PERFLUOROOCTANE SULFONATE (PFOS) AND OTHER PERFLUORINATED COMPOUNDS IN WATER, SEDIMENT, PLANKTON AND FISH TISSUES COLLECTED FROM MAIN RIVERS AND LAKE IN KOREA

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Introduction

The unique characteristics regarding to the resistance to hydrolysis, photolysis, metabolism and bio-degradation processes in environment as well as thermal stability combined with the widespread application of perfluorinated compounds (PFCs) make them not only ubiquitously scattered in many different types of both abiotic and biotic matrices but also harmed the containing ecosystems and caused exposure in humans [1, 2]. In 2009, PFOS which is a specific PFC and its salts were included into Annex B of The Stockholm Convention on persistent organic pollutants.

The concentration of PFCs in some particular polluted areas in Korea has seemed higher than among the Asian countries and the other regions around the world [3, 4]. The previous studies from Korea have also reported the accumulation of PFCs in human blood [5], birds [1, 6], minke whales and common dolphins [7], Asian periwinkles and rockfish [3], coastal and ocean waters [8, 9]. However, available studies on PFCs status in Korea freshwater ecosystems such as lake or river are limited. Here, we carried out a study in 2010 and 2012 to determine the current status and extent of PFOS and other PFCs concentration in both non-living components and living components belonged to 6 main rivers and lake in Korea.

Materials and methods

Occurrences and concentrations of 10 perfluorinated compounds involving PFOS, perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnA), perfluorododecanoic acid (PFDoA), perfluorohexane sulfonate (PFHxS), perfluorodecane sulfonate (PFDS) were determined in water, sediment, plankton, blood and liver tissues of crucian carp (*Carassius auratus*) and mandarin fish (*Siniperca scherzeri*) collected from 17 sampling sites in total located in 6 main rivers and lake of Korea including Bukhan, Namhan, Nakdong, Nam, Sangsa and Yeongsan (Fig. 1).

Sampling: 1L clean polypropylene (PP) bottle pre-rinsed with Milli-Q water, methanol and water from specific sampling site was carefully sunk to collect surface water. Surface layer of sediment samples were collected using a clean and methanol rinsed PP spoon and stored in methanol pre-cleaned 50 ml PP tubes. Phytoplankton, micro-zooplankton and meso-zooplankton samples were vertically collected by using NORPAC® plankton net with 3 mesh sizes of 20, 60, 200 ㎛, respectively. The investigated fish tissues were directly collected at sampling fields. Sexes, body weight, body length, Hepatosomatic index (HSI) and Gonadosomatic index (GSI) of fishes were also determined. Water, sediment samples were transported in ice bag to the laboratory and keep at 4°C until extraction. Biota samples were stored in ice box filled with dry ice at field and kept at -20°C in laboratory until extraction.

Sample extraction and analysis: The method of [10] was applied for water sample extraction. Sediment samples was extracted based on the method of [11]. Biota samples were briefly extracted based on the method reported elsewhere [1, 2]. Concentration of PFCs were analyzed by the Agilent 1100™ HPLC interfaced with the Applied Biosystems API 2000™ electrospray ionization tandem mass spectrometer (ESI-MS/MS). 10 µL aliquot of the extracted sample was injected to a guard column connected serially to an analytical column. Temperature of analytical column was fixed at 35°C. Flow rate in mobile phase was 300 µL /min. To quantify the target materials in MS/MS, a multiple reaction monitoring mode was utilized.

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Procedural blanks were prepared to check possible contaminations occurred in the extraction procedure. The regression coefficient (r^2) of calibration curves for all target analytes which were drawn by 9 curve points of native external standards at 0.1, 0.2, 0.5, 1, 2, 5, 10, 20, 50 ng/L were higher than 0.99. The detection limit values of PFCs ranged from 0.01- 0.1 ng/L, 0.01 - 0.02 ng/g dry weight (dw) and 0.01 – 0.1 ng/g wet weight (ww) in water, sediment and biota, respectively. The recovery rate (%) of surrogate materials were in the acceptable range (from 73.6±5.8 to 130.8±26.9). The concentrations of target analytes were not corrected for recovery rates.

