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Polychlorinated Biphenyls Residue in Citrus and Vegetables in the Jordan Valley, Jordan

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Abstract

In this study, four types of citrus fruits (Lemon, Orange, Mandarin, and Grapefruit) and four types of vegetables (eggplant, zucchini, cucumber, and tomato) irrigated with different types of water at three sites in the Jordan valley were evaluated for their polychlorinated biphenyl (PCBs) content.

The concentrations of six types of PCBs (PCB52, PCB101, PCB138, PCB153, PCB180, and PCB209) in the citrus fruits varied according to the type of the species and within each type.

The concentrations of PCBs in the citrus fruits were in the range of 2-13.8 ppb, 3.6-9.8 ppb, 11.0-138.0 ppb and 5.2-501.5 ppb for the grapefruit, orange, lemon, and mandarin, respectively. Although all citrus trees were irrigated with the same water, a big variation was observed with each fruit species suggesting other PCBs sources than the irrigation water; atmospheric deposition could, most probably, be a contamination pathway.

For the vegetables, the total PCBs concentrations were higher for the plants irrigated with the KTD water than those of the same plant species irrigated with mixed water which indicates that irrigation water and contaminated soils are the main sources of PCBs in the investigated vegetable types. This study has also found that the variation in the PCB levels in the fruits and vegetables irrigated with the same type of water is due to accumulation from other sources present in the environment rather than from the irrigation water.

Keywords: polychlorinated biphenyl, irrigation water, soil, citrus, vegetables.

1. Introduction

PCBs are a class of persistent organic pollutants (POPs) produced mainly by industry in the production of plasticizers, adhesives, and flame retardants among others (WHO / IPCS, 1993). The occurrence of some organic pollutants including PCBs in an aquatic environment is a major concern as they have been responsible for many health problems when existing above certain limits (Bakogluo et al., 2004, Ulrech, 2005). Therefore, they were banned from being produced or used by the Stockholm convention agreement 2001 (Stockholm convention, 2011). Although their use has been now largely phased out, but due to being highly resistant to breakdown, they still exist in the environment. The highly lipophilicity of these compounds causes their accumulation in the accessible food chains, therefore, many researches were done on their presence in the diets.

Human exposure to PCBs is risky, and that is why PCBs have been the target of many researches worldwide. Once PCBs are produced, they can be transported through the atmosphere and accumulate on other locations (Benanoue, 2009). PCBs can enter the food chains through different routes such as irrigation with polluted water (Al-Nasir and Batarseh, 2008, Shahalam et al., 1998) or when being transferred and deposited on fruits and vegetables.

There are many studies showing the uptake of PCBs by plants through irrigation with polluted water as well as plantation in contaminated soils. The accumulation of POPs in plants can vary due to different factors such as atmospheric pollution, leaf-morphology, and physiology (Franzaring and Van der Eerden, 2000).

In Jordan, many investigations showed that the irrigation water originating from the King Talal dam was within the acceptable international and national limits (Al-Omari et al., 2019 and Yahya et al., 2017). Fandi et al., 2009 maintains that the pollution tendencies of the surface waters of the KTD reservoir were attributed to high levels of organic pollutants. Al-Nasir and Batarseh, 2008 reported a PCBs residue in four types of vegetables (Okra, eggplant, paprika, and tomato). The uptakes of PCBs as well as other organic pollutants such as phenols and polycyclic aromatic hydrocarbons were dependent on the plant variety and plant part, and they showed different uptake concentrations.

The Jordan valley is known for its importance for food production in Jordan, and its products are partially exported to many countries around the world. Therefore, it is important to examine the contamination of the eatable parts of these products in terms of their PCBs content, for the reason that all previous investigations done in the area were on the fruit as a whole. Therefore, the present work is aimed at investigating the concentration of PCBs in irrigation water, soil, and the eatable parts of four types of citrus and vegetables irrigated with water of different sources to estimate its influence on these diets' contamination.

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2. Methodology

2.1 Study Area

The investigation area is located in the Jordan valley near Deir-Alla village at the mouth of King Talal Dam (KTD) in the Jordan valley. The area is characterized by its arid hot climatic conditions with intensive agricultural activities where many types of vegetables and fruits are planted.

