Bull. Chem. Soc. Ethiop. **2022**, 36(2), 329-337.
DOI: https://dx.doi.org/10.4314/bcse.v36i2.7

ISSN 1011-3924
Printed in Ethiopia
Online ISSN 1726-801X

OBSOLETE PESTICIDE RESIDUE LEVEL ANALYSIS AND TOXICOLOGICAL RISK EVALUATION FROM HADYA ZONE DUMP SITE, ETHIOPIA

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(Received December 15, 2021; Revised April 22, 2022; Accepted April 28, 2022)

ABSTRACT. Hadya Zone (HZ) obsolete pesticide dump site was selected for determinations of OCPs from soil and assessing the toxicological risks to environment and human health. Composite soil samples were collected and extracted using Soxhlet (SOX) extraction method and analyzed for p,p'-DDT, p,p'-DDE, p,p'-DDD, α-BHC, β-BHC, γ-BHC, δ-BHC, γ-chlordane, α-chlordane, and endosulfan sulfate using GC-MS. The total average concentrations of ΣDDTs, ΣBHCs, chlordanes, endosulfans were 41.92, 5.91, 11.47, 0.44 ng/g, respectively. DDTs, chlordanes and BHCs were categorized as causing potential risks to the eco-environment exceeding the threshold value (2.5 ng/g) set by Netherlands' soil quality standards. However, endosulfans residual concentrations were below the threshold value. Carcinogenic risk assessments to humans were evaluated using incremental lifetime cancer risks (ILCRs) via three exposure routes (ingestion, dermal contact; inhalation). The average ILCRs values were 2.54×10^{-8}, 1.68×10^{-8}, and 2.71×10^{-8} for the three age groups: childhood, adolescence, and adulthood, respectively. The result shows the cancer risk to humans from the soil matrix in the study site was acceptable. However, since the dumping site is at high elevation, there is high probability to be transported via runoff to different environmental matrices: river water, sediment and biota. Thus, if the residue analyses of all these environmental matrices are considered, the risk might be increasing.

KEY WORDS: Obsolete pesticide, Dump site, Toxicological risk assessment, OCPs

INTRODUCTION

Pesticides have become an integral part of modern agricultural production and health sector for increasing agricultural yield and controlling vectors, respectively. However, exposure to these chemicals grows into public health concern, particularly in countries where environmental safety regulations are not strictly implemented and the knowledge of safe handling procedures is inadequate [1, 2]. Another problem related with pesticides is that most pesticides expire two years after production and become obsolete. Obsolete pesticides are defined as old, buried, banned or no longer identifiable and sometimes converted to other chemical components which are even more toxic than the original pesticides [3-6].

Globally, a significant quantity of obsolete pesticides (half a million tons) is in storage [7]. Africa has accumulated an estimated amount of 50,000 tons of obsolete pesticide stocks with countries such as Ethiopia, Botswana, Mali, Morocco, South Africa and Tanzania each estimated to have more than 1,000 tons [3, 8]. The inventories carried out in Ethiopia showed the presence of over 400 stores with 1,500 tons of obsolete pesticides and 1,000 tons of contaminated equipment [9]. The obsolete pesticides in developing countries including Ethiopia [5] have been there in various stores for decades in open or inappropriate stores causing considerable negative impacts on public health and the environment. In addition to poor storage, there exists no disposal facilities and legal policy for safe disposal of these banned chemicals resulting in dumps of obsolete pesticides in landfills and open storage sites [10].

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The toxicological effects of organochlorine pesticides (OCPs) range from the acute illnesses to chronic poisonings such as neurological damage, birth defects, endocrine dysfunction and potential carcinogenicity [10-14]. They also exhibit high lipophilic properties and hence accumulated in lipid tissues of organisms leading to biomagnification at higher trophic levels in the food chain.

Soil pollution by persistent obsolete pesticides becomes a major global threat affecting several of the United Nations Sustainable Development Goals (UNSDGs) associated with the soil environment. This raise concerns not only on soil functions, soil biodiversity and food safety but also on the off-site transport of contaminants via wind and water-driven erosion. Such off-site transport may impair the functioning of ecosystems and represent additional exposure routes to soil contaminants for humans and other non-target organisms [15]. Hence, there is a dire need to monitoring and surveillance of these pollutants in soil matrix particularly in Africa.