Statistic: In environmental chemistry, inadequate replacing data of nondetect observations (NDs) received from substitution methods such as ½ limit of detection (LOD), $\sqrt{2}$ LOD, 0.42 LOD, random values from zero to LOD and zero was reported in [12]. In this study, the parametric method of regression on order statistics function and the nonparametric method of Kaplan-Meier built in the statistical software of ProUCL 4.1 (USEPA) were utilized to treat data sets with 0 % < %NDs < 80%. Alternatively, if the %NDs in a data set was exceed 80% or number of distinct observation in a data set was smaller than 5, which is the minimum distinct observation size required to run ProUCL 4.1, all NDs were assigned as zero [13]. Spearman's correlation analysis and Student's t-test were also performed by using SPSS® (IBM, version 21) to investigate correlations and statistical differences between selected data groups.

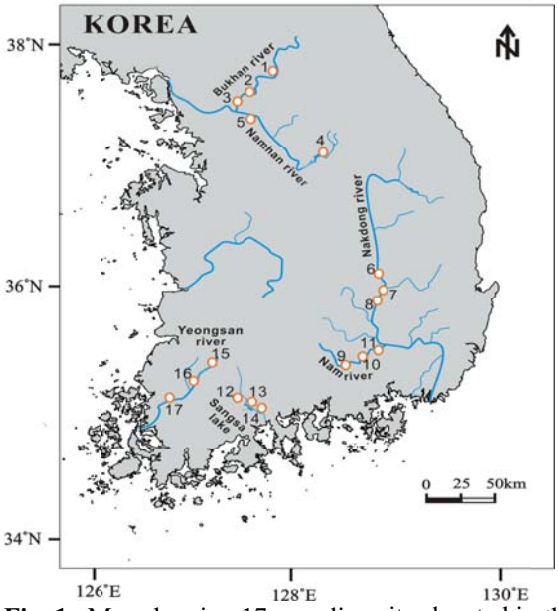

Fig. 1. Map showing 17 sampling sites located in the main rivers and lake in Korea

Results and discussions

The overall observations of PFCs in surveyed media were showed in Table 1.

Table 1. Overview of PFCs analysis results

Item	Water	Sediment	Plankton	Carp blood	Carp liver	Mandarin blood	Mandarin liver
Sampling site (n)*	17	17	2[a]	7[b]	7[b]	2[a]	2[a]
Analyzed sample (n)	19	27	12	69	69	20	20
Detected sample	n (%)	n (%)	n (%)	n (%)	n (%)	n (%)	n (%)
PFHxA	15 (79)	6 (22)	0 (0)	7 (10)	0 (0)	0 (0)	0 (0)
PFHpA	16 (84)	3 (11)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)
PFOA	17 (89)	24 (89)	0 (0)	24 (35)	9 (13)	19 (95)	9 (45)
PFNA	16 (84)	24 (89)	10 (83)	16 (23)	30 (43)	16 (80)	0 (0)
PFDA	12 (63)	24 (89)	1 (8)	69 (100)	69 (100)	19 (95)	2 (10)
PFUnA	15 (79)	23 (85)	6 (50)	69 (100)	69 (100)	20 (100)	20 (100)
PFDoA	12 (63)	27 (100)	7 (58)	69 (100)	67 (97)	20 (100)	15 (75)
PFHxS	18 (95)	9 (33)	0 (0)	29 (42)	4 (6)	0 (0)	0 (0)
PFOS	19 (100)	27 (100)	6 (50)	69 (100)	58 (84)	20 (100)	20 (100)
PFDS	0 (0)	0 (0)	0 (0)	1 (1)	9 (13)	18 (90	0 (0)
Average detected	14 (74)	17 (62)	3 (25)	35 (51)	32 (46)	13 (66)	7 (33)

*: site 4& 5 were surveyed in 2010 and others were surveyed in 2012; a: collected in site 4 and 5; b: collected in site 2, 4, 5, 7, 10, 13, 16

Water: PFOA and PFOS were steadily detected at the highest concentrations (Table 2). The mean percentages of PFOA and PFOS concentration in total PFCs concentration were 24.06% and 36.61%, respectively. The greatest concentrations of PFOA and PFOS were found in sampling site 7 and 16, respectively which are located in just downstream from discharge points of waste water treatment plants of Daegu and Gwangju metropolitan city. Except Nakdong river and Yeongsan river, the sum PFCs concentrations of water samples collected in other rivers and lake were mostly less than 10 ng/L.

The ratio of PFOS concentrations to PFOA concentrations was in the large range of 0.23 to 39.30 (mean = 4.15). This suggests that there were variety of PFCs sources contributing to the contamination of PFCs in water samples. Therefore, it's necessary to employ further investigations to clearly identify the existing and status of PFCs sources.

