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Semi-volatile organic pollutants in Jordanian surface water

Yahya R. Tahboub ^{a,b,*}, Mohammad F. Zaater ^a, Dima F. Khater ^a

^a Department of Chemical Sciences, Faculty of Science and Arts, Jordan University of Science and Technology, Irbid 22110, Jordan

^b Department of Chemistry, Faculty of Science, Islamic University in Medina, Medina, Saudi Arabia

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Abstract Jordan relies heavily on rainwater stored in reservoirs because it has extremely limited alternative water resources. These reservoirs are essential for drinking water and irrigation, so monitoring their water quality is extremely important. In this study we monitored 55 semi-volatile organic chemicals (SVOCs) in monthly samples from the Zarqa River and three reservoirs between April and August 2010, and also screened the samples for non-target pollutants. Twelve SVOCs were detected and quantified at King Talal Dam followed by ten at Zarqa River and four each at Wadi Al-Arab and Mujib dams. Phenol and phthalate esters were detected at wide range concentrations (0.01–25 µg/L) in all water samples. Phenol has the highest level at the Zarqa River (18.5 ± 5.5 µg/L) followed by King Talal (12.5 ± 2.5 µg/L) and Wadi Al-Arab (2.5 ± 0.63 µg/L). Phthalate esters (total) were detected at the Zarqa River, King Talal, Wadi Al-Arab, and Mujib dams at levels 3.85, 3.75, 1.03 and 0.12 µg/L. Other contaminants, including polyaromatic hydrocarbons (PAHs), were detected at low concentrations in the King Talal Dam reservoir and Zarqa River samples. Two non-targeted phosphate plasticizers, tri-*n*-butylphosphate and tris(2-chloroethyl)phosphate were identified in the July and August Zarqa River samples. A comparison study reveals that SVOC pollution of surface water in Jordan is within acceptable international limits, but long-term monitoring programs should be implemented.

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1. Introduction

Jordan is amongst the ten countries that are poorest in water resources. Rainfall is the only renewable source of

water in Jordan. Over 90% of the annual rainfall (8000 million cubic meters [MCM]) is lost to evaporation. Therefore, the Jordanian government has constructed dams to store excess rainfall in reservoirs in the rainy seasons. Some reservoirs, such as the King Talal Dam reservoir, also receive treated waste water (Alqadi and Kumar, 2011). Monitoring the quality of surface water in Jordan is very important because the reservoirs are essential sources for drinking water and other domestic and agricultural activities (Fandi et al., 2009). Monitoring programs for heavy metals and volatile organic chemicals, especially trihalomethanes (THMs), and persistent organochlorine pollutants (POPs) have been established for many drinking

* Corresponding author at: Department of Chemical Sciences, Faculty of Science and Arts, Jordan University of Science and Technology, Irbid 22110, Jordan. Tel.: +962 795561392.

E-mail address: tahboub@just.edu.jo (Y.R. Tahboub).

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and surface water resources in Jordan (Al-Omari et al., 2004; Batarseh, 2011).

The occurrence of harmful substances in the environment, including anthropogenic semi-volatile organic compounds (SVOCs), is a major concern. SVOCs, including polyaromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), phthalic acid esters (PAEs), organochlorine pesticides (OCPs) such as DDT, and polybrominated diethyl ethers (PBDEs), have been responsible for a range of health problems in humans and animals (Ohe et al., 2004; Ulrich, 2005; Bakoglu et al., 2004). Some SVOCs can be transported in the atmosphere and condense in relatively cold locations, such as surface water bodies (Benanou, 2009). PCBs and some OCPs have been internationally classified as persistent organic pollutants (POPs) and banned from being produced or used by the Stockholm Convention agreement in 2001 (Stockholm Convention, 2011). Monitoring programs for organic contaminants, including SVOCs, in water usually use GC/MS analysis. A relatively large water sample (up to 1 L) is usually extracted by either liquid–liquid extraction (LLE) (Schwarzbauer and Heim, 2005; Schwarzbauer and Ricking, 2010) or solid phase extraction (SPE) (Li et al., 2011; Yoshida et al., 2004). Reconstituted extracts are analyzed by GC/MS.