In Ethiopia, only few studies pertaining the occurrence of OCPs in soils, chewable parts of khat plant, wheat, milk, lakes, fish, and birds from different parts of the country [1, 2, 9, 12, 16-20] have been published. However, the residue level analyses from obsolete pesticide dump sites and associated toxicological risk assessment studies are scant. Therefore, the purpose of this study is to evaluate the level of obsolete pesticide residues at Hadya dump site and also assess the potential ecological and health risks due to exposure to obsolete OCPs. This study serves as a valuable source of toxicological information to regulatory bodies and various stakeholders working in public health and environmental issues.

EXPERIMENTAL

Description of sampling site

Field visit was made in 2016 to observe the status of obsolete pesticide dump site in Hadya Zone, Hosaina town, located in the Southern Nations, Nationalities, and People's Regional State of Ethiopia, having a latitude and longitude of 7°33′N 37°51′E.

Figure 1 shows a map and a photograph of the buried obsolete pesticides located within the compound of the Hadya zone agricultural development department (the point shown on the map of Hosaina town). The department is located at the central part of the town at an elevation of 2321 m (Figure 2) and thus the dumping site is in the residential area. As it can be seen in Figure 2, on the left side of the dumping site there is a “Batena” river at lower elevation of 2272 m at the nearest distance of 1.8 km away from the dump site. Therefore, during the rainy season, runoff from this site might contaminate the “Batena” river located at the close proximity of the town.

Sample collection and preparation

Soil samples were collected in October 2016 from the dumping site of HZ agricultural development department compound following soil sampling methodology described by Hussen *et al.* [1] with slight modifications. A composite soil sample of 10 cores was randomly taken from 10 cm depth using a spade. Slices of 5 cm thickness were taken along the vertical wall of the holes. All increments collected were pooled on a plastic sheet with an area of about 2 m^2 and mixed manually. For further homogenization, the soil sample was divided into six portions over the plastic sheet and a small amount was taken from each portion to make a sub-sample of approximately 500 g. This was kept in a polyethylene plastic bag, after being wrapped in aluminium foil, and then transported to the laboratory in a chilled insulating box. The soil sample was air dried, ground in a mortar with a pestle, sieved through a 2 mm pore sieve and stored in a refrigerator at 4 ºC until the time of use.

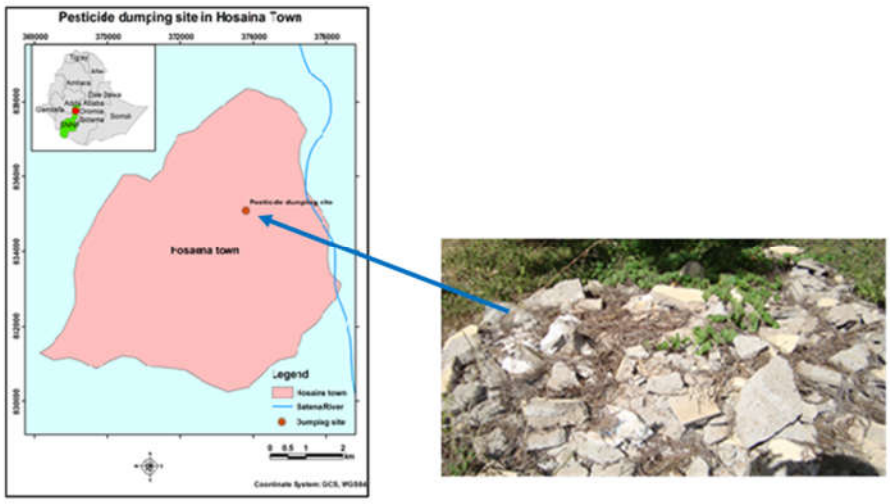

Figure 1. Map showing the location of the obsolete pesticides dumping site in the study area.

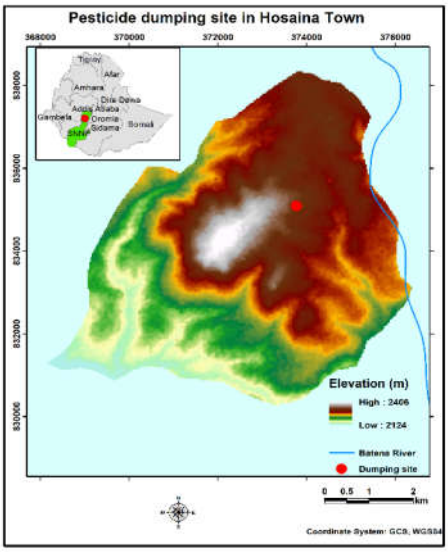

Figure 2. Elevation map of the study area.

