per day, the daily intake of PFOS and PFOA by the population of the area under evaluation was calculated (0.78–1.74 and 12.6 ng, respectively). It was found that drinking water might be a source of exposure to PFCs as important as the dietary intake of these pollutants.
Conclusions The contribution of drinking water (tap and bottled) to the human daily intake of various PFCs has been compared for the first time with data from dietary intake of these PFCs. It was noted that in certain cases, drinking water can be a source of exposure to PFCs as important as the dietary intake of these pollutants although the current concentrations were similar or lower than those reported in the literature for surface water samples from a number of regions and countries.
Recommendations and perspectives Further studies should be carried out in order to increase the knowledge of the role of drinking water in human exposure to PFCs.

Keywords Bottled water · Municipal drinking water · Perfluorinated chemicals · River water · Tarragona (Catalonia · Spain)

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1 Background, aim, and scope

Perfluorochemicals (PFCs) are a group of chemicals that include the perfluoralkyl acids and their salts: perfluoroalkyl sulfonates, perfluoralkyl carboxylates, and including poly-fluorinated telomer alcohols and their derivatives. These

compounds have been widely used for more than 50 years in commercial and industrial applications such as surfactants, lubricants, paper and textile coatings, polishes, food packaging, and fire-retarding foams among others (OECD 2002).

The reported global distribution and environmental persistence of PFCs (Kannan et al. 2005, Sinclair et al. 2006), as well as their presence in humans and wildlife (Apelberg et al. 2007; Calafat et al. 2007; Emmett et al. 2006; Ericson et al. 2007; Kärrman et al. 2007), together with the adverse health effects detected in laboratory animals (Fuentes et al. 2007a, b) has generated a considerable interest regarding these compounds on an international scale. However, data regarding potential toxicity in humans are very scarce. Although the routes of human exposure to PFCs are not well characterized yet, it has been hypothesized that dietary intake could be one possible, and perhaps as for POPs such as PCDD/PCDFs, PCBs, PBDEs, etc., the most important way of exposure. Recently, we estimated the dietary intake of PFCs for various age–gender groups of a nonexposed general population from Tarragona Province (Catalonia, Spain; Ericson et al. 2008). The intake of perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA), and perfluoroheptanoic acid (PFHpA), the only PFCs that could be detected, was lower than those reported for Canada (Tittlemier et al. 2007) and considerably lower than those previously reported for the UK (UK FSA 2006), the only two countries where data from studies concerning this same issue have been reported. Although our results as those from Canada suggest a correlation between dietary intake and human blood levels of PFOS, it cannot be extended to the levels of other PFCs.

In order to increase the knowledge on the human exposure sources to these pollutants, in the present study, we estimated the intake of 14 PFCs through drinking water (tap and bottled) by the population of Tarragona Province. River water samples in the same area were also analyzed for the occurrence of PFCs.

2 Materials and methods

2.1 Sample collection

In February 2007, water samples of different origin were collected in several locations of Tarragona Province. Three kinds of water were sampled: municipal drinking (tap) water, river water, and bottled water. Municipal drinking water was obtained in public fountains of the most populated towns in Tarragona Province: Reus, Tarragona, Tortosa, and Valls. River water samples were collected at two points of the Ebro River (Garcia and Mora), as well as in the Francolí and Cortiella Rivers. Finally, bottled water of four commercial companies (Font Vella, Cardó, Veri, and Caprabo), whose water spring has different origins, were purchased from a supermarket. All samples were collected in 1.5 L polyethylene (PET) bottles and precleaned with ultra pure water. Before sampling, PET bottles were repeatedly rinsed with each sample of water. Water samples were stored at 10°C until analysis.

2.2 Extraction

Water samples were filtered with glass microfiber filters, Whatman® (Schleicher and Schuell, Maidstone, UK) using only the dissolved phase during analysis. Samples were concentrated using solid phase extraction (Taniyasu et al. 2005). Briefly, 500 mL of water were used for extraction after adjusting the pH to 4 using an HCl solution. Extraction standards, $^{13}C_4$-PFOS and $^{13}C_4$-PFOA (Wellington Laboratories, Guelph, Ontario, Canada), and 10 mL of methanol (MeOH) were added. After 10 min, water samples were loaded onto Waters Oasis® WAX single use cartridges (6 cm^3/150 mg) previously conditioned with 4 mL MeOH and 4 mL water. Vacuum was used to speed up the concentrations of water samples. After drying, SPE cartridges were eluted with 4 mL acetate buffer solution (discarded) and 2 mL 2% NH_4 in MeOH (target fraction). This fraction was filtered (2 µm nylon filter) and evaporated under nitrogen. The final volume was set to 500 µl including $^{13}C_5$-labeled PFNA added as performance standard and 300 µL of 2 mM sodium acetate (Merck, Darmstadt, Germany) in water.