The PFOA and PFOS concentrations in the present study were relatively lower than other regions in Korea or in Japan, China and USA [3, 8, 9, 14, 15, 16, 17, 18, 19]. Excluding Bukhan river, Nam river and Sangsa lake, mean PFOS concentration investigated in Nakdong river, Yeongsan river and Namhan river were higher than Maximum Permissible Concentration in freshwater of 2.6 ng/L, the level at which no harmful effects on aquatic organisms are expected [20]. This result poses potential risks to the aquatic organisms and human consuming fishes in these rivers.

Table 2. Concentraion (ng/L) of PFCs in water showed in min~max (mean)

Site (n)	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnA	PFDoA	PFHxS	PFOS	PFDS	∑PFCs
Bukhan (3)	0.11~0.31 (0.18)	0.12~0.27 (0.19)	0.56~1.41 (0.94)	0.29~0.52 (0.38)	0.10~0.21 (0.14)	0.19~0.32 (0.24)	0.10~0.12 (0.11)	ND~0.72 (0.39)	0.83~1.84 (1.27)	ND	2.31~5.71 (3.85)
Namhan (4)	ND	ND~0.45 (0.26)	ND~0.64 (0.20)	ND~0.32 (0.08)	ND~0.11 (0.02)	ND	ND	0.50~3.97 (2.03)	0.67~6.25 (3.30)	ND	1.17~10.86 (5.90)
Nakdong (3)	0.51~7.94 (3.82)	0.71~3.43 (1.85)	3.56~8.34 (6.50)	0.83~4.49 (2.32)	0.53~4.80 (2.13)	0.28~1.13 (0.59)	0.13~0.33 (0.20	0.89~1.71 (0.21)	6.27~8.46 (7.36)	ND	14.71~40.63 (26.00)
Nam (3)	0.86~1.31 (1.03)	0.45~0.91 (0.68)	3.40~4.65 (3.84)	0.53~0.69 (0.62)	0.19~0.33 (0.26)	0.17~0.21 (0.20)	0.07~0.13 (0.10)	0.23~0.37 (0.32)	0.87~1.06 (0.98)	ND	7.09~9.61 (8.01)
Sangsa (3)	0.02~0.18 (0.10)	ND~0.18 (0.06)	0.29~0.63 (0.43)	0.14~0.33 (0.21)	0.05~0.07 (0.06)	0.10~0.13 (0.12)	0.06~0.08 (0.06)	0.03~0.11 (0.07)	0.25~0.99 (0.59)	ND	1.51~1.83 (1.70)
Yeongsan (3)	0.93~1.33 (1.11)	0.41~0.79 (0.60)	2.43~4.66 (3.97)	0.54~1.08 (0.85)	0.14~1.10 (0.64)	0.13~0.73 (0.41)	0.10~0.31 (0.21)	0.42~1.63 (1.03)	1.18~15.07 (11.06)	ND	8.47~25.19 (18.68)
Range	ND~7.94	ND~3.43	ND~8.34	ND~4.49	ND~4.80	ND~1.13	ND~0.33	ND~3.97	ND~15.07	ND	1.17~40.63
Mean	0.98	0.59	2.49	0.71	0.52	0.25	0.11	0.90	3.89	ND	10.44

Sediment: Mean PFOS concentration was 0.12 ng/g dw and contributed 32.40% in sum PFCs concentration. The PFOS concentrations in this study were relatively lower than those reported in previously studied in Germany, China and USA [11, 21, 24] but greater than other investigations employed in Japan [15, 22, 23]. PFDoA (mean concentration = 0.05 ng/g dw) was the next predominant PFC. Sum PFCs concentrations were ranged from 0.03 to 1.09 ng/g dw and 17 – 136 (mean = 57) fold greater than those in water samples. The sorption and desorption of PFCs depends on salinity [24, 25], pH and sediment characteristics [25] but interactions between PFCs concentrations in water and sediment haven't understood well until now.

Plankton: The greatest mean PFC concentration observed in the plankton samples was 2.08 ng PFOS/g ww. It is worth noting that the next greatest mean PFC which was detected as 0.36 ng PFDoA/ g ww was approximately 6 fold less than the mean concentration of PFOS. The mean concentrations of remaining detected PFCs were in the order of PFNA > PFUnA > PFDA. Li et al. 2008 [26] was also found the predominant role of PFOS in a zooplankton sample collected in Beijing, China. In this study, the mean concentration of PFOS in zooplankton samples (Table 3) were relatively less than those collected in China and Eastern Arctic [26, 27] but higher than those sampled in western Arctic [28].