Chemicals and materials

Acetone (Laboratory reagent grade, GLC assay 99 %), diethyl ether (Laboratory reagent grade, ASC, ISO, Spain), *n*-hexane (HPLC grade, Riedel-de Haën, Germany), activated alumina absorbent and sodium sulfate anhydrous (assay after drying 99%) were used. Ammonium chloride (Lab Tech Chemical) was used for treatment of soil sample. Acetone/*n*-hexane (1:1, v/v) was used

to extract all OCPs from the soil matrix. OCPs reference standard solutions (α-BHC, β-BHC, γ-BHC, δ-BHC, endosulfan-I, endosulfan-II, endosulfan sulfate, *p,p'*-DDE, *p,p'*-DDT, *p,p'*-DDD, α-chlordane and γ-chlordane) were obtained from Ethiopian Ministry of Agriculture and Rural Development.

Extraction and cleanup of OCPs from the soil sample

Soxhlet extraction of soil sample was carried out following the protocol described in Hussen *et al.* [16] with slight modifications. To avoid any cross contamination, all Soxhlet (SOX) glassware were baked at 450 °C for 12 h in a furnace and further refluxed with 250 mL of extracting solvent for 17 h prior to use. Triplicate soil samples (4 g each) were taken from the composite soil sample (500 g) mixed with equivalent weights of sodium sulfate were ground using mortar and a pestle, and quantitatively transferred into extraction thimbles. The samples were continuously refluxed with fresh solvent for 320 cycles (for 16 h at rate of 20 cycles/h) [1, 16] and the extracts were evaporated to 10 mL using a rotary evaporator (ROTAVAC valve Heidolph, Germany). Clean-up protocol was carried out using the glass columns (20 mm i.d. and 32 cm length) packed with 10 g of activated alumina (activated overnight at 200 ºC) and topped with 2 g of sodium sulfate. Pre-washing the columns with 40 mL of acetone/n-hexane (1:1, v/v), the extracts were eluted using 60 mL of extracting solvent. Eluates were finally evaporated to volumes of about 1.5 mL using the rotary evaporator.

Residue level analysis of OCPs with GC-MS

An Agilent GC/MSD (6890 GC; 5975N Mass Selective Detector, Agilent Technologies Inc., USA) equipped with an Agilent HP-5ms column (30 m x 0.25 mm x 0.25 µm, 5% phenyl methyl siloxane) was used for the analysis. The injection mode was fully automated and carrier gas was helium with a flow rate of 1.0 mL/min. The initial oven temperature was 120 °C for 0.50 min which was ramped to 230 °C at a rate of 7 °C/min and ramped to 240 °C at a rate of 2 °C/min and then ramped to 285 °C at a rate of 10 °C/min and held for 8 min. The temperature of the quadrupole mass filter was 150 °C. The ionization mode was electron impact carried out at 70 eV. The temperature for the ion source and the transfer line was 230 °C and 280 °C, respectively. The MSD was operated in SIM mode of m/z 60 to 500. Quality assurance was maintained by method blanks, calibration curves ($R^2 > 0.99$) and retention times of the corresponding standard reference solutions.

Risk assessments

Carcinogenic risk evaluation of OCPs was carried out using incremental lifetime cancer risk (ILCR) approach. ILCR denotes the incremental probability that an individual will develop cancer during his lifetime due to exposure to a potential chemical carcinogen [21]. Here, the ILCR of population groups at HZ dump site influenced by exposures to obsolete OCPs contaminants from the soil were evaluated. The exposure to a probable carcinogen compound was classified according to three major pathways: ingestion, skin contact, and inhalation for the three age groups: children, adolescent, and adults. The age groups are classified as Children (0-10 years); adolescents (11-18 years); adults (19-70 years) [22]. Thus, the ILCR of the three exposure pathways within the scope of the study was calculated using the following equations [21, 22]:

$$ILCRs_{ingestion} = \frac{C_{soil} \times \left(CSF_{ingestion} \times \sqrt[3]{BW/70}\right) \times IR_{soil} \times EF \times ED}{BW \times AT \times CF}$$

$$ILCRs_{dermal} = \frac{C_{soil} \times \left(CSF_{dermal} \times \sqrt[3]{BW/70}\right) \times SA \times FE \times AF \times ABS \times EF \times ED}{BW \times AT \times CF}$$

$$ILCRs_{inhalation} = \frac{C_{soil} \times \left(CSF_{inhalation} \times \sqrt[3]{BW/70}\right) \times IR_{air} \times EF \times ED}{BW \times AT \times PET}$$

where, $ILCRs_{ingestion}$, $ILCRs_{dermal}$ and $ILCRs_{inhalation}$ are the incremental cancer risks via ingestion, dermal contact and inhalation of carcinogenic compounds from the contaminated soil, respectively. C_{soil} refers to the contaminant concentration in soil (mg/kg). The details of other parameters/exposure factors used to calculate the increasing possibility of humans to get cancer during their lifespan via exposure to a carcinogenic compound are listed in Table 1; whereas the carcinogenic slope factors (CSFs) of OCPs obtained from the integrated risk information system (IRIS) are listed in Table 2. The total cancer risks in different age groups were estimated as the sum of individual risks for the three exposure pathways.