2.3 Analytical procedure

The levels of the following PFCs were determined: PFOS, PFOA, PFHpA, perfluorobutane sulfonate (PFBuS), perflurohexane sulfonate (PFHxS), 1H,1H,2H,2H-perfluorooctanesulfonic acid (THPFOS), perfluorodecanesulfonate (PFDS), perfluorohexanioc acid (PFHxA), perfluorononanioc acid (PFNA), perfluorodecanoic acid (PFDA), perfluoroundecanioc acid (PFUnDA), perfluorododecanoic acid (PFDoDA), perfluorotetradecanoic acid (PFTDA), and perfluorooctanesulfonamide (PFOSA). Analysis of PFCs was performed using an Agilent 1100 HPLC system (Agilent, Waldbronn, Germany) coupled to an HP 1100 mass spectrometric detector (MSD, Waldbronn, Germany) with an atmospheric electrospray interface operating in negative ion mode. Separation was performed on a Discovery HS C18 (50 mm length, 2.1 mm i.d., 3 um particles, 120 Å pore size) column with a guard column of the same material (20 mm length, 2.1 mm i.d., 3 µm particles, 120 Å pore size) from Supelco (Bellefonte, PA, USA). Both columns were kept at 40°C. An extra guard column (Hypercarb graphitic carbon, 4 mm length, 10 mm i.d., 5 um particle size, Termo Hypersil-Keystone, Bellefonte, PA, USA.) was

inserted between the pump and the injector to remove any fluorochemicals originating from the HPLC system. Injection volume was 10 μL, and the flow rate was set to 300 μL/min. The mobile phases consisted of 2 mM ammonium acetate (Fluka, Steinheim, Germany) in methanol (Labscan, Dublin, Ireland) and 2 mM ammonium acetate in water. The mobile phase gradient started at 35% methanol followed by a 20-min ramp to 90%, a 2-min hold followed by a 3-min washing sequence with 100% methanol, and then reverting to initial conditions allowing 7 min stabilization time. The MS settings were as follows: nitrogen nebulizer gas temperature 350°C, nebulizer gas pressure 20 psi, nitrogen drying gas flow 13 mL/min, and capillary voltage 3,500 V.

2.4 Quantification and quality assurance

Quantification of PFCs was performed using the internal standard method. Labeled $^{13}C_4$-PFOS was used as an extraction standard for the sulfonates, whereas $^{13}C_4$-PFOA was used for the carboxylates. The performance standard $^{13}C_5$-PFNA was used to monitor the recovery of the extraction standards. Recoveries of 71–118% and 68–110% were achieved for $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, respectively. The limit of detection (LOD) was determined as three times the signal to noise ratio. One blank sample (HPLC grade water), one spiked water sample, and one reference water sample were extracted with every batch of samples showing good repeatability for the method. External quality assurance was performed by successful participation (z scores <2) in the first and second interlaboratory study on perfluorinated chemicals (van Leeuwen et al. 2007).

3 Results

The concentrations of 14 PFCs in the 12 water samples here analyzed are summarized in Table 1. With respect to the four samples corresponding to municipal drinking (tap) water, the highest PFC levels were found in the Valls sample (No. 4), in which PFHpA (3.02 ng/L; 8.30 pmol/L), PFOS (0.44 ng/L; 0.88 pmol/L), and PFOA (6.28 ng/L; 15.2 pmol/L) were detected. In the sample of tap water from Reus (No. 1), PFHxS (0.28 ng/L; 0.70 pmol/L), PFOA (0.98 ng/L; 2.37 pmol/L), PFNA (0.52 ng/L; 1.12 pmol/L), and PFOS (0.73 ng/L; 1.46 pmol/L) were detected, while in that of Tarragona (No. 2), PFHpA (0.64 ng/L; 1.76 pmol/L), PFHxS (0.28 ng/L; 0.70 pmol/L), PFOA (1.07 ng/L; 2.58 pmol/L), PFNA (0.22 ng/L; 0.47 pmol/L), and PFOS (0.87 ng/L; 1.74 pmol/L) were found. The lowest PFC contamination in tap water corresponded to the sample collected in Tortosa (No. 3), in which only PFOA (0.32 ng/L; 0.77 pmol/L) and PFOS (0.39 ng/L; 0.78 pmol/L) were detected.