The water used for irrigation originates from three sources, namely: a) King Talal dam collecting water originating from small springs, surface runoff and effluents from treated wastewater of the Kherbet Alsamra. The water quality of this water source is known to have a high concentration of many pollutants such as heavy metals, PAHs and PCBs (Batarseh,et al., 2003). b) Fresh water with a very low pollutant content from the King Abdullah Canal (KAC) (Jiries et al., 2004). c) a mixture of these two types.

2.2 Site Selection

The criteria of site selection are based on the type of water used for irrigation as mentioned earlier. The three selected sites are:

Site A: The area surrounding the KTD where the dam water is used without any modification for irrigation.

Site B: The area located to the north of Deir-Alla which receives only water from King Abdullah Canal without any impact of the KTD water.

Site C: The area located to the south of Deir-Alla where the irrigation water is a mixture of the KTD and the KAD water.

From site B, four types of eatable citrus plant species were selected (Lemon, grapefruit, orange, and mandarin). For sites A and C, four types of vegetables (eggplant, cucumber, squash, and tomato) from areas already planted, and on soil already irrigated with different types of water by the local farmers.

2.3 Sampling

2.3.1 Sample Collection

Water: Thirty-six water samples were collected on a monthly basis over a period of one year representing the three types of irrigation water used in this study. Sampling of each site was done in triplicates for quality assurance.

Soil: Sixty soil samples (20 samples from each site) were collected from the investigated area at two depths, from the upper soil (0-15 cm) and from the lower soil (15-30 cm) as most of the roots exist at these depths.

Plant: For plants, only the eatable parts of the plants were collected from each site as these are the parts which may have a direct impact on human health. The samples were collected at the end of the growing period when the fruits and vegetables are ripe and ready to be eaten. Four kinds of pealed citrus fruits from site B and four types of vegetables from sites A and C were selected for this study.

2.3.2. Sample Pre-treatment

The collected water samples were acidified for pH=2 and filtrated via a vacuum filtration apparatus. A one-litter sample was transferred subsequent to the Solid Phase Extraction (SPE) HLB Qasis column (cartridge) using a varian SPE apparatus equipped with a vacuum pump purchased from (Varian, Australia). For the soil and plants, the samples were homogenized and stored at -20 Co until the time of analysis.

2.3.3 Water Extraction

After the passage of the water samples through the pre-treatment stage, the SPE column was pre-conditioned prior to use. The samples were first washed with 6ml of ethyl acetate, followed by 6ml of methanol; and finally with 6ml of HPLC water. After that, the samples were loaded to the cartridge, and the cartridge was washed with 6ml 5% methanol in water. The targeted compounds were eluted with 6ml of ethyl acetate, and dried with Na2SO4, and the elute was concentrated to a final volume via a nitrogen stream. Finally, the residue was stored in amber glass vials for analysis.

2.3.4 Soil Extraction

The wet homogenized samples containing 50g of soil material were placed into a 500ml Erlenmeyer flask. Extraction was carried out with 2:1 acetone/water mixture (after being adjusted to its moisture content) (v:v) overnight using a horizontal shaker at a shaking velocity of 220 cycle/min. The liquid/liquid partitioning was performed by adding 15g of NaCl and 100ml cyclohexane, and the mixture was shaken for one hour. The organic layer was decanted into a 250ml Erlenmeyer flask and dried over 15g sodium sulfate. 100 ml of the extract was evaporated and dissolved in 5ml of 1:1 ethylacetate and a cyclohexane mixture (v:v), (Al-Nasir and Batarseh, 2008).

2.3.5 Plant Extraction

The wet homogenized samples containing 100g of fruit materials were placed into a 500ml Erlenmeyer flask. Extraction was carried out with 100ml of acetone overnight using a horizontal shaker at a shaking velocity of 220 cycle/min. The liquid/liquid partitioning was performed by adding 15g of NaCl and 100ml cyclohexane, and the mixture was shaken for one hour. The organic layer was decanted into a 250ml Erlenmeyer flask and dried over 15g sodium sulfate. 100ml of the extract was evaporated and dissolved in 5ml of 1:1 ethylacetate and a cyclohexane mixture (v:v), (Al Nasirand Batarseh, 2008).