Table 1. Values of the parameters used for the estimation of the incremental lifetime cancer risk (adopted from [21]).

Exposure parameters	Unit	Childhood	Adolescence	Adulthood
Body weight (BW)	kg	13.95	46.75	58.75
Ingestion rate (IRsoil)	kg	13.95	46.75	58.75
Exposure frequency (EF)	d/yr	350	350	350
Exposure duration (ED)	yr	6	14	30
Average life span (AT)	d	LT × 365	LT × 365	LT × 365
Lifetime (LT)	yr	72	72	72
Surface area (SA)	cm^2/d	2800	2800	5700
Dermal exposure ratio (FE)	Unitless	0.61	0.61	0.61
Dermal surface factor (AF)	mg/cm	0.2	0.2	0.07
Dermal absorption factor (ABS)	Unitless	0.13	0.13	0.13
Inhalation rate (IRair)	m^3/d	10.9	17.7	17.5
Particle emission factor (PET)	m^3/kg	1.36×10^9	1.36×10^9	1.36×10^9

Table 2. The parameters used in calculating the incremental cancer risk (ILCR) for humans exposed to environmental pollutants [21, 22].

OCPs	$CSF_{injestion}$	CSF_{dermal}	$CSF_{inhalation}$
p,p'-DDE	3.40×10^{-1}	4.86×10^{-1}	NA
p,p'-DDD	2.40×10^{-1}	3.43×10^{-1}	NA
p,p'-DDT	3.40×10^{-1}	4.86×10^{-1}	3.40×10^{-1}
gChlordane	3.50×10^{-1}	7.00×10^{-1}	3.50×10^{-1}
HCB	1.60	3.20	NA
Endosulfan	NA	NA	NA

NA = values not available.

RESULTS AND DISCUSSION

Residual levels of OCPs

The OCPs residue levels found in the soil samples collected from HZ obsolete pesticides dump site is presented in Table 3. The concentrations obtained were compared with some other studies from similar dump sites and their potential risk evaluations were also discussed. The concentrations of p,p'-DDT (14.94 ng/g dw) and its isomers p,p'-DDE (24.09 ng/g dw) and p,p'-DDD (2.89 ng/g dw) were all detected in HZ dump site soil sample. The total concentrations of

DDTs were 41.92 ng/g. As compared to other similar dump sites (Hochiminh, Vietnam (23 ng/g) and Chennai, India (24 ng/g)), the concentration from the present study was almost twice the total residue levels of DDTs reported elsewhere [23]. On the other hand, it was found to be much lower than the concentrations reported (350 ng/g) at Phnom Penh dump site, Cambodia [23] and at pesticide old storage sites of east African surface soils (12-282 mg/g) reported in Tanzania [24]. In another study [25] at Lahore dump site in Pakistan, a wide range of concentration (from not detected – 820 ng/g) had been reported.

When considering the DDT profiles, the ratio of (p,p'-DDE + p,p'-DDD)/p,p'-DDT can be used as an indicator of the residence time of p,p'-DDT in the environment as its level decrease with time and major metabolites being p,p'-DDE and p,p'-DDD. A ratio > 1.0 indicates that the application was done in the past and a value < 1 indicates a relatively recent application [26]. From HZ dump site soil sample, a ratio of 1.81 was obtained indicating a long time ago dumping of OCPs in the study area.

Endosulfan sulfate is the major metabolite of endosulfan-I and Endosulfan-II. From Table 3, the concentrations of both types of endosulfans (I and II) in the investigated soil samples were not detected. On the other hand, endosulfan sulfate was detected (0.44 ng/g). This implies that almost all endosulfan-I and endosulfan-II have been converted to their major metabolite endosulfan sulfate indicating the obsolete pesticides in this site were dumped long time ago. Chlordane commonly exists in two isomeric forms: γ-chlordane and α-chlordane and both were detected at concentration of 6.15 and 5.32 ng/g, respectively.

Potential ecological risk assessment

The ecological risk posed by OCPs to the soil environment was evaluated by comparing the residual concentrations of OCPs with related soil quality standard guidelines. On the basis