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Determination of DDT and Metabolites in Surface Water and Sediment Using LLE, SPE, ACE and SE

**Linda L. Sibali · Jonathan O. Okonkwo ·
Caliphs Zvinowanda**

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Abstract Surface water and sediment samples collected from Jukskei River in South Africa, were subjected to different extraction techniques, liquid–liquid (LLE), solid-phase extraction (SPE), activated carbon extraction (ACE) and soxhlet extraction (SE) for sediment. The samples were extracted with dichloromethane, cleaned in a silica gel column and the extracts quantified using a Varian 3800 GC-ECD. The percentage recovery test for 2,4′DDT, DDE and DDD and 4,4′DDT, DDE and DDD in water ranged from 80%–96% and 76%–95% (LLE); 56%–76% and 56%–70% (SPE) and 75%–84% (ACE), respectively; while that recoveries for sediment samples varied from 65%–95% for 2,4′DDT, DDE and DDD and 80%–91% for 4,4′DDT, DDE and DDD. The high recoveries exhibited by ACE compared very well with LLE and SE. This was not the case with SPE which exhibited the lowest value of recoveries for both 2,4 and 4,4′DDD, DDE and DDT standard samples. The mean concentrations of DDT and metabolites ranged from nd-1.10 µg/L, nd-0.80 µg/L, nd-1.21 µg/L and 1.92 µg/L for LLE, SPE, ACE and SE, respectively. The total DDT (2,4′ and 4,4′-DDT) in water and sediment samples ranged from 1.20–3.25 µg/L and 1.82–5.24 µg/L, respectively. The low concentrations of the DDT metabolites obtained in the present study may suggest a recent contamination of the river by DDT.

Keywords Extraction methods · DDT · Metabolites · Surface water · Sediments

Organohalogenated compounds (OHCs), particularly organochlorine pesticides (OCPs) are ubiquitous environmental contaminants. Of all the OCPs, dichlorodiphenyltrichloroethane (DDT) has received most attention because of its early success for the eradication of malaria-carrying mosquitoes in many countries. The impact of DDT on the environment was first brought to light in the early 1960s. Consequently, the use of DDT for both agricultural and public health was discontinued in several countries. However, some countries including South Africa, has been exempted to use it only for public health purposes in some parts of the country where malaria is rife.

DDT and its metabolites are highly lipophilic, bioaccumulative, persistent and have the ability to interfere with hormonal functions in mammals and wildlife (Colborn 1991). Several researchers have reported the detection of these compounds in various fatty tissues, particularly in human milk (Takahashi et al. 1981; Kanja et al. 1986; Bouwman et al. 1990; Atuma et al. 1998; Okonkwo et al. 1999, 2008; Okonkwo and Kampira 2002; Mutshatshi et al. 2008), in sediments (Iwata et al. 1994), water (Hernandez et al. 1993; Meintjies et al. 2000; London et al. 2000; Sibali et al. 2008) and air (Iwata et al. 1994).

Since DDT and its metabolites can occur in the environment at low concentration levels, efforts have been geared towards developing sensitive extraction or preconcentration methods before analysis. For water samples, methods such as liquid–liquid extraction (LLE) and solid-phase extraction (SPE) (Hernandez et al. 1993) appear to be the most popular with solid-phase micro-extraction making an entry just recently. Activated carbon has also

L. L. Sibali
Scientific Services, Rand Water, Vereeniging 1930, South Africa

J. O. Okonkwo (✉) · C. Zvinowanda
Department of Environmental, Water and Earth Sciences,
Faculty of Science, Tshwane University of Technology, Private
Bag X680, Arcadia, Pretoria, South Africa
e-mail: OkonkwoOJ@tut.ac.za

been used for preconcentrating OCPs before analysis. For sediment extraction, methods such as Soxhlet extraction (SE) supercritical fluid extraction (SFE), microwave assisted extraction (MAE) and accelerated solvent extraction (ASE) have been reported for OCP determination (Naude et al. 1998; Molins et al. 1997; Heemken et al. 1997). The problem with the aforementioned hyphenated methods (with the exception of SE) is one of cost and maintenance.