We report, for the first time, the results of monitoring 55 target SVOCs, which are considered to be high priority pollutants, in Jordanian surface water. Non-target pollutants are also reported, but were not monitored. Sampling was performed between April and August 2010.

2. Experimental

2.1. Sample sites and sampling

Four sites, located in different regions of Jordan, were selected for monitoring, and are shown in Fig. 1. The Wadi Arab Dam is in northern Jordan and can store up to 17 MCM of water from that region's rainfall. The Wadi Mujib Dam is located in southern Jordan and can store up to 31 MCM of water from that region's rainfall. The King Talal Dam is the major dam in Jordan, and is located in the middle of the country, on the Zarqa River. It can store more than 70 MCM of rainwater from the middle region of Jordan and treated wastewater from the Al-Samra natural water treatment plant. The Zarqa River, also known as Seal Jarash, transfers rainwater and treated wastewater to the King Talal Dam reservoir, and crosses many industrial areas in the middle of Jordan.

Monthly water samples were collected from the four sampling sites between April and August, 2010. Sampling was performed according to USEPA (1982) handbook for sampling and sample preservation of water and wastewater. Bank sampling was employed for collecting samples from shallow Zarqa River, while boat sampling was employed for dams. The water samples were collected using 1 L pre-cleaned amber glass vessels, from the middle of the three reservoirs and at 30 cm depth from the Zarqa River. The samples were analyzed within two weeks of sampling, to avoid analyte degradation. Samples were stored in the dark, in a refrigerator at 4 °C. The samples were filtered through 47 mm diameter, 0.45 µm pore size fiberglass filters supplied by Whatman (Maidstone, UK).

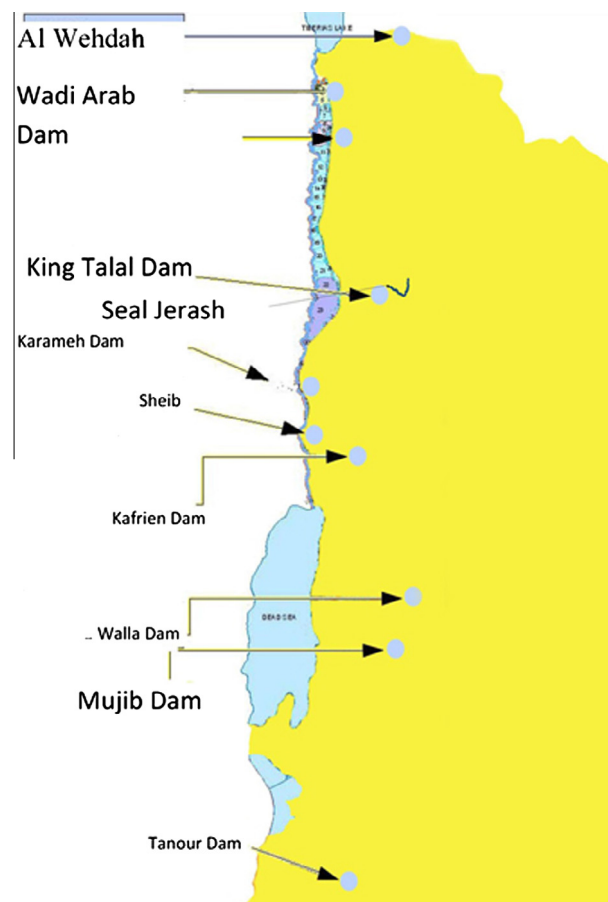


Figure 1 Map of Jordan showing its major dams.

2.2. Reagents and standards

Pesticide quality dichloromethane and acetone were purchased from Lab-Scan (Gliwice, Poland), pesticide grade petroleum ether and hexane was purchased from Scharlau Chemie (Barcelona, Spain), and anhydrous sodium sulfate was purchased from AppliChem (Darmstadt, Germany).