2.3.6 Sample Cleanup

The sample solvents were concentrated to near dryness using a rotary evaporator, and the trace of solvents was removed under a gentle nitrogen stream. The alumina was activated overnight at 180oC, then, it was partly deactivated with 2% H2O and shaken for two hours by a horizontal shaker. 10g of the deactivated alumina and 1.0g of dried anhydrous Na2SO4 were loaded on top of the chromatography column to separate the non–polar from the polar constituents of the sample matrix. The analyzed samples were transferred to the alumina Column and eluted with 50ml of n-hexane, then in a rotary evaporator and were concentrated in a gentle nitrogen stream. The sample was divided into four portions each of 1ml into a GC amber vial for analysis as shown in chart 3.2 representing the stage of sample cleaning.

2.4 Analysis

A Schimadzu GC chromatograph model 2010 equipped with a 63Ni electron capture detector was used for the analysis of PCBs. A 30 m DB–5 fused silica capillary column with a 0.25 lm film thickness and a 0.32 mm inner diameter was used for the quantification of organochlorine compounds (J & W Scientific, USA). Helium (grade 5.5) was employed as carrier gas (1 ml min_1) and nitrogen as make up gas (58 ml min_1). The oven temperature was programmed from 60 _C (1 min) to 160 _C (1 min) at 15 _C min_1, then to 220 _C (5 min) at 5 _C min_1, finally at 3 _C min_1 to 280 _C (10 min). The injector and detector temperatures were maintained at 250 _C and 300 _C, respectively. The splitless injection volume was 1 ll. The Schimadzu Post-Run software was used for data analysis and quantification.

3. Results and Discussion

As vegetables and citrus are the main products in the Jordan valley, their quality is of vital importance. To ensure the population health, knowledge of diet quality intake of certain persistent pollutants such as PCBs is of vital importance. [Grassi et al. 2010]. The PCBs residue in both vegetables and pealed citrus fruits in the investigated sites are given below.

3.1 Vegetables

A total of forty-two vegetable samples were collected from the Jordan valley representing four types of vegetables and two types of irrigation water. The concentration of six types of PCBs (PCB52, PCB101, PCB138, PCB153, PCB180, and PCB209) in addition to total PCBs found in the respective vegetables were summarized in terms of minimum, mean, mean, and the percentage of the detected PCBs are shown in Table 1.

Table 1. PCBs concentrations in ppb for vegetables collected from sites A and C

	PCB52	PCB101	PCB138	PCB153	PCB180	PCB209	Σ PCBs	PCB52	PCB101	PCB138	PCB153	PCB180	PCB209	Σ PCBs
	Tomato (Site C) n=8							**Tomato (Site A) n=8**						
Min	0.4	0.6	BDL	BDL	BDL	3.0	1.0	24.0	5.6	BDL	9.3	1.5	19.5	1.5
Max	9.2	6.0	BDL	BDL	BDL	26.9	30.6	24.0	7.9	BDL	9.3	1.5	19.5	51.4
Mean	3.4	2.4	BDL	BDL	BDL	13.4	13.4	24.0	7.1	BDL	9.3	1.5	19.5	22.6
Detection%	83	83	ND	ND	BDL	83	ND	20	20	ND	50	20	20	ND
	Zucchini (Site C) n=8							**Zucchini (Site A) n=8**						
Min	5.4	18.4	0.6	BDL	1.0	6.8	1.6	1.7	4.5	2.8	BDL	0.8	0.7	7.3
Max	5.4	18.4	0.6	BDL	1.0	6.8	25.2	10.3	13.5	7.0	BDL	2.6	44.3	73.2
Mean	5.4	18.4	0.6	BDL	1.0	6.8	13.4	4.7	9.9	4.6	BDL	1.7	22.5	27.2
Detection%	20	20	20	ND	20	20	ND	80	60	60	ND	40	40	ND
	Eggplant (Site C) n=8							**Eggplant (Site A) n=8**						
Min	0.1	7.3	0.2	0.8	0.1	0.2	1.3	2.7	11.6	5.5	13.7	1.8	1.1	13.4
Max	1.1	11.0	0.2	0.8	7.8	0.2	18.8	58.2	74.6	5.5	24.4	1.8	25.2	104.6
Mean	0.6	9.2	0.2	0.8	3.9	0.2	9.5	28.5	34.7	5.5	19.0	1.8	13.1	52.2
Detection%	40	40	20	20	40	20	ND	60	60	20	40	20	40	ND
	Cucumber (Site C) n=8							**Cucumber (Site A) n=8**						
Min	0.1	0.5	0.3	0.4	0.2	0.3	0.4	3.7	0.5	0.1	BDL	5.5	BDL	0.5
Max	0.1	0.9	0.3	0.4	0.2	0.5	1.5	3.7	2.8	0.1	BDL	5.5	BDL	9.2
Mean	0.1	0.6	0.3	0.4	0.2	0.4	0.9	3.7	1.6	0.1	BDL	5.5	BDL	4.2
Detection%	14	43	14	14	43	57	ND	25	50	25	ND	25	ND	ND

n represents The number of samples, BDL represents below detection limit, and ND represents Not Determined.