Several reports have been published on the detection of DDT and its metabolites in water and sediment using GC-ECD from various countries (Hernandez et al. 1993; Iwata et al. 1994). In South Africa, studies of water monitoring for OCPs, particularly, DDT have shown widespread detection of this compound in surface water and sediment samples (Hassett et al. 1987; Naude et al. 1998; Meintjies et al. 2000; London et al. 2000; Fatoki and Awofolu 2003; Sibali et al. 2008). These studies used predominantly LLE, SPE and SE for water and sediment samples, respectively, but not activated carbon. The present study compares the sensitivity of ACE method to the traditional methods such as liquid–liquid (LLE) and solid-phase extractions (SPE) in preconcentrating environmental problematic compounds such as DDT and its metabolites. There has been a recent call to use granulated activiated carbon for water treatment instead of others because it is most robust and reliable (Burke 2006). We have reported on an improved method to quantify DDT in surface water using activated carbon (Okonkwo et al. 2007). The use of activated carbon is in line with the search for cheaper and more environmentally friendly extraction methods for routine purposes. In addition this study is expected to show whether the level of contamination within the study area is high enough to be a public health concern. Such information is extremely important as South Africa is geared towards formulating a water quality protection strategy, in relation to endocrine disruptors.

Materials and Methods

The study area for this investigation was Jukskei River Catchment in South Africa as shown in Fig. 1. A high potential for pollution exists in the Jukskei River Catchment, since urban runoff from the metropolitan area of Johannesburg readily reaches the river system. The potential sources of the pollutants in the catchment include the sewage treatment works at Midrand, Diepsloot (Johannesburg Northern Sewage Treatment works), Modderfontein and Esther Park, industrial input from Kelvin Power Supply and AECI in Modderfontein, informal housing areas and agriculture, notably irrigated agriculture.

Water samples were collected using 2.5 L Winchester bottles from the river mentioned above in March, July and October from 2005 to 2006, from several sampling points to represent upstream, middlestream and downstream. Before use the bottles were first rinsed with the water samples and thereafter immersed about 5 cm below the surface. The water samples were thereafter preserved, by adding about 5 mL analar grade HNO_3 and kept at 4°C before use. Sediment samples were collected from about the same locations as the water samples into clean wide-

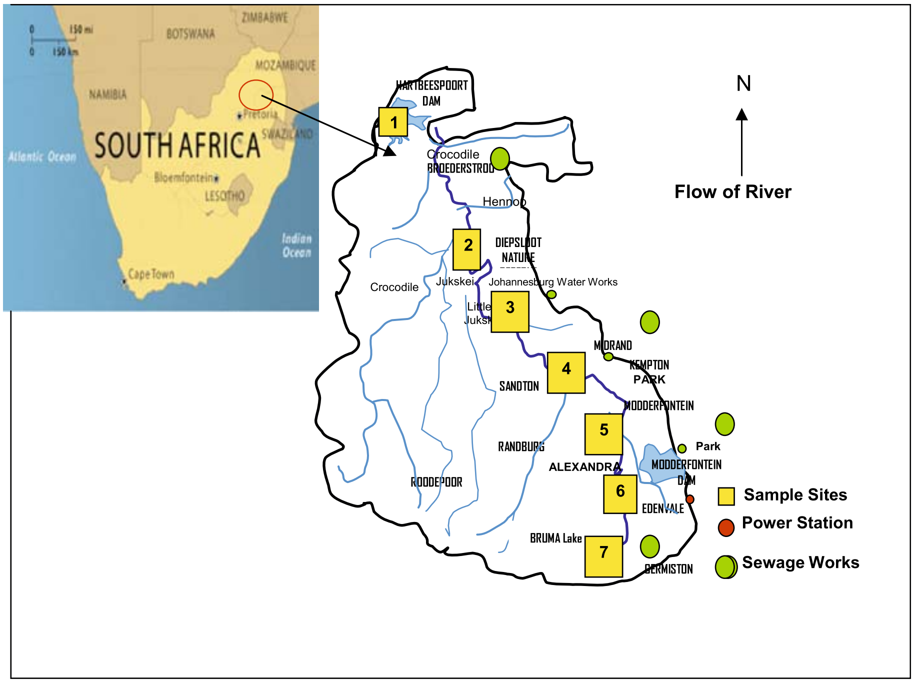

Fig. 1 Map of South Africa (*top left*) and sampling sites (*bottom right*) at the Jukskei River catchment Area (DWAF 1992; Sibali et al. 2008). *Note*: 1 = Haartebeesport Dam – S1 (S1BS1 and S1BS2), 2 = Before Johannesburg Water Works – S2 (SB2 and SM2), 3 = After Johannesburg Water Works – S3 (SB3 and SM3), 4 = Sandton /Khayalami – S4 (SB4 and SM4), 5 = Marlboro – S5 (SB5 and SM5), 6 = Alexander – S6 (SB6 and SM6), 7 = Bruma lake – S7 (S7BS1 and S7BS2)