A standard containing 1000 µg/mL of 55 SVOCs (EPA 625 mixture) in CH₂Cl₂ was purchased from Sigma–Aldrich (St Louis, MO, USA). The mixture components are shown in Table 1. Internal standards 2-nitrophenol, fluoranthene and di-*n*-octyl phthalate were also purchased from Sigma–Aldrich.

2.3. Extraction

A serial liquid–liquid (LLE) extraction was performed at two pHs, pH > 11 for base/neutral extractable analytes and pH < 2 for acid extractable analytes. One liter of a sample was transferred to a 2 L separatory funnel, and 60 mL of a 1:1 mixture of dichloromethane and petroleum ether was used for the extraction. After each extraction the organic layer was allowed to separate from the aqueous phase for at least 20 min. The extraction procedure was repeated twice at each pH. The combined extract was dehydrated by passing it through anhydrous sodium sulfate. The dried extract was rotary evaporated (40 °C, 130 rpm).

Table 1 Targeted SVOACs with their GC/MS retention times and quantification ions.

Compound name	Quantification ions	Retention time (min)	Compound name	Quantification ions	Retention time (min)
Poly aromatic hydrocarbons (PAHs)			Hexachlorobenzene	284, 142, 249	24.00
Naphthalene	128, 127, 129	15.08	Nitrobenzene	123, 93, 77	12.93
2-Chloronaphthalene	163, 164, 127	18.78	2,6-Dinitro toluene	165, 63, 89	20.03
Acenaphthylene	152, 151, 153	20.06	Methyl 4,6-dinitro toluene	179, 177, 103	–
Fluorene	166, 165, 167	22.27	Phenols		
Phenanthrene	178, 179, 176	25.28	Phenol	94,66, 39	10.41
Anthracene	178, 176, 179	25.45	2-chlorophenol	128, 65, 130	10.61
Fluoranthene	202, 101, 203	29.97	2-nitrophenol	139, 109, 65	13.92
Pyrene	202, 200, 203	29.76	2,4-dimethyl phenol	107, 122, 77	14.22
Benzo(a)anthracene	228, 229, 226	33.63	2-methyl-4,6-dinitrophenol	198, 105, 121	22.54
Chrysens	228, 226, 229	33.74	4-chloro-3-methyl phenol	107, 142, 77	17.01
Benzo(b)fluoranthene	252, 253, 125	37.97	2,4,6-trichloro phenol	196, 198, 200	18.23
Benzo(k)fluoranthene	252, 253, 125	38.02	2,4-dinitro phenol	184, 154, 63	20.83
Benzo(a)pyrene	252, 253, 125	38.17	4-nitrophenol	139, 109, 65	21.19
Indeno(1,2,3)pyrene	276, 138, 227	–	Pentachloro phenol	184, 154, 107	24.67
Dibenzo(a,h) anthracene	258, 139, 279	–	2,4-dichloro phenol	162, 63, 98	14.67
Benzo(g,h,i) perylene	276, 138, 227	–	Chlorinated ethers		
Phthalate Esters (PAEs)			Bis(2-chloro ethyl)ether	93, 63, 95	10.6
Dimehtyl phthalate (DMP)	163, 194, 164	19.91	Bis(2-chloro isopropyl)ether	45, 41, 121	–
Diethyl phthalate (DEP)	149, 177, 150	22.18	Bis(2-chloroethoxy)	93, 63, 123	14.51
Di- <i>n</i> -butyl phthalate (DNP)	149, 150, 104	27.43	4-chlorophenoxy	204, 206, 141	
			Phenyl ether		
			Others		
Butyl benzyl phthalate	149, 91, 206	32.16	Hexachloro butadiene	225, 223, 227	15.59
Bis(2-ethylhexyl) phthalate	149, 167, 43	34.17	Isophorone	92, 95, 138	13.72
Di- <i>n</i> -octyl phthalate	149, 167, 150	36.83	Carbozole	167, 165, 139	26.04
Chloro- and nitrated benzenes			Azobenzene	77, 51, 182	22.88
1,3-Dichlorobenzene	146, 148, 111	11.04	Hexachloroethane	117, 119, 201	12.68
1,4-Dichlorobenzene	146, 148, 111	11.28	N-nitrosoamine	42, 74, 44	3.93
1,2-Dichlorobenzene	146, 148, 111	11.69	4-bromophenyl phenyl ether	248, 250, 141	23.92
1,2,4-Trichlorobenzene	180, 182, 145	14.89			