In relation to the four river water samples analyzed in the current survey, the lowest PFC pollution corresponded to the sample collected in the Cortiella River (No. 8) where the 14 analyzed PFCs were under the respective detection limits. In contrast, the most polluted sample was that collected from the Francolí River (No. 7), where PFHpA, PFHxS, PFOA, PFNA, PFOS, PFOSA, and PFDA were detected. In the Ebro River samples (Nos. 5 and 6), PFHpA, PFHxS, PFOA, PFNA, and PFOS and PFHxS, PFOA, PFNA, and PFOS, respectively, were detected at similar concentrations in both samples.

Table 1 Concentrations of PFCs (ng/L) in water samples of different sources: municipal drinking water, river water, and bottled water

Sample	Tap water				River water				Bottled water			
	1	2	3	4	5	6	7	8	9	10	11	12
PFBuS	<0.27	<0.27	<0.27	<0.27	<0.27	<0.27	<0.27	<0.27	<0.27	<0.27	<0.27	<0.27
PFHxA	<0.87	<0.87	<0.87	<0.87	<0.87	<0.87	<0.87	<0.87	<0.87	<0.87	<0.87	<0.102
PFHpA	<0.61	0.64	<0.61	3.02	0.72	<0.61	3.38	<0.61	0.40[a]	<0.61	<0.61	<0.61
PFHxS	0.28	0.28	<0.18	<0.18	0.40	0.43	0.78	<0.18	<0.18	<0.18	<0.18	<0.18
THPFOS	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
PFOA	0.98	1.07	0.32	6.28	1.9	1.45	24.9	<0.22	0.34	<0.16	<0.39	0.67
PFNA	0.52	0.22 [a]	<0.42	<0.42	0.44	0.36 [a]	0.64	<0.42	0.20 [a]	0.13 [a]	<0.42	<0.42
PFOS	0.73	0.87	0.39	0.44	1.59	2.47	5.88	<0.24	<0.24	<0.24	<0.24	<0.24
PFOSA	<0.19	<0.19	<0.19	<0.19	<0.19	<0.19	0.20	<0.19	<0.19	<0.19	<0.19	<0.19
PFDA	<0.82	<0.82	<0.82	<0.82	<0.82	<0.82	0.49	<0.82	<0.82	<0.82	<0.82	0.63
PFUnDA	<0.43	<0.43	<0.43	<0.43	<0.43	<0.43	<0.43	<0.43	<0.43	<0.43	<0.43	<0.43
PFDS	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
PFDoDA	<0.34	<0.34	<0.34	<0.34	<0.34	<0.34	<0.34	<0.34	<0.34	<0.34	<0.34	<0.34
PFTDA	<0.90	<0.90	<0.90	<0.90	<0.90	<0.90	<0.90	<0.90	<0.90	<0.90	<0.90	<0.90

[a] Close to the LOQ, LOD S/N ratio

In the present study, the samples of bottled water were the least contaminated by PFCs. In the Veri sample (No. 11), all the 14 analyzed PFCs were below the respective LODs, whereas in those of Cardó (No. 10) and Caprabo (No. 12), only PFNA (0.13 ng/L; 0.28 pmol/L) and PFOA (0.67 ng/L; 1.62 pmol/L), respectively, could be detected. In the Font Vella sample (No. 9), PFHpA (0.40 ng/L; 1.10 pmol/L), PFOA (0.34 ng/L; 0.82 pmol/L), and PFNA (0.20 ng/L; 0.43 pmol/L) were detected. PFOS could not be found in any of the samples. It is noteworthy that generally, the few detected PFCs in the bottled water showed lower concentrations than those found in samples of municipal drinking water.