mouth glass bottles from a depth below 5 cm and kept frozen prior to sample analysis.

The glassware used were of Pyrex brand and was soaked overnight in 25% chromic acid after which it was washed thoroughly with liquid soap and rinsed with de-ionized water and acetone and then finally oven dried at 100°C overnight. The Winchester bottles (2.5 L) and wide-mouth glass bottles (1 L) used for water and sediment sample collections, respectively were also subjected to the same cleaning regime described earlier. All solvents, *n*-hexane and dichloromethane (Alfa Aesar) used were spectrophotometric grade (99+%) and HNO_3 analar grade. The silica gel (70–230 mesh), anhydrous sodium sulphate and activated charcoal (50–100 mesh) used in the column clean up and adsorption experiments, respectively were analytical grade obtained from Industrial Analytical (Pty) Ltd. Before use the anhydrous sodium sulphate and activated carbon were pre-heated in a muffle furnace between 350°C and 400°C for 4 h. All organochlorine standards used were obtained from Dr. Ehrenstorfer Gmbh, Augsburg, Germany, and these are shown in Table 1.

A Varian 3800 Gas Chromatograph (GC) coupled with electron capture detector (ECD) and capillary column DB-5 (5%-phenyl–95%-dimethylpolysiloxane, 30 m × 0.25 mm i.d and 0.25 μm thickness). The oven temperature was programmed as follows: 100°C for 1 min ramped at 20–150°C and then ramped at 7°C/min to 280°C. Injector and detector temperatures were 260°C and 300°C, respectively. Carrier gas used was helium (99.99%) at a flow rate of 3 mL/min, and nitrogen (99.99%) used as the make-up gas. The split ratio was 1:20. These instrument conditions were found to be adequate for optimum results. All analysis was carried out in triplicate and the injection volume was 1 μL.

Standard stock solutions (1000 μg/mL) of the organochlorines were prepared in dichloromethane and diluted to lower working concentrations. The lower concentrations were then injected (1 μL) into the GC and the retention time recorded as means of six determinations. Quantitation of unknown samples was accomplished by the comparison of peak heights for peaks in the unknown samples, confirmed by the retention times, to those of calibration mixtures of standard pesticides.

Table 1 List of DDT and its metabolites investigated

Compound	Molecular mass	Molecular formula	% Purity
2,4′-DDD	320	$C_{14}H_{10}Cl_4$	99.9
4,4′-DDD	320	$C_{14}H_{10}Cl_4$	99.9
2,4′-DDE	324	$C_{14}H_8Cl_4$	99.9
4,4′-DDE	320	$C_{16}H_8Cl_4$	99.9
2,4′-DDT	354	$C_{14}H_9Cl_5$	99.9
4,4′-DDT	355	$C_{16}H_9Cl_5$	99.9

The response factor (RF) of the organochlorine standards relative to the internal standard, polychlorinated nitrobenzene (PCNB), were carried out by injecting a mixture of the standards together with the internal standard at a concentration range of 0.1–100 μg/mL. The response factor was calculated using the equation:

$$\text{Response Factor} = \frac{\text{Peak area of analyte}}{\text{Peak area of IS(PCNB)}} \quad (1)$$

A calibration of instrument response against concentration was therefore obtained. Polychlorinated nitrobenzene (PCNB) was chosen since it is not likely to be found in real environmental samples in the chosen study area. Also PCNB is detectable by electron capture detector used in this study with its peak well separated from most organochlorine compounds. Solvent and method blanks with IS were run in sequence to check for contamination, peak identification and quantification. These were determined from the linear regression equation (shown below) obtained from the calibration curve of each pesticide standard according to Miller and Miller (1998).

$$\text{Detection limit} = Y_b + 3S_b \quad (2)$$

where Y_b is the value of the blank; S_b is value of the standard error of the regression equation

Due to unavailability of certified reference materials (CRM), spiking method was applied in the recovery test to validate the analytical methods used.