to 5 mL and then concentrated to 0.30 mL under a stream of nitrogen. 50 μ L of internal standard mixture, 2-nitrophenol for benzenes and phenols, fluoranthene for PAHs and di-*n*-octyl phthalate for PAEs, was added to each sample before quantitative analysis. These internal standards never detected in Jordanian surface water.

2.4. GC/MS analyses

A Shimadzu OP5050 GC/MS system (Tokyo, Japan), consisting of a GC-17 gas chromatograph, an OP-5050 mass spectrometer, and a Class-5000 data system, was used for the instrumental analysis. Separation was achieved using an Optima 5 accent capillary column (30 m, 0.25 mm, 0.25 μ m film thickness) (Macherey Nagel, Düren, Germany). The carrier gas was 99.999% helium, used at a flow rate of 0.7 mL/min. The injector and transfer line temperatures were 300 °C and 280 °C, respectively. 1 μ L of each sample was injected in split mode with a split ratio of 1:10. The MS was used in electron ionization (EI⁺) mode, at 70 eV. Both full scan mode (total ion chromatogram, TIC), for identification, and selective ion monitoring (SIM) mode, for quantification, were used. The temperature program was: initial temperature 70 °C (held for 1 min), increased by 20 °C/min to 180 °C, increased by 2 °C/min to 240 °C (held for 1 min), increased by 8 °C/min to 310 °C (held for 10 min).

3. Results and discussion

Our main objective was to monitor the concentrations of SVOACs in Jordanian reservoirs. Fifty-five pollutants from the EPA 625 method were targeted, and two non-targeted plasticizers, tri-*n*-butylphosphate and tris(2-chloroethyl)phosphate were also found. Other pollutants, relating to natural organic humic acids were identified but not quantified.

Fig. 1 shows the eleven major dams in Jordan. The Al-Wehda Dam has the largest reservoir, with a capacity of 150 MCM. However, it is only filled to 10% of its capacity, for political reasons, and, therefore, was not considered in this study. The other three selected reservoirs make up more than 75% of the total Jordanian water storage capacity. We also monitored the Zarqa River because it transfers treated waste water from the Samra natural water treatment plant (> 20 MCM/year) to the King Talal Dam reservoir. The monitoring period was from April to August because the other months are either rainy (December to March) or the reservoirs are drained of most of their water (September to November) for irrigation and domestic purposes.

The target compounds and their GC/MS parameters are summarized in Table 1. Our validation results were within accepted values using standard methods for water and wastewater analysis (Eaton and Franson, 2005).

Figs. 2 and 3 show typical GC/MS total ion current (TIC) chromatograms for the water samples taken from the King

