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The dietary risk assessment of perfluorooctanoic acid (PFOA) and perfluorosulfonic acid (PFOS) in the root crops from the survey of the residue in agricultural soil and the crops

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Abstract

Perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) are persistent organic pollutants (POPs) that are prohibited from being produced and used. However, they remain in the agricultural environment and are transferred to crops. In addition, PFOA is classified as possibly carcinogenic to humans. To fully understand the exposure and risk of PFOA and PFOS for human in crops, a residue survey and estimation of dietary exposure assessment are needed. Therefore, we investigated the residues of PFOA and PFOS in upland soil and cultivated root crops. The average residues of PFOA and PFOS in the soil were < 0.054–0.541 μg kg^{-1} and 0.024–0.111 μg kg^{-1}, and 0.067–0.193 μg kg^{-1} and < 0.02 μg kg^{-1} in the crops, respectively. The average PFOA residues were higher than PFOS in the soil and crops. The estimated daily intakes of PFOA and PFOS in the crops were 0.284 ng kg_{bw}^{-1} day^{-1} and 0.023 ng kg_{bw}^{-1} day^{-1}, and the estimated hazard quotients were 0.355 and 0.013, respectively. In addition, the excess cancer risk of PFOA was calculated to be 1.99×10^{-8}. Thus, the non–carcinogenic and carcinogenic risks of PFOA and PFOS were not notable from the surveyed residues in the crops. However, the risks may be higher when the residues in other food crops are considered.

Keywords: Dietary exposure, Hazard quotient, Perfluorooctanesulfonic acid, Perfluorooctanoic acid, Estimated daily intake

Introduction

Perfluoroalkyl acids (PFAAs) are a group of synthetic perfluorinated compounds that have been extensively used in the fabric, paper, electronics, and many other industries since the end of the twentieth century [1–3]. However, they are listed as persistent organic pollutants (POPs) by the Stockholm Convention owing to their high stability, bioaccumulation factor, and the potential toxicity of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) in humans and wildlife [4–11]. PFOA and PFOS released from different products into the environment contaminate the air, soil, and water, and can remain in the environment for over a decade [6, 12–15]. In South Korea, PFOA and PFOS residues in the environment, including the agricultural environment, are surveyed and monitored under the POPs Control Act; the levels of these residues are reportedly below a part-per-billion [16–19]. Further, as environmental residues can be transferred and accumulated into crops, they

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should be controlled to keep them from entering the human food web [19–23].

Recently, the bioconcentration factors (BCFs) of PFOA and PFOS have been reported for various crops and animals. The factors were low (below 10) for crops; however, PFOA and PFOS were frequently detected in various crops [17, 19, 22–25]. The dietary exposure to PFOA and PFOS has not yet been fully estimated because of the lack of information on their residues in food crops [2, 21, 26].

The first reference doses (RfDs) of PFOA and PFOS were announced to be 1500 and 150 ng kg^{-1} day^{-1} in 2008 by the European Food Safety Authority (EFSA), and then revised to 0.8 and 1.8 ng kg^{-1} day^{-1} in 2018 [21]. In addition, PFOA is classified into Group 2B as a possible carcinogen for humans [27, 28]. Based on the revised RfDs of PFOA and PFOS, dietary exposure risk assessments have been performed on some food crops. However, the exposure risks have not been considered for root crops, which typically have higher residue levels than leafy and fruit vegetables because of their high BCFs [18, 29, 30]. Therefore, this study aimed to investigate the residues of PFOA and PFOS in upland soil and cultivated root crops, and perform a dietary risk assessment based on a residue survey focusing on upland soil during 2018–2019.