About 1 L double distilled water obtained directly from the glass distiller was spiked with a mixture of DDD, DDE and DDDT at concentration range, 0.1–100 μg and then extracted with dichloromethane (3 × 10 mL) using a separating funnel. Thereafter, the extracts were dried with anhydrous sodium sulphate and concentrated to a smaller volume, 2 mL, using rotary evaporator. Blanks, i.e., double distilled water unspiked with DDD, DDE and DDT mixture was also prepared and treated as described earlier.

One gram C_{18} cartridge (Supelco) normal phase was activated by treating the sorbent with a “matrix-like” solvent and for water samples about 10 mL methanol was allowed to flow through the cartridge under self pressure. Double distilled water (10 mL) was then passed through the cartridge. Thereafter about 1 L of double distilled water spiked with a mixture of DDD, DDE and DDT at fortification range of 0.1–100 μg was passed through the cartridge at a flow rate of 10–15 mL/min. The cartridge was rinsed with some double distilled water and then air dry for about 20 min. The retained DDD, DDE and DDT were then eluted with 3 × 10 mL dichloromethane. The extracts were collected, evaporated and concentrated to a smaller volume, 5 mL.

About 10–15 g of activated charcoal (50–100 mesh) was packed in a glass column. To activate the activated charcoal, 5 mL methanol and 10 mL double distilled water were passed through the column at a flow rate of 15–20 ml/min. Thereafter 1 L of double distilled water spiked with a mixture of DDD, DDE and DDT at concentration range of 0.1–100 μg was passed through the column. The column was rinsed with double distilled water. The DDD, DDE and DDT mixture retained in the activated charcoal in the column was eluted with dichloromethane. The eluted fractions were then collected and reduced to smaller volume, 5 mL.

About 10 g dried, sieved and pre-extracted sediment sample was spiked with a mixture containing DDD, DDE and DDT within the concentration range 0.1–100 μg and placed in a Whatman extraction thimble. This was extracted overnight with 150 mL dichloromethane. The extract was allowed to cool, filtered and reduced to a smaller volume, 5 mL, for column chromatography clean up process.

The extracts collected from the above extraction methods were subjected to a clean up process. A slurry solution of silica gel (1%–2% v/m) prepared in hexane was packed in a glass column. About 0.5–1 cm^3 of anhydrous sodium sulphate was placed at the top of the column to remove any water that may be present in the extracted samples. Before use the column was conditioned by passing 20 mL of hexane through the column. Thereafter, the reduced extracts from liquid–liquid extraction (LLE), solid phase extraction (SPE), activated charcoal (ACE) and Soxhlet extractions described above were introduced into separate columns slowly in order not to disturb the packed column. The introduced extracts were eluted with dichloromethane (50 mL) at a flow rate of 40–50 mL/min. The extracts were collected, and concentrated to smaller volumes. 2 μg of the internal standard, PCNB, was added to the concentrated extracts and re-constituted to 2 mL with the extracting solvent for analysis.

One liter water samples collected from Jukskei River were first prefiltered using 0.45 μm PTFE fibre glass filters (Millipore, Bedford, MA, USA) were placed in separating funnels and extracted with dichloromethane (3 × 20 mL). The extracts obtained were reduced to smaller volumes and passed through a silica gel column as described earlier. After column chromatography, the extracts were concentrated and reconstituted to 2 mL after the addition of 2 μg PCNB as the internal standard and then analysed.

About 1 L of water samples collected from the above mentioned river were prefiltered as described above and then passed through C_{18} cartridge after conditioning as outlined above, at a flow rate of 15–20 mL/min. The other steps as described earlier were then repeated.

About 1 L of water samples prefiltered as described earlier were passed through a column packed with 10–15 g of activated carbon at a flow rate of 15–20 mL/min, after activation with 5 mL and 10 mL methanol and double distilled water, respectively. The column was rinsed with double distilled water. Thereafter the contents of the column were eluted with dichloromethane. The extracts collected were subjected to column chromatography and then reduced to 2 mL after the addition of internal standard.