Table 3. Range and mean concentration (ng/g ww) of PFOS and sum PFCs in plankton

	Phytoplankton	Micro-zooplankton	Meso-zooplankton
PFOS	ND~0.70 (0.21)	ND~11.07 (2.82)	ND~12.67 (3.21)
∑PFCs	0.30~2.15 (1.12)	0.10~12.47 (3.61)	0.20~12.98 (3.94)

Fish tissues: PFOS was consistently found at the highest concentration and accounted for 37.44%, 57.47%, 49.28% and 52.92% in sum PFCs concentration of carp blood, carp liver, mandarin fish blood and mandarin fish liver, respectively. Followed by PFOS, the next 3 predominant PFCs of PFUnA, PFDA, PFDoA were detected in all surveyed tissues. Mean PFOS concentrations of fish blood in this study were relatively lower than those in Korea, Japan, China reported by [29, 26, 14].

Similarly, FPOS, PFDA, PFUnA and PFDoA in fish liver or other tissues collected from Korea, Taiwan, Japan or USA reported in previous studies [3, 7, 14, 17, 29, 30] were higher than those in the present research. Significant correlation between PFOS and PFDA concentration in blood and liver were both found in crucian carp and mandarin fish (Fig. 2 and Fig. 3). This result suggests that blood can be used for nonlethal monitoring of PFOS and PFDA in these fishes.

Table 4. Range and mean concentration (ng/g ww or ng/ml) of PFCs in fish tissues

	Crucian carp (n=69)		Mandarin fish (n=20)	
f/m (ND)*	50/18 (1)		12/7 (1)	
BW (g)	76.40 ~ 973.19 (237.74)		52.58 ~ 424.60 (134.85)	
BL (cm)	12.50 ~ 32.00 (19.40)		15.20 ~ 29.40 (19.27)	
HSI	0.45 ~ 7.71 (2.93)		0.79 ~ 3.01 (1.67)	
GSI	0.29 ~ 16.23 (5.05)		0.09 ~ 2.77 (0.63)	
Concentration	Blood	Liver	Blood	Liver
PFHxA	ND ~ 0.36 (0.02)	ND	ND	ND
PFHpA	ND	ND	ND	ND
PFOA	ND ~ 0.89 (0.09)	ND ~ 0.33 (0.03)	0.06 ~ 0.34 (0.19)	0.09 ~ 0.33 (0.13)
PFNA	ND ~ 13.22 (1.46)	ND ~ 0.86 (0.07)	0.03 ~ 1.00 (0.21)	ND
PFDA	0.44 ~ 20.58 (5.15)	0.06 ~ 3.48 (0.75)	ND ~ 28.33 (12.20)	0.38~ 5.78 (1.68)
PFUnA	0.88 ~ 45.16 (7.11)	0.04 ~ 5.01 (0.80)	9.98 ~ 52.39 (20.32)	1.93 ~ 8.04 (4.53)
PFDoA	0.11 ~ 19.18 (3.20)	ND ~ 2.08 (0.43)	3.10 ~ 13.94 (6.74)	0.92 ~ 3.17 (1.76)
PFHxS	ND ~ 4.96 (0.17)	ND ~ 0.30 (0.01)	ND	ND
PFOS	0.18 ~ 145.23 (13.93)	ND ~ 43.76 (6.15)	3.68 ~ 233.68 (60.62)	1.61 ~ 114.99 (19.38)
PFDS	ND ~ 0.60 (0.04)	ND ~ 0.58 (0.05)	0.08~ 1.27 (0.44)	ND ~ 0.23 (0.01)
∑PFCs	1.72 ~ 236.29 (31.18)	0.15 ~ 54.64 (8.29)	31.08 ~ 296.72 (100.72)	6.13 ~ 131.58 (6.13)

*: female/male (not determined); BW: body weight; BL: body length

The ratio of PFCs concentrations in fish blood to corresponding concentrations in fish liver was widely altered. For instance, the ratio of PFOS concentration in blood to liver varied from 0.71 to 5.17 in crucian carp and from 0.75 to 12.53 in mandarin fish. This probably suggests a disequilibrium contaminating status in PFCs concentrations between liver and blood and indicates an ongoing expose of fish to PFCs [14]. With the pattern of protein rich tissues, it's necessary to note that PFOS accumulates in both liver and blood but binds to proteins rather than lipids [31, 32] and the contaminating level of perfluorinated acids among those tissues has been correlated with the presence of fatty acid binding proteins [33].