There was a great variation in the types of PCBs among the vegetable types as well as in the total PCBs concentrations (the sum of individual PCBs in each sample) within each type of vegetable as well as between vegetable types and the irrigation water used. Generally, the total PCBs concentrations were significantly higher for the plants irrigated with KTD water than the same plant species irrigated with mixed water type. This indicates that water quality is the main source of PCBs in the investigated area. For vegetables irrigated with KTD, eggplant showed the highest sum of PCBs concentrations ranging from 13.4 to 104.6 ppb with a mean value of 52.2. On the other hand, cucumber showed the lowest concentration ranging from 0.4-1.5 ppb with a mean value of 0.9 ppb. The high variation in the PCBs residue in the different plant species could be attributed to the physiology of the plants rather than to the irrigation water and soil quality.

In general, the occurrence of different types of PCBs was not congener within each vegetable species as its occurrence and concentrations varied with the plant species at the same site. However, it can be confirmed that PCB52, PCB101 and PCB209 were the most detected PCBs in tomato samples irrigated with KTD as they were detected in more than 83% of the samples.

3.2 Citrus

For citrus, a total number of twenty-one citrus samples were analyzed for six types of PCBs (PCB52, PCB101, PCB138, PCB153, PCB180, and PCB209) in grapefruit, orange, lemon and mandarin. A statistical summary (minimum, maximum and means) of individual PCBs concentrations as well as their sum for four types of citrus are presented in Table 2.

Table 1. PCBs concentrations in ppb for different types of citrus fruits collected from the investigated site

	PCB52	PCB101	PCB138	PCB153	PCB180	PCB209	Σ PCBs	PCB101	PCB138	PCB209	Σ PCBs
	Grapefruit n=5							Orange n=5			
Min	0.1	0.1	0.1	1.1	0.7	0.1	2.0	1.0	1.9	0.7	3.6
Max	0.3	6.9	7.0	1.6	4.1	0.1	13.8	29.3	1.9	9.8	9.8
Mean	0.2	3.5	4.4	1.3	2.5	0.1	8.3	15.1	1.9	4.9	5.8
Detection %	40	40	80	40	80	20		40	20	75	
	Lemon n=5						Mandarin n=6				
	PCB52		PCB101	PCB180	PCB209	Σ PCBs	PCB52	PCB101	PCB180	PCB209	Σ PCBs
Min	7.5		22.9	1.6	74.8	11.0	1.1	37.8	0.4	2.9	5.2
Max	20.2		42.5	20.2	74.8	138.0	234.2	37.8	218.0	119.0	501.5
Mean	12.8		32.7	7.9	74.8	128.2	62.2	37.8	73.8	30.0	109.6
Detection %	80		40	60	20	ND	66	15	50	100	ND

n represents The number of samples, BDL represents below detection limit, and ND represents Not Determined.

Among the citrus samples, lemon showed a mean concentration higher than those of other analyzed citrus samples as its mean concentration was 128.2 ppb ranging from 11.0 to138.0 ppb, followed by mandarin with a mean concentration of 109.6 ppb ranging from 5.2 to 501.5 ppb, followed by grapefruit with a mean concentration of 8.3 ppb ranging from 2.0 to13.8 ppb. Orange showed the lowest concentration of 5.8 ppb ranging from 3.6 to9.8 ppb.

The types of detected PCBs in the analyzed samples varied according to the citrus species. For orange, only three PCBs were detected (PCB101, PCB138, and PCB209), whereas for lemon and mandarin, four types of PCBs were detected (PCB52, PCB101, PCB180, and PCB209). For grapefruit, all the analyzed PCBs were detected. This could be attributed to the physiology of the plant rather than to the environment which was the same for all of the citrus types including the large surface/content ratio of these different citrus species.

The concentration of PCBs in the citrus samples were much lower than that in the vegetables which is due to the sample preparation, as the citrus fruits were pealed before extraction which removed a high quantity of PCBs with the skin.