10 g of dried and sieved sediment samples were soxhlet extracted using dichloromethane overnight. The extracts were filtered and subjected to column chromatography for clean up. The extracts collected were treated as described earlier, i.e., concentration of extracts followed addition of internal standard.

Results and Discussion

The retention times and detection limits achieved are shown in Table 2. The detection limits were 0.01–0.55 μg/L and 0.10–1.95 μg/kg for water and sediment samples, respectively and these compared well with that in the literature (DWAF 1992).

Figure 2 shows the recoveries of the DDT and its metabolites standards from spiked water and sediment samples using different extraction methods. As can be seen from Fig. 2, the percentage recoveries are fairly high for all the extraction methods employed in this study. The high percentage recoveries obtained validated the methods of extraction employed in this study. However, it can be observed from Fig. 2, that the percentage recovery obtained using solid-phase extraction method appeared to be the lowest value of recoveries for both 2,4 and 4,4′DDD, DDE and DDT standard samples. This may be attributed to the dissolution of the standards into the plastic cartridge used for the extraction. The percentage recoveries exhibited by the activated carbon extraction method measured very well with liquid–liquid and soxhlet extractions. This is

Table 2 Retention times and detection limits of, DDD, DDE and DDT standards

Compound	Retention time (min)	Detection limit (μg/L)
2,4′-DDD	10.370	0.52 (0.96)
4,4′-DDD	12.369	0.52 (0.57)
2,4′-DDE	6.432	0.55 (1.95)
4,4′-DDE	9.534	0.55 (1.26)
2,4′-DDT	12.844	0.40 (0.85)
4,4′-DDT	14.301	0.01 (0.10)

Values in parenthesis = sediment samples (μg/g)

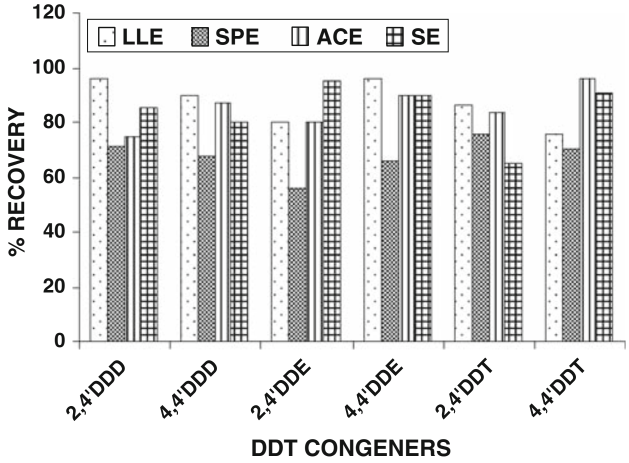

Fig. 2 Percentage recoveries of DDT and metabolites with different extraction methods

an indication of the effectiveness of the activated carbon method employed in this study.

Table 3 shows the mean concentrations of total DDT and metabolites detected in the various surface water and sediment samples collected from different points along Jukskei River. It can be seen from Table 3, that DDD, DDE and DDT were detected in the water and sediment samples collected in March and July for all the sampling points. For the October sampling month, DDE and DDT were detected using liquid–liquid extraction. This pattern was not repeated with the use of other extraction methods. For SPE and SE none of the analytes were detected in any sampling points. However DDD, DDE and DDT were detected using ACE in the middle and down stream sampling points.

It can also been observed from Table 3, that the values for the middle stream were consistently higher than the other sampling points for all the months. Sediment samples showed higher total DDT (nd-1.92 µg/L than the water samples. This is not surprising since it is a well-known phenomenon that sediments act as sinks to a wide range of pollutants. The concentrations obtained in the present study are higher than that reported by Fatoki and Awofolu (2003) in water bodies from the Eastern Cape, but lower than the concentrations in sediment samples reported by Naude et al. (1998). The values obtained in the present study are more within the same range as reported by London et al. (2000) in water samples from the rural parts of Western Cape.

The total DDT concentration values observed in Table 4 for the middle stream under liquid–liquid and activated charcoal extraction methods are marginally higher than the WHO guideline value of 2 µg/L in drinking water. Considering the fact that some local inhabitants make use of the river for different domestic purposes, there is cause for concern.