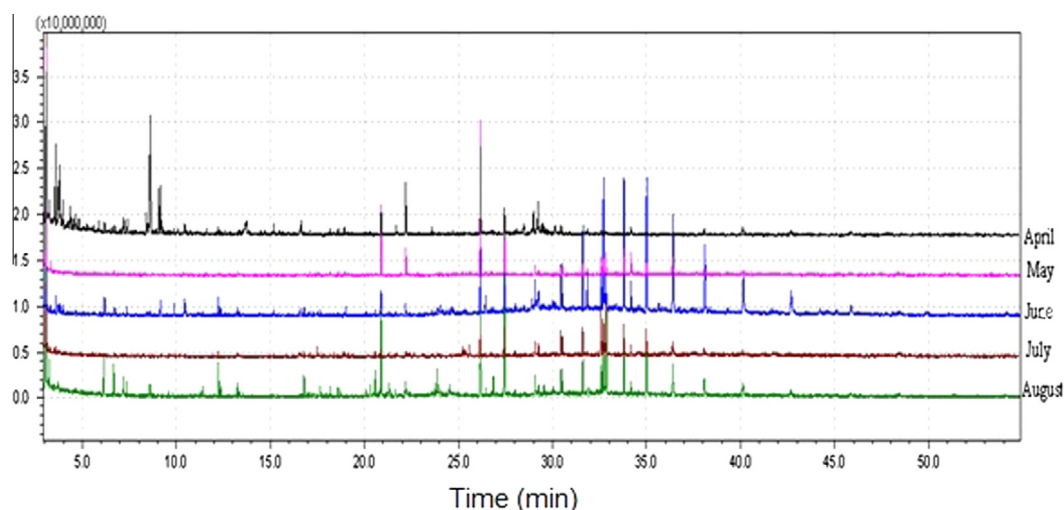


Figure 2 Typical GC/MS TIC chromatograms for water samples collected from the King Talal Dam reservoir.

Talal Dam reservoir (Fig. 2) and the other three sites (Fig. 3A–C). Fig. 2 includes the GC/MS chromatograms for the monthly samples taken from the King Talal Dam reservoir, as overlays, to illustrate the consistency of the chromatographic peaks present and their monthly intensity variations. The monthly sample overlays are shown because treated surface water from the Zay treatment plant was contaminated with wastewater on many occasions. To determine if the source of contamination was the King Talal Dam reservoir,

these chromatograms were used as a chemical fingerprint for King Talal reservoir water.

Table 2 presents results of identified SVOCs and their levels in each reservoir. Twelve compounds were identified and quantified in King Talal Dam followed by ten in Zarqa River and four each in the other two dams. Two non-targeted phosphate plasticizers, tri-*n*-butylphosphate and tris(2-chloroethyl)phosphate were identified in the July and August samples of Zarqa River. Concentration levels of SVOCs in King Talal Dam and