Materials and methods

Standards and reagents

PFOA and PFOS analytical standard solutions of native and isotope-labeled ($^{13}C_{4-}$ and $^{13}C_{8-}$) were purchased from Wellington Laboratories, Inc. (ON, Canada). All solvents (acetonitrile, methanol, tetrahydrofuran, and water) and reagents (acetic acid and ammonium acetate) were used a high–performance liquid chromatography (HPLC) grade from Merck (Germany). Hydrophilic–lipophilic balance (HLB) cartridges (0.5 g, 6 mL, Oasis, Waters Co., Ireland), powdered ENVI-Carb™ (Supelco, PA, USA), and membrane filter and syringe filter (nylon, Silicycle, Quebec, Canada) were purchased.

Sampling site and sampling

Soil and root crops (carrot, garlic, onion, potato, radish, and sweet potato) were collected from 176 different agricultural sites (6 provinces, 38 cities) in South Korea between April 2018 and November 2019. The soil samples were collected about 3 kg, to a depth of 0.15 m using an auger and placed in polypropylene (PP) bags. The soil samples were collected in triplicates in each farmland, and a composite representative for each site was obtained by mixing equal weights. The soil samples were dried at room temperature for 5 days in a fume hood, after passed through a 2 mm sieve, and stored at − 20 °C until analysis. In this study, the selected root crops were carrot, garlic, onion, potato, radish, and sweet potato. The Korean diet consists mainly of crops, and the intake of root crops selected in this study is high among the daily intake crops. Each crop sample was collected 3 kg on the farm with three replications. The sampled crops were ground with dry ice and stored at − 20 ℃ until analysis.

Analytical sample preparation for residual PFAAs in soil

The analytical method of residual PFAAs in soil was reported by Choi et al. [18] with slight modification. One gram of soil was extracted with 1.0% aqueous acetic acid (10 mL) with sonication for 20 min and mechanical shaking for an hour. The extracts were centrifuged at 4000 rpm for 10 min, and supernatants were collected in a new PP tube. The original soil was re–extracted with a mixture solvent with methanol and 1.0% aqueous acetic acid (9/1, *v/v*) (10 mL), and the extraction was repeated three times. The soil extract was concentrated to 15 mL under N_2 gas on Hurricane–Eagle (ChungminTech Co. Ltd., Seoul, Korea) and diluted with deionized water (DW) to 50 mL. HLB SPE cartridge was preconditioned with 10 mL of methanol, followed by 10 mL of DW, and the diluted sample was loaded at a rate of 1.3–1.6 mL min^{-1}. The cartridge was washed with 5 mL of 30% methanol and was eluent with 10 mL of methanol. The eluent was concentrated and re–dissolved with 1 mL of methanol. The extract was cleaned up with 20 mg of powdered ENVI-Carb™ and then vortexed and filtered using a syringe filter then isotope-labeled internal standards were added to the filtrate.

Analytical sample preparation for residual PFAAs in crops

The analytical method of residual PFAAs in crops was reported by Choi et al. [18] with slight modification. Briefly, the crops were washed under running water to remove soil, and the samples were ground with dry ice. Ten gram of sample was extracted with 75% methanol with sonication for 20 min and mechanical shaking for an hour. The extract was centrifuged at 4000 rpm for 10 min, and the supernatant was collected. The extracted crop was re–extracted using 75% of aqueous tetrahydrofuran (10 mL), and the extraction was repeated three times. All extracts were combined and concentrated to 10 mL under a nitrogen stream. As described above, the extract was diluted with DW to 50 mL and then extracted with HLB SPE. The eluent was concentrated and re–dissolved with 1 mL methanol. The extract was cleaned up with 20 mg of powdered ENVI–Carb™. The mixture was vortexed and filtered using a syringe filter then isotope–labeled internal standards were added to the filtrate.