That the metabolites of DDT were not detected during the month (with the exception of LLE and ACE extraction methods) of October may be due to seasonal variation in the concentration levels. October period is the early days of the southern summer, and therefore there is a lot of rain during this period. It is possible that the dilution effect may

Table 3 Mean concentrations of DDT and metabolites in water (µg/L) and sediment (µg/kg) from different sampling points in Jukskei River, South Africa (n = 3)

Extraction	March			July			October		
Sampling point	DDD	DDE	DDT	DDD	DDE	DDT	DDD	DDE	DDT
LLE									
MS	0.65	1.10	0.85	0.90	1.26	1.04	nd	0.55	nd
DS	0.51	0.80	0.69	0.30	0.65	0.55	nd	nd	nd
US	0.21	0.20	0.23	0.30	0.13	0.22	0.15	0.11	0.15
SPE									
MS	0.50	0.59	0.51	0.50	1.0	0.80	nd	nd	nd
DS	nd	0.71	0.49	0.54	0.89	0.57	nd	nd	nd
US	0.10	0.25	0.15	0.20	0.16	0.26	nd	nd	nd
ACE									
MS	0.52	0.93	0.75	0.79	1.21	0.80	0.50	0.75	0.65
DS	0.52	0.82	0.56	0.48	1.0	0.62	nd	nd	nd
US	0.18	0.21	0.11	0.15	0.14	0.17	0.10	0.06	0.14
SE									
MS	1.0	1.58	1.02	1.53	1.92	1.75	nd	nd	nd
DS	0.42	0.72	0.66	0.26	0.62	0.32	nd	nd	nd
US	0.11	0.31	0.13	0.18	0.14	0.12	nd	nd	nd

ws water sample, *ss* sediment sample, *nd* not detectable, *MD* middle stream, *DS* down stream, *US* upper stream, *LLE* liquid–liquid extraction, *SPE* solid phase extraction, *ACE* activated charcoal extraction, *SE* soxhlet extraction

Table 4 Total DDT (DDT, DDE and DDD) concentrations in water (μg/L) and sediment (μg/kg) from different sampling points in Jukskei River, South Africa (n = 3)

Sampling point	Extraction method	Collection month		
		March	July	October
MS (ws)	LLE	2.6	3.2	0.6
DS (ws)	LLE	2.0	1.5	nd
US (ws)	LLE	0.6	0.7	0.3
MS (ws)	SPE	1.6	2.3	nd
DS (ws)	SPE	1.2	2.0	nd
US (ws)	SPE	0.5	0.6	nd
MS (ws)	ACE	2.2	2.8	1.9
DS (ws)	ACE	1.8	2.1	nd
US (ws)	ACE	0.6	0.5	0.3
MS (ss)	SE	3.6	5.2	nd
DS (ss)	SE	1.8	1.2	nd
US (ss)	SE	0.6	0.4	nd

ws water sample, *ss* sediment sample, *ND* not detectable, *MD* middle stream, *DS* down stream, *US* upper stream, *LLE* liquid–liquid extraction, *SPE* solid phase extraction, *ACE* activated charcoal extraction, *SE* soxhlet extraction

have played a role in the observed values. Also that lower concentrations of the metabolites were obtained in the present study could be an indication of a recent contamination of the river by DDT. High concentrations of DDE and DDD would have suggested a more prolonged contamination resulting in the break down of DDT into its metabolites.

This study has shown the presence of DDT and metabolites in the river studied. The levels have been found to be marginally higher than the WHO guidelines. Possible sources would include: sewage and waste dump sites around the river. The activated carbon extraction technique used in this study compared favourably well to the other extraction techniques. Considering the fact that activated carbon often derived from coconut shell, coal or wood, which has been activated by heating at high temperatures are readily available and fairly cheap to produce, this can be employed in monitoring programme involving DDT and its metabolites in surface water samples. However, work still needs to be done to use activated carbon extraction method used in this study to extract other persistent organic pesticides from water sample. After all, little is known about the levels of other persistent organic contaminants in the chosen study area.