4 Discussion

The daily intake of PFOS, one of the most important perfluorinated surfactants with more than 50 years of production, through tap water (assuming an intake of 2 L tap water/day) by individuals living in the sampling towns would be the following: in Reus, 1.46 ng; in Tarragona, 1.74 ng, and 0.78 and 0.88 ng in Tortosa and Valls, respectively. In our recent study on the dietary exposure to PFCs (Ericson et al. 2008), the lower-bound and upper-bound intake of PFOS was calculated by assuming that if in a food item the level of this compound was under the LOD, this concentration would be equal to zero (ND=0) or one half of that limit (ND=1/2 LOD), respectively. For an adult of 70-kg body weight living in Tarragona Province, the dietary intake of PFOS was 62.5 or 74.2 ng (assuming ND=0 or ND=1/2 LOD, respectively; Ericson et al. 2008). For an adult of 70 kg of body weight, the daily intake of PFOS through tap water consumption exceed the average intake of PFOS through the daily consumption of pulses, cereals, fruits, and oils and fats, assuming ND=0. Since the point of view of risk assessment, it must be noted that the international regulatory organisms (WHO, EU, US EPA, etc.) have not established safety limits yet for PFOS. However, for guidance purposes, the 3M Company, the main manufacturer of PFOS developed a lifetime Drinking Water Health Advisory (DWHA), which was estimated to be 1.0 μg PFOS/L (assuming a consumption of 2 L per day of contaminated water; 3M 2001), a value that according to experimental studies in primates would not represent imminent health risks (Hansen et al. 2002). For this compound, the levels found in the current study are notably lower than the DWHA.

Concerns have been also raised over the potential health effects of PFOA. In the present survey, the daily intake through the municipal water of Valls would be 12.6 ng, a quantity similar than that derived from the average daily consumption of milk, the only food item where PFOA could be detected in our recent food study (Ericson et al. 2008). However, it should be taken into account that food preparation was carried out by washing foodstuffs with tap water. Therefore, water might also be an additional source of the levels of PFOS and PFOA found in food.

In the scientific literature, there are very few reports concerning PFC levels in drinking water. In the USA, high tap water values were found in samples of Little Hocking Water Association (OH). PFOA concentrations ranged from 1,600 to 6,900 ng/L in the distribution system (RIWA 2006). In Japan, Saito et al. (2004) analyzed the levels of PFOA and PFOS in tap water samples in the Osaka area and the Tohoku district. For PFOA, the geometric mean was between 0.7 and 40.0 ng/L, while that of PFOS was between <LOD and 12.0 ng/L. The authors concluded that although the effects of exposure to PFOA at 40 ng/L remain unknown, it was previously reported that PFOA might increase the risk of prostate cancer (Gilliland and Mandel 1993). In Europe, Skutlarek et al. (2006) determined the levels of 12 PFCs in drinking water samples collected in public buildings of the Rhine–Ruhr area (Germany). The maximum concentration of all samples taken in the Rhine–Ruhr area was found at 598 ng/L (sum of seven detected PFCs), with the major component being PFOA (519 ng/L). PFOA and PFOS levels ranged between ND and 519 ng/L and ND and 22 ng/L, respectively. These concentrations are remarkably higher than those found in the present study and also higher than those reported by the same authors for PFCs in drinking water samples outside the Ruhr area.

Some results of recent surveys in which the levels of a number of PFCs, mainly those of PFOS and PFOA, have been determined in surface freshwater samples are summarized in Table 2. It can be seen that the levels of PFOS and PFOA detected in the current study are, in general terms, among the lowest reported concentrations. These levels are of the same order of magnitude that those found in Japan by Saito et al. (2003, 2004) and Yoshida et al. (2007). To date, Japan is the country for which most data have been reported.

5 Conclusions

In the current survey, the contribution of drinking water (tap and bottled) to the human daily intake of various PFCs has been compared for first time with data from dietary intake of these PFCs. We have noted that in certain cases, drinking water can be a source of exposure to PFCs as important as the dietary intake of these pollutants. Moreover, river water samples of the same area were also analyzed. The concentrations found were similar or lower than most levels found in the literature.