Instrumental analysis

The LC-QTOF-MS system consisted of a Dionex UltiMate 3000 Quaternary Analytical LC high-pressure

Table 1 Analytical method validation of PFOA and PFOS in soil and crop

	Matrix	LOQ (μg kg^{-1})	Recovery (%)	CV (%)	Linearity (R^2)
PFOA	Soil	0.020	81.6	8.7	> 0.998
	Crop	0.002	73.1	9.2	> 0.999
PFOS	Soil	0.020	80.4	9.6	> 0.999
	Crop	0.002	70.4	8.9	> 0.996

liquid chromatography (HPLC) system and a Bruker Impact II TM Ultra-High Resolution Qq-Time-Of-Flight (UHR-QqTOF, Bruker, Billerica, MA, USA). The separations of PFOA and PFOS in soil and crops matrix were used in an ACQUITY UPLC BEH C18 column (1.7 μm, 100 mm × 2.1 mm, Waters, Milford, MA, USA), while a Luna C18 column (3 μm, 50 mm × 2.0 mm, Phenomenex. Inc., Torrance, CA, USA) as a pre-column was placed after pump exit to delay solvent impurity. The elute and detailed instrumental conditions were described in Additional file 1: Table S1.

Calculation on estimated daily intake (EDI) of agricultural products for PFAAs

EDIs of PFOA and PFOS were calculated based on the residual concentration of PFOA and PFOS in each crop, an estimate of the daily intake of crops, and average body weight (Eq. 1) [31, 32]. Food intake and average body weight by age were obtained from the 2019 Nation Food & Nutrition Statistics provided by the Korean Health Industry Development Institute (Additional file 1: Tables S2, S3) [33].

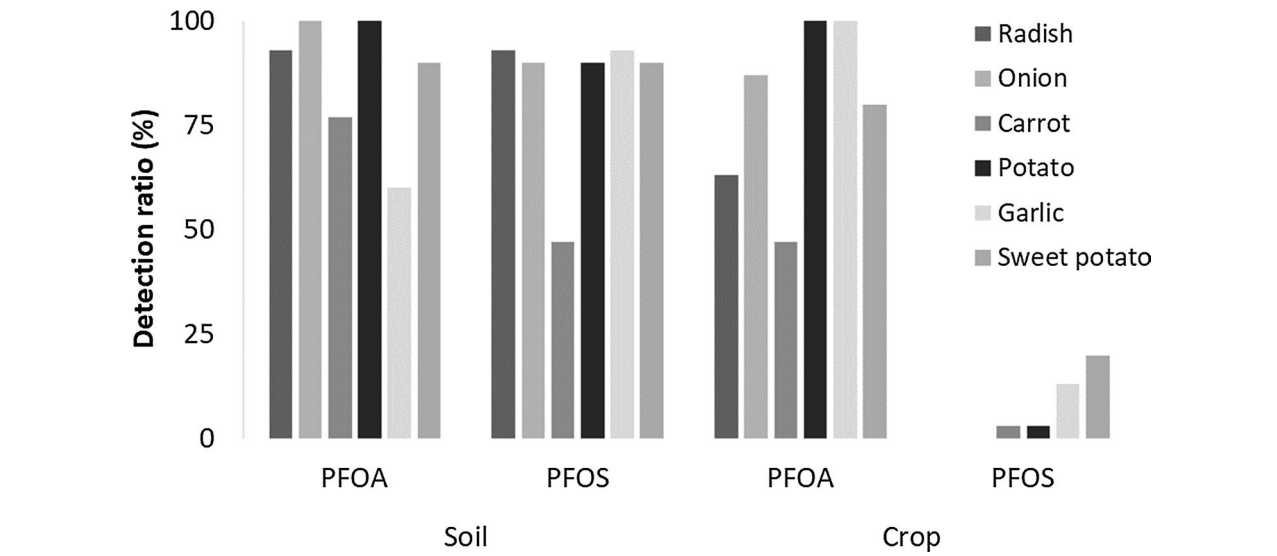

Fig. 1 Detection ratios of PFOA and PFOS in the upland soil and the cultivated crops in South Korea

Table 2 The residue concentrations (μg kg^{-1}) of PFOA and PFOS in upland soil and crops