Only significant negative correlations were observed between HSI with the blood to liver ratio of PFOS and PFNA ($p < 0.05$) in crucian carp. The blood to liver concentration ratio of PFOS and PFNA thus decreased with the increasing of crucian carp HSI. Alternatively, significant positive correlation was detected between GSI with the blood to liver ratio of PFOS ($p < 0.05$) in mandarin fish. The blood to liver ratio of PFOS, therefore, increased with increasing sexual maturity of mandarin fish, which is represented by GSI.

Some previous studies have reported gender-specific differences in the PFCs concentration in aquatic animals [6, 34]. In our study, there was no significant difference ($p > 0.05$) in all PFCs concentration between sexes of

investigated fish tissues except PFNA in mandarin blood. The PFNA concentration in female mandarin fishes was significantly greater than those in males ($p < 0.05$). Interestingly, PFNA was also the only PFC has the significant positive correlation with crucian carp body weight and body length ($p < 0.05$). According to the above results, the different PFCs composition profiles in the surveyed fish tissues suggest the chemical compound-specific, fish species-specific and tissue-specific bioaccumulation.

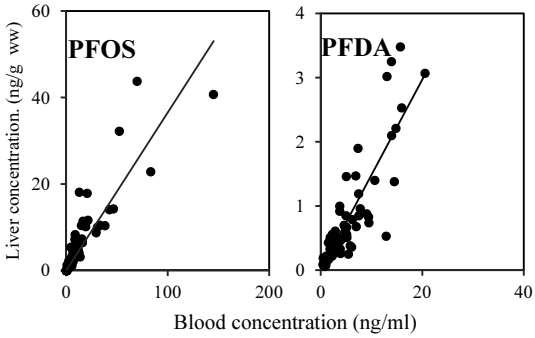

Fig. 2. Relationship between PFOS and PFDA concentrations in blood and liver of crucian carp (Spearman's correlation, n = 69, $r_{PFOS} = 0.953$, $p_{PFOS} < 0.001$; $r_{PFDA} = 0.494$, $p_{PFDA} < 0.001$)

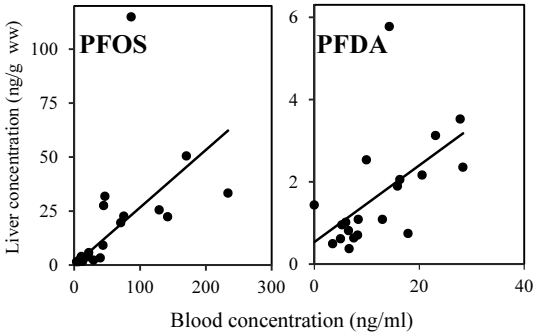

Fig. 3. Relationship between PFOS and PFDA concentrations in blood and liver of mandarin fish (Spearman's correlation, n = 20, $r_{PFOS} = 0.880$, $p_{PFOS} < 0.001$; $r_{PFDA} = 0.675$, $p_{PFDA} = 0.001$)

Bioconcentration factor (BCF): The present study showed increasing order of mean BCFs of PFOS in biota: phytoplankton (196 L/kg) < zooplankton (3,233 L/kg) < crucian carp liver (4,567 L/kg) < crucian carp blood (11,167 L/kg) < mandarin liver (24,718 L/kg) < mandarin blood (73,612 L/kg). This result was consistent with earlier studies which reported the positive correlations of PFOS concentrations in biota with the increasing of trophic level [1, 26, 27, 39] and the higher contaminating of PFOS in fish blood than liver [36].In addition, the similar patterns of BCFs of PFHxA, PFOA, PFDA, PFUnA, PFDoA and PFHxS regarding to fish blood and liver contaminating were also identified.

The average value of BCFs of PFOS observed in fish tissues in this study were relatively higher than those observed in variety tissues of fishes and some other aquatic animals in both field and laboratory studies reported in [31, 35, 36, 37, 38]. Although PFOS concentrations in water samples were comparable with PFOA concentrations, the BCFs of PFOA in biota were 18 – 100 fold less than those characterized by PFOS. Regardless for PFOS, only BCF of PFNA was determined in the collected plankton samples. The BCFs of PFNA in phytoplankton and zooplankton were 1,449 and 1,312 (L/kg), respectively.

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