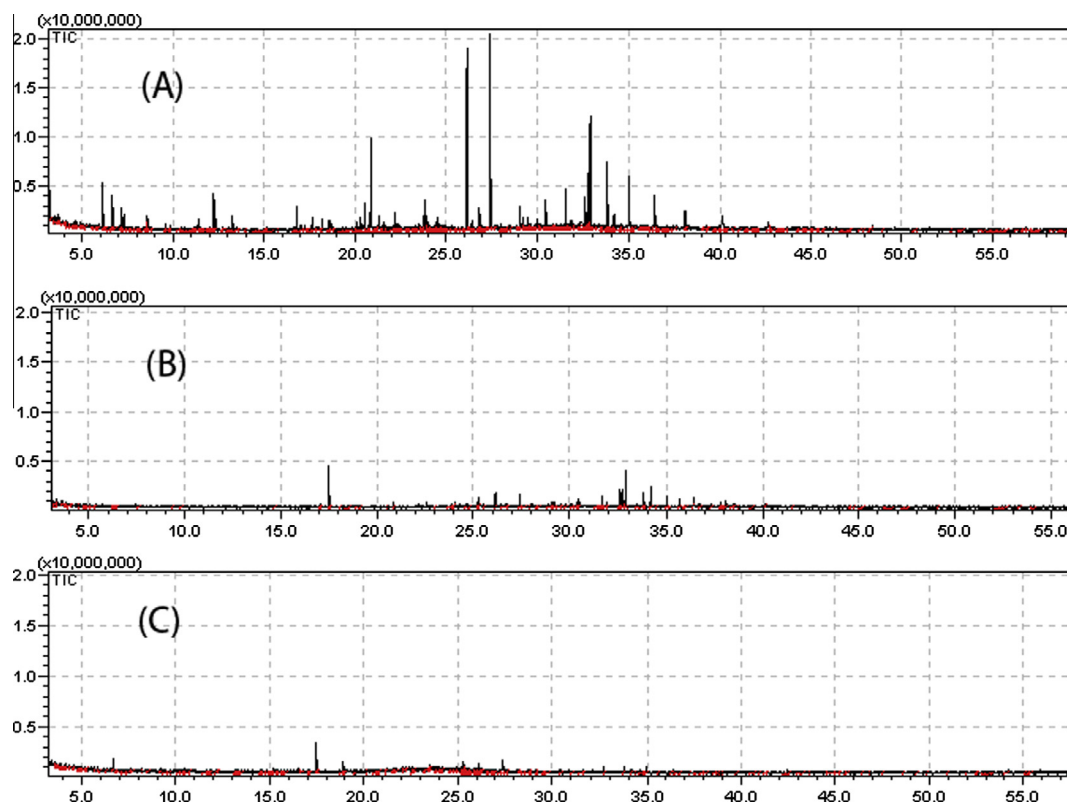


Figure 3 Typical GC/MS TIC chromatograms for water samples collected in August from: A: the Zarqa River (Seal Jarash); B: the Wadi Arab reservoir; C: the Wadi Mujib reservoir.

Table 2 Targeted SVOCs identified and quantified in studied sites.

Compound	Mean \pm S.D ($\mu\text{g/L}$)		Wadi Al-Arab	Mujib
	King Talal	Zarqa River		
Di- <i>n</i> -butylphthalate (DnBP)	1.20 \pm 0.35 (0.55–1.6)	1.60 \pm 0.75 (0.82–2.1)	0.35 \pm 0.15 (0.23–0.80)	0.05 \pm 0.03 (0.01–0.08)
Butyl benzyl phthalate (BBP)	1.80 \pm 0.45 (0.75–2.1)	1.40 \pm 0.66 (0.75–2.1)	0.43 \pm 0.23 (0.25–0.70)	0.03 \pm 0.02 (0.01–0.07)
Bis(2-ethylhexyl)phthalate (DEHP)	0.75 \pm 0.15 (0.45–0.95)	0.85 \pm 0.35 (0.25–1.15)	0.25 \pm 0.15 (0.05–0.40)	0.04 \pm 0.03 (0.01–0.06)
Phenol (Ph)	12.5 \pm 2.5 (8.2–14.6)	18.5 \pm 5.5 (10.5–24.6)	2.50 \pm 0.63 (1.53–0.32)	ND
Dichlorophenol (DCPh)	0.25 \pm 0.15 (0.15–0.40)	0.45 \pm 0.35 (0.15–0.80)	ND	ND
1,2-Dichlorobenzen (DCB)	0.04 \pm 0.03 (0.01–0.07)	0.05 \pm 0.35 (0.02–0.10)	ND	0.01 \pm 0.01 (ND–0.02)
Acenaphthalene (Acen)	0.015 \pm 0.010 (0.005–0.020)	0.025 \pm 0.035 (0.015–0.070)	ND	ND
Naphthalene (Naph)	0.035 \pm 0.025 (0.010–0.070)	0.055 \pm 0.035 (0.015–0.095)	ND	ND
Phenanthrene (Phen)	0.007 \pm 0.005 (ND–0.010)	0.015 \pm 0.025 (0.010–0.040)	ND	ND
Anthracene (Anth)	0.005 \pm 0.005 (0.005–0.010)	0.008 \pm 0.010 (0.003–0.020)	ND	ND
Pyrene (Py)	0.005 \pm 0.010 (ND–0.015)	ND	ND	ND
Benzo(a)anthracene (BAnth)	0.003 \pm 0.008 (ND–0.011)	ND	ND	ND

ND: not detected.

Table 3 Detected SVOCs at King Talal Dam, cited literature and international values ($\mu\text{g/L}$).