Crop	Average residue (detection ranges, μg kg^{-1})			
	Soil		Crop	
	PFOA	PFOS	PFOA	PFOS
Radish	0.541 (< 0.020–5.579)	0.111 (< 0.020–0.698)	0.067 (< 0.020–0.356)	< 0.020
Onion	0.173 (0.032–0.586)	0.067 (< 0.020–0.342)	0.193 (< 0.020–0.698)	< 0.020
Carrot	0.124 (< 0.020–0.308)	0.061 (< 0.020–0.565)	0.088 (< 0.020–0.768)	< 0.020 (< 0.020–0.025)
Potato	0.156 (0.073–0.342)	0.065 (< 0.020–0.303)	0.150 (0.035–0.375)	< 0.020 (< 0.020–0.021)
Garlic	0.098 (< 0.020–0.342)	0.088 (< 0.020–0.359)	0.161 (0.070–0.471)	< 0.020 (< 0.020–0.247)
Sweet potato	0.054 (< 0.020–0.141)	0.024 (< 0.020–0.103)	0.111 (< 0.020–0.300)	< 0.020 (< 0.020–0.027)

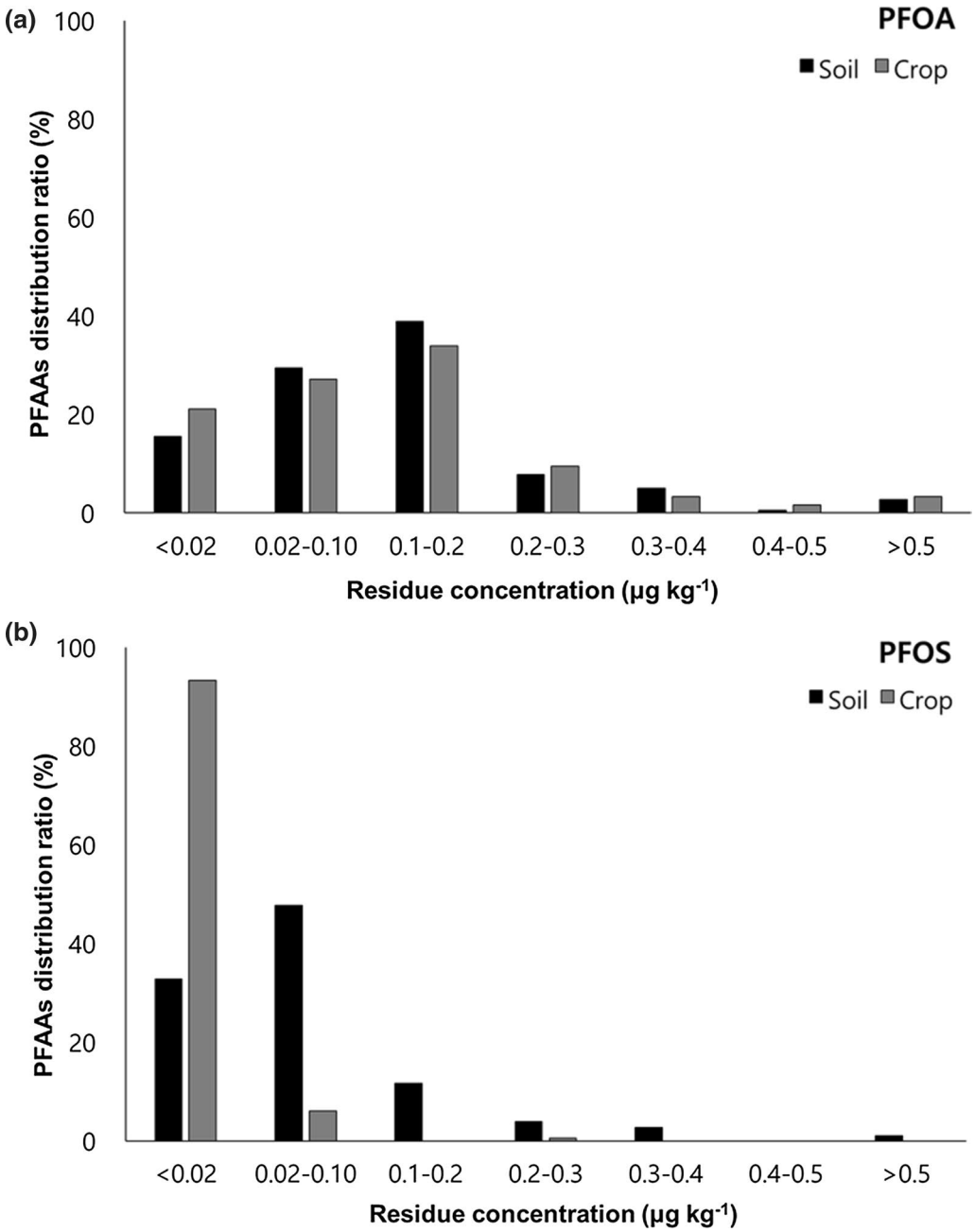

Fig. 2 Distribution of PFOA (**a**) and PFOS (**b**) in the upland soil and the cultivated crops in South Korea

$$\mathrm{EDI}\left(\mathrm{ng\,kg_{bw}^{-1}\,day^{-1}}\right)=\frac{\left\{\left[\text{Daily intake of crop per person}\left(\mathrm{g\,day^{-1}}\right)\right]\times\left[\text{Residual concentration}\left(\mathrm{ng\,g^{-1}}\right)\right]\right\}}{\text{Average body weight}\ (\mathrm{kg})} \quad (1)$$

Table 3 EDIs and HQs of PFOA and PFOS from the sampled crops

Crop	EDI (ng kg_{bw}^{-1} day^{-1})		HQ	
	PFOA	PFOS	PFOA	PFOS
Radish	0.044	0.004	0.055	0.002
Onion	0.124	0.005	0.155	0.003
Carrot	0.026	0.001	0.033	0.001
Potato	0.052	0.004	0.065	0.002
Garlic	0.011	0.004	0.014	0.002
Sweet potato	0.027	0.005	0.034	0.003
Total	0.284	0.023	0.355	0.013

Non–Carcinogen risk assessment of agricultural products for PFAAs