3.3 Irrigation Water

PCBs were analyzed in twenty-four irrigation water samples (8 samples from each site). The results showed that only few PCBs were found in the irrigation water at low concentrations. At site A, only three types of PCBs were detected, PCB101 was found in only one sample at a concentration of 0.012 ppb, PCB138 was found in two samples at the concentrations of 0.01 and 0.035 ppb, and PCB153 was found in only one sample at a concentration of 0.004 ppb. For site B, only two samples showed a PCB content as it was 0.152 ppb from PCB101 and 0.08 ppb from PCB209. Site C showed a higher variety as four PCBs were detected in the irrigation water. Two out of eight samples showed a PCBs content. PCB101, PCB138, and PCB153 were detected at the concentrations of 0.017,0.062 and 0.019 ppb respectively in one sample. The other sample contained three types of PCBs (PCB101, PCB138 and PCB180) at the concentrations of 0.013, 0.059 and 0.012 ppb respectively. Therefore, the irrigation water is not the source of PCBs in the investigated sites.

3.4 Soil

The concentration of PCBs collected from the three investigated site are summarized in Table 3 in terms of minimum, mean, median, and maximum percentages. Results showed that there is a variation in the abundance and concentration of PCBs among the sites as well as within each site. The highest concentration was found at site A with an average value of the sum of PCBs 108.7 ppb ranging from 9.9 - 373.4 ppb. The most frequent detected PCB was PCB101 as it was detected in all of the analyzed samples. Other PCBS were detected at different frequencies.

Table 3. PCBs concentrations in ppb for soils at sites irrigated with different types of irrigation water

	PCB52	PCB101	PCB138	PCB153	PCB180	PCB209	Σ PCBs
Soil (Site B) n=9							
Min	1.1	2.0	1.2	1.4	1.6	1.0	2.6
Max	34.4	7.0	1.6	4.6	1.6	1.0	15.3
Mean	7.6	4.8	1.4	3.0	1.6	1.0	8.3
Detection %	100	56	22	22	11	11	ND
Soil (Site A) n=7							
Min	5.1	5.6	2.0	1.2	0.1	BDL	9.9
Max	5.1	363.6	36.6	9.7	1.0	BDL	373.4
Mean	5.1	97.9	10.0	4.5	0.7	BDL	108.7
Detection %	14	100	72	57	57	ND	ND
Soil (Site C) n=7							
Min	0.7	0.4	2.8	BDL	0.3	BDL	0.4
Max	3.9	176.5	94.0	BDL	1.5	BDL	107.0
Mean	1.7	40.0	48.4	BDL	0.9	BDL	24.2
Detection %	57	86	29	ND	43	ND	ND

n represents The number of samples, BDL represents below detection limit, and ND represents Not Determined.

The lowest PCBs were detected at site B, as the source of water is mainly fresh groundwater resources. The concentration at this site ranged from 2.6 to 15.3 ppb with an average value of 8.3 ppb. PCB52 was the most detected type as it existed in all of the analyzed samples, whereas PCB180 and PCB209 were detected in only one sample out of the nine analyzed samples.

The concentration of PCBs at site C showed values ranging between site A and site B as the irrigation water is a mixture of the water at site A and site B. The PCBs concentration at this site ranged from 0.4 – 107.0 with an average value of 24.2 ppb. The most frequent PCB was BCB101 which was detected in 86% of the analyzed samples.

The PCBs concentrations in some of the soil samples collected from site A were much higher than those collected from industrialized cities like Moscow. They ranged from 3.1 to 42 ppb [Wilcke et al., 2006], however, the other investigated sites were comparable with the PCBs concentrations in soils irrigated with wastewater at other sites in the world such as Shenyang, China where their concentrations ranged from 4.4-20.14 ppb [Song et al., 2006].

Therefore, it can be concluded that the main source of PCBs in the investigated area was the irrigation water as it was highest at site A and lowest at site B.

4. Conclusions

The concentrations of six target PCBs were found to vary according to the type of fruits as well as with the irrigation water. The different plant types showed different concentrations of various PCBs. Among the citrus fruits, lemon showed the highest concentration and orange was the lowest among the analyzed citrus samples. For vegetables, the impact of irrigation water was clear as vegetables irrigated with KTD water showed higher PCBs values than those irrigated with the mixed water. Variation within each type of the target fruits and vegetable was noticed indicating that irrigation water was not the main source of PCBs but other contamination pathways existing in the environment including the atmospheric deposition.

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