Table 2 Levels of perfluorinated compounds (mainly PFOS and PFOA) in surface freshwater samples—a summary of recent surveys

Country/location	Analyzed compounds	Concentrations (ng/L)	Remarks	Reference
USA/Tennessee River	PFOS and PFOA	PFOS: 18.4–144 PFOA: <25–598	40 river water samples	Hansen et al. (2002)
Japan/various geographical locations	PFOS	median: 1.68 range: 0.30–1.57	The highest PFOS levels were found in Jinzu (135.0 ng/L) and Tama (157 ng/L) Rivers	Saito et al. (2003)
Canada-USA/ Eire and Ontario Lakes	PFOS, PFOA and 6 other PFCs	PFOS: 21–70 PFOA: 27–50	In all samples, N-EtFOSAA and FOSA levels were also above the limit of quantification (LOQ)	Boulanger et al. (2004)
Japan/Osaka and Tohoku areas	PFOS and PFOA	Geometric means (tap water) PFOS: <LOD-12.0 PFOA: 0.7–40	In Osaka city, PFOA in drinking water (40 ng/L) was significantly higher than in other areas	Saito et al. (2004)
USA/MI and IN, various rivers	PFOS, PFOSA, PFOA and PFHS	PFOS: 2.6–3.5 PFOA: 4.4–14.7	PFOSA and PFHS levels: under the LOQ	Kannan et al. (2005)
USA/NY State	PFOS, PFOA, PFHS and PFOSA	PFOS: 1.6–756 PFOA: 15–49 PFHS: 0.9–7.4	51 surface water (lakes, rivers, canals) samples from 9 major water bodies	Sinclair et al. (2006)
Japan and other 7 countries over the world	PFOS and PFOA	PFOS: 0.5 (Vietnam and Thailand)–8.3 (Japan) PFOA: 0.6 (Malaysia)–9,579 (Ai River, Japan)	River water samples	Tanaka et al. (2006)
Japan	PFOS, PFOA and 4 other PFCs	PFOS: <5.2–10 PFOA: 7.9–110	River water samples	Senthilkumar et al. (2007)
China/Pearl and Yangtze Rivers	14 different PFCs	PFOS: <0.01–99 PFOA: 0.85–260	Lower (or even lower) levels than the LOQ were found for 12 PFCs	So et al. (2007)
Sri Lanka	13 different PFCs	PFOS: 0.66–47 PFOA: 0.83–12.4	Fresh water samples collected from various places	Guruge et al. (2007)
Japan/Osaka, Yodo River system	PFOS, PFOA, PFHxS, PFBS and PFOSA	PFOS: 1.6–104 PFOA: 3.8–311	River water samples. Positive correlation between PFOS and PFOA levels	Takagi et al. (2007)
Japan/Hyogo Pref., various rivers	PFOS and PFOA	Geometric means PFOS: 2.1; PFOA: 17	Highest concentrations: PFOS: 650; PFOA: 410	Yoshida et al. (2007)
USA/NC, Cape Fear drainage basin	10 different PFCs	Maximum values for PFOS and PFOA: 13 and 287, respectively	22% and 32% of samples had PFOS and PFOA levels greater than 43 and 40 ng/L, respectively (conservative safe water levels for protection of avian life)	Nakayama et al. (2007)
Germany/Ruhr river area	12 different PFCs	PFOS: <2–193 PFOA: <2–3640	Highest levels in the Moehne River at HeidbergLower levels in the Rhine River	Skutlarek et al. (2006)
Germany/Roter Main River	PFOS and PFOA	PFOS: 2.2–2.6 PFOA: 3.2–3.4	River water samples upstream a wastewater treatment plant	Weremiuk et al. (2006)
Italy/"Affected" rivers around Lake Maggiore	PFOS, PFOA and other 5 PFCs	PFOS: <0.1–38.5 PFOA: 0.6–15.6	Lower levels in mountain rivers	Loos et al. (2007)
Spain/Tarragona, Catalonia	14 different PFCs	PFOS: <0.24–5.88 PFOA: <0.22–24.9	River water samples	This study

6 Recommendations and perspectives

Because the number of analyzed samples has been rather limited, further studies should be carried out in order to increase the knowledge of the role of drinking water in human exposure to PFCs. Moreover, additional information about processes of water transfer is needed because preparation of food using tap water might even increase the daily intake from water.

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