SVOC	(1)	(2)	(3)	(4)	(5)
DnBP	1.20	0.453	0.582	NT	19 ^b
BBP	1.80	5.75	0.04	NT	–
DEHP	0.75	1.15	0.463	NT	1.30 ^a , 16 ^b
Ph	12.5	NT	NT	2.26	7.7 ^a , (46), 4 ^b
DCPh	0.25	NT	NT	6.0	0.2 ^b
1,2-DCB	0.04	NT	NT	NT	0.7 ^b
Acen	0.015	0.785	0.013	0.12	5.8 ^b
Naph	0.035	NT	0.132	0.55	1.2 ^a , 1.1 ^b
Phen	0.007	0.316	0.029	0.10	0.40 ^b
Anth	0.005	1.005	0.002	0.179	0.10 ^a
Py	0.005	0.225	0.026	0.06	0.025 ^b
BAnth	0.003	0.449	0.003	0.131	0.018 ^b

(1) King Talal. (2) Wu et al., 2009. (3) Pirierto et al., 2007. (4) Kot-Wasic et al., 2004. (5) Guideline values.

NT: not targeted.

^a EU, 2008.

^b Canadian water quality guidelines for protection of aquatic life (CCME, 2013).

Zarqa River are much larger than respective SVOCs in the other reservoirs. The Wadi Al-Arab and Mujib dam reservoir samples were of drinking water quality and most pollutants were found at much lower concentrations than their guideline values (WHO, 2011) in all samples.

Phenol was the major contaminant in the Zarqa River and King Talal Dam with levels of 18.5 and 12.5 $\mu\text{g/L}$, and with much lower concentration at Wadi Al-Arab Dam (2.5 $\mu\text{g/L}$). Phenol is extensively used as antiseptic in Jordan. Phthalate esters (DnBP, BBP, DEHP) were detected at significant concentrations ($\mu\text{g/L}$) in all water samples. DEHP is environmentally a major concern, but its major root is food. WHO sets

8.0 $\mu\text{g/L}$ as guideline value for DEHP in drinking water. This value is much higher than the largest detected value (1.15 $\mu\text{g/L}$ at Zarqa River). Six out of sixteen targeted PAHs were detected in water samples collected from the King Talal Dam reservoir and the Zarqa River, but, at much lower concentrations (ng/L).

Table 3 presents a comparison between King Talal Dam results and three similar studies conducted at the Yangtz River (China) (Wu et al., 2009), estuarine water (Lamjako-Spain) (Pirierto et al., 2007) and the Vistuala River (Poland) (Kot-Wasic et al., 2004). Comparable results were reported for the four studies. Total PAEs vary between 1.085 (Pirierto et al., 2007)

and 7.35 µg/L for (Wu et al., 2009). King Talal Dam has a median value of 3.75 µg/L. However, King Talal Dam has much lower total PAHs of 0.070 µg/L followed by 0.205, 1.140 and 2.78 µg/L for the other three sites.

Another comparison was made between King Talal Dam results and guideline values for surface water (EU, 2008; CCME, 2013). Phenol only exceeds guideline values by a factor of 2 (EU, 2008) and 3 (Alberta Environment (1999)), however, EU (2008) allows maximum allowed concentration of 46 µg/L. DCPH is slightly higher (0.25 versus 0.20 µg/L), while, other parameters especially PAHs have much smaller values. These comparisons confirm the good quality of Jordanian surface water regarding pollution with SVOCs.

4. Conclusions

This study is the first to report comprehensive analysis of SVOCs in Jordanian surface water. These results show that King Talal Dam and Zarqa River have larger number of pollutants and concentration levels. This conclusion is expected since both of them are located in middle Jordan, where about 80% of population reside. King Talal Dam and Zarqa River water are currently used for restricted irrigation. Wadi Al-Arab Dam is located in northern Jordan and Mujib Dam is located in southern Jordan. Both of them have much refined water than the other two reservoirs. Their water is currently used for restricted and unrestricted irrigation. Comparison study of similar studies along with international guideline values (Table 3) reveals the good quality of Jordanian surface water.

We conclude that the SVOC pollution of surface water in Jordan is within acceptable international limits, but long-term monitoring programs, especially for plasticizers, phenolic chemicals, and polyaromatic compounds, should be implemented.

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