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References

Atuma SS, Hansson L, Johnsson H, Slorach S, de Wit CA, Linstrom G (1998) Organochlorine pesticides, polychlorinated biphenyls and dioxins in human milk from Swedish mothers. Food Addit Contam 15(2):142–150

Bouwman H, Coopan RM, Reinecke AJ, Becker PJ (1990) Levels of DDT application for malaria control. WHO Bulletin OMS 68

Burke M (2006) Water treatment using activated carbon. Chemistry World, April, pp 51–56

Colborn T (1991) Epidemiology of Great Lakes bald eagles. J Toxicol Environ Health 33:395–453

DWAF (1992) Analytical methods manual, TR 151. Department of Water Affairs and Forestry, Pretoria

Fatoki OS, Awofolu T (2003) Methods for the determination of persistent organochlorine pesticide residue in water and sediments by capillary gas chromatography and electron-capture detection. J Chromatogr A 983:225–236

Hassett AJ, Viljoen PT, Liebenberg JJE (1987) An assessment of chlorinated pesticides in the major surface water resources of the Orange Free State during the period September 1984 to September 1985. Water SA 13(3):133–136

Heemken OP, Theobald N, Wenclawiak BW (1997) Comparison of ASE and SFE with soxhlet, sonification and MethANOLIC saponification extractions for the determination of organic micropollutants in marine particulates matter. Anal Chem 69:2171–2180

Hernandez F, Morell I, Bettran J, Lopez FJ (1993) Solid-phase extraction of pesticide residues from groundwater: comparison between extraction carridges and extraction discs. Anal Chim Acta 283:297–303

Iwata H, Tanabe S, Sakai N, Ishmura A (1994) Geographical distribution of persistent organochlorines in air, water and sediments from Asia and Oceania and their implication for global distribution from lower latitudes. Environ Pollut 85:15–33

Kanja L, Skare JU, Nafstad I, Maitai CK, Lokken P (1986) Organochlorine pesticides in human milk from different areas of Kenya. J Toxicol Environ Health 19:449–464

London I, Dalvie MA, Cairan-Cross E, Solomon A (2000) The quality of surface and grounjdwater in the rural Western Cape with regard to pesticides. Water Research Commission Report No. 795/1/00

Meintjies E, van der Merwe L, du Preez JL (2000) Qualitative and quantitative evaluation of estrogen and estrogen-mimicking substances in the water environment. Water Research Commission Report No. 742/1/00

Miller JC, Miller JN (1998) Statistics for analytical chemistry, 2nd edn. Ellis Horword, Chichester

Molins C, Hogendoorn EA, Heusinkweld HAG, Van Zoonen P, Baumann RA (1997) Microwave assisted solvent extraction (MASE) of organochlorine pesticides from soil samples. Int J Environ Anal Chem 68:155–169

Mutshatshi TN, Okonkwo JO, Botha B, Agyei N (2008) Organochlorine residues in maternal milk from inhabitants of the Thohoyandou area, South Africa. J Toxicol Environ Chem 90(4):695–706

Naude Y, De Beer WHJ, Jooste S, Van der Merwel, Van Rensburg SJ (1998) Comparison of supercritical fluid extraction and soxhlet extraction for the determination of DDT, DDD and DDE in sediments. Water SA 24(3):205–214

Okonkwo JO, Kampira L (2002) Organochlorine pesticides in mother's milk in Swaziland, 1996–1997. Bull Environ Contam Toxicol 68(5):740–746

Okonkwo JO, Kampira L, Chingakule DDK (1999) Organochlorine insecticides residues in human milk: A study of lactating mothers in Siphofaneni, Swaziland. Bull Environ Contam Toxicol 63:243–247

Okonkwo JO, Sibali LL, McCrindle R, Senwo ZN (2007) An improved method to quantify dichlorodiphenyltrichloroethane (DDT) in surface water using activated carbon. Environ Chem Lett 5:121–123

Okonkwo JO, Mutshatshi TN, Botha B, Agyei N (2008) DDT, DDE and DDD in human milk from South Africa. Bull Environ Contam Toxicol 81:348–354

Sibali LL, Okonkwo JO, McCrindle R (2008) Determination of selected organochlorines pesticides (OCP) compounds from the Jukskei River catchment area in Gauteng South Africa. Water SA 34(5):611–621

Takahashi W, Sadin D, Takei G, Wong L (1981) Organochlorine pesticides residues in human milk in Hawaii, 1979–1980. Bull Environ Contam Toxicol 27:506–511