The hazard quotient (HQ) was calculated to evaluate the potential for non–cancer health hazards to occur from exposure to PFOA and PFOS with available non–cancer health guidelines as reference dose (RfD) (Eq. 2) [34]. RfD of PFOA and PFOS were obtained from EFSA [35].

$$\mathrm{HQ} = \frac{\mathrm{EDI}\left(\mathrm{ng\,kg_{bw}^{-1}\,day^{-1}}\right)}{\mathrm{Reference\ dose}\left(\mathrm{ng\,kg_{bw}^{-1}\,day^{-1}}\right)} \quad (2)$$

Carcinogen risk assessment of agricultural products for PFOA

The International Agency for Research on Cancer (IARC) classified the PFOA as possibly carcinogenic to humans (Group 2B) [29, 36]. The excess carcinogen risk (ECR) was calculated using the EDI and cancer slope factor (Eq. 3). For PFOA, US. Environmental Protection Agency (US EPA) estimated a cancer slope factor of 0.07 (mg kg_{bw}^{-1} $day^{-1})^{-1}$ [36].

$$\mathrm{ECR} = \left(\mathrm{ng\,kg_{bw}^{-1}\,day^{-1}}\right) \times \mathrm{Cancer\ slope\ factor} \left[\left(\mathrm{ng\,kg_{bw}^{-1}\,day^{-1}}\right)^{-1}\right] \quad (3)$$

Results and discussion

Quality assurance

Linearities of PFOA and PFOS were measured in the range of 0.020 and 2.00 μg L^{-1} and the R^2 were > 0.996 (Table 1). The limit of quantitations (LOQs) for PFOA and PFOS were determined to 0.020 μg kg^{-1} for soil and 0.002 μg kg^{-1} for crops. The analytical method validation was performed by determining the recoveries associated with the relative standard deviation of PFOA and PFOS. Recovery was measured at 0.050 μg kg^{-1} with $^{13}C_8$-PFAAs in spiked soils and the crops. The recoveries of PFOA and PFOS ranged from 70.4 to 81.6% in the soil and root crops. The precision of PFOA and PFOS ranged from 8.7 to 9.2% for soil and 8.9 to 9.6% for crops.

PFOA and PFOS residues in soil and crops

Residual PFOA and PFOS in the soil environment can be transferred and accumulated in cultivated crops. Thus, a survey of residues in the soil environment is the first step towards understanding crop residues and performing dietary exposure risk assessments for humans or animals. In this study, the residue survey focused on upland soil during 2018–2019. PFOA and PFOS were widely distributed in the agricultural environment and the detection ratios in the soil were ranged on 60–100% (average, 86.6%) and 47–93% (average, 83.8%), respectively (Fig. 1); and the average residues were 0.054–0.541 μg kg^{-1} for PFOA and 0.024–0.111 μg kg^{-1} for PFOS in the soil (Table 2). The total residue distributions of PFOA and PFOS in the soil was recorded to exceed 90% in < 0.05 μg kg^{-1} (Fig. 2) and each of the PFOA and PFOS residues was drastically decreased in comparison with the residues (< 0.05–1.57 μg kg^{-1} for PFOA and < 0.05–2.16 μg kg^{-1} for PFOS) in the national survey in 2013 [16]. In addition, the average residue of total PFOA and PFOS was 0.260 μg kg^{-1} which was four times lower than the downstream area of Nakdong-river in 2013–2017 [18]. This decrease in the soil could be explained by the prohibition of production and use of PFOA and PFOS by the enforcement of the POPs Control Act in Korea; the Act prohibited the production and use of PFOA and PFOS from 2013 [18].

In the crops, the detection ratios were 47–100% (average 79.5%) for PFOA and 0–20% (average 6.5%) for PFOS (Fig. 1). For PFOA, the residue distribution ratios in the soil were 29.4% for 0.02–0.1 μg kg^{-1} and 38.9% for 0.1–0.2 μg kg^{-1}. Similar residue distribution leves of PFOA were observed in the crops (Fig. 2); potato and garlic demonstrated detection ratios of 100%, whereas onion and sweet potato showed detection ratios of > 80% (Table 2). The average residues of PFOA in each root crop were 0.067–0.193 μg kg^{-1}. However, PFOS was not detected in most of the crops (93.3%) despite the residue distribution in the soil being widely identified. For PFOS, carrot, onion, potato, and radish recorded detection ratios < 5% (Table 2), and the detected residue distribution exhibited differently with PFOA (Fig. 2). The average residues of PFOS in each root crop were < 0.020 μg kg^{-1}. The highest detected residue for PFOA was 0.768 μg kg^{-1} in carrot, whereas this was 0.247 μg kg^{-1} for PFOS in garlic. These lower detection ratios and average residue levels were expected for PFOS compared with PFOA

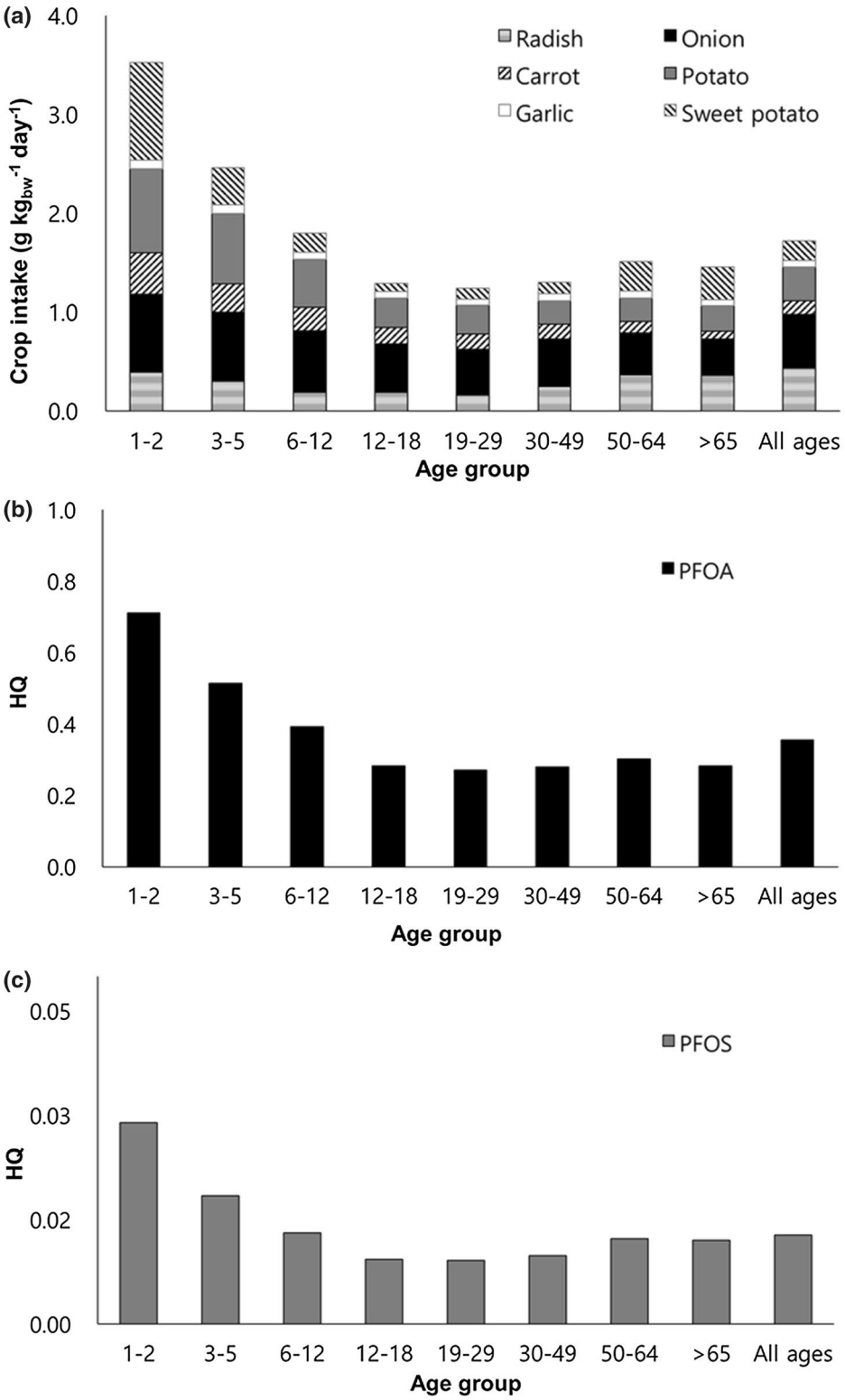

Fig. 3 Dietary intake amounts of the crops (**A**) and HQs (**B**) of PFOA and PFOS (**C**) by age group

Table 4 The ECR of PFOA in the root crops

Crop	ECR
Radish	3.10×10^{-09}
Onion	8.68×10^{-09}
Carrot	1.83×10^{-09}
Potato	3.62×10^{-09}
Garlic	7.89×10^{-10}
Sweet potato	1.88×10^{-09}
Total	1.99×10^{-08}

in crops because of the low BCF in root crops and the immobilization effect of soil minerals in the soil environment [18, 22, 37–39]. In the soil environment, PFOA and PFOS are adsorbed to the soil as electrostatic interaction, hydrophobic interaction, ligand, and ion exchange, and hydrogen bonding [37–39]. Millinovic et al. reported that PFOS showed both the highest sorption and the lowest sorption reversibility in the soil. In contrast, PFOA showed lower sorption and high reversibility compared with PFOS [37]. This difference would be due to the physicochemical properties such as hydrophobicity and functional group. In addition, Wei et al. reported that PFOS adsorption on soil was a positive correlation with soil physicochemical properties such as Al_2O_3, soil organic carbon, and Fe_2O_3 [38]. The sulfonate group in PFOS would be fixed as a metal combined complex with Al or Fe in soil mineral and then it would be immobilized in the soil environment [37–39].

Dietary exposure risk assessment of PFOA and PFOS

As dietary exposure route is considered a significant contributing route to PFOA and PFOS for humans, estimating the exposure amount is the first step in the risk assessment of the residues in root crops. The total EDIs of PFOA and PFOS for the root crops were 0.284 and 0.023 ng kg_{bw}^{-1} day^{-1}, respectively. The EDIs of PFOA and PFOS for each crop ranged from 0.011 to 0.124 ng kg_{bw}^{-1} day^{-1} and 0.001 to 0.005 ng kg_{bw}^{-1} day^{-1}, respectively (Table 3). Onion showed the highest EDI (0.124 ng kg_{bw}^{-1} day^{-1}), which it was two-fold higher than the previously reported EDI for onion (0.057 ng kg_{bw}^{-1} day^{-1}) in the downstream of Nakdong River [18]. Based on the reported RfDs, the total calculated HQs were 0.355 for PFOA and 0.013 for PFOS. The total HQ for PFOA was 27-fold higher than for PFOS.

When the HQ was analyzed by age group, the infant group (1–2 year olds) showed the highest HQs (0.711 for PFOA and 0.065 for PFOS), while the young adult group (19–29 year olds) had the lowest HQs (0.271 for PFOA and 0.021 for PFOS) (Fig. 3). The dietary intake of the food crops per bodyweight was higher in the younger age groups, although the intake amount per person was lower for the younger age groups than the adult groups (Fig. 3a). Further, the exposure risk of PFOA and PFOS for the infant group was three times higher than for the adult group.

As PFOA was classified as a possible carcinogen to humans in Group 2B by the IARC [29], the ECR of PFOA in the root crops was estimated based on the calculated EDI. The calculated ECR for each crop ranged from 7.89×10^{-10} to 8.86×10^{-9} (Table 4), and the total ECR was below the guideline value (1.00×10^{-6}). As there is a continuous decrease of the residue of PFOA and PFOS in the agricultural soil environment from the prohibition of use and production of the PFAAs, the residues and the EDIs in the crops were expected to decline slowly with time.

The dietary exposure risk for PFOA and PFOS was assessed from a residue survey of agricultural soil and cultivated root crops. The risk of non-carcinogenic and carcinogenic exposures of PFOA and PFOS in the surveyed crops did not exceed the safety guideline for Koreans. However, we determined that the exposure risk of PFOA in infants was higher than in the other age groups. Furthermore, the estimated total EDIs of PFOA and PFOS in dietary food for the Korean population would be higher than this because this didn't include the exposure amount from other major dietary sources of PFOA and PFOS such as meat, fish, and dairy. Thus, the risk reduction of PFOA (HQ = 0.711 for infants) would initially need to consider decreasing the residue in crop soil because these crops are an essential source of food and feed in the food chain.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s13765-022-00728-4.

Additional file1: Table S1 The instrumental condition of LC-QTOF-MS/MS for quantitative analysis of PFOA and PFOS. **Table S2** The average body weights by age. **Table S3** The daily food intakes (g day^{-1}) by age

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Author contributions

G–HC, D–YL, A–RS and B–YM performed the sampling of the analysis samples and quantitative analysis; G–HC and D–YL collected and discussed the results; J–HK designed and supervised all the results and wrote the manuscript. All authors read and approved the final manuscript.

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Availability of data and materials
The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests
The authors declare that they have no competing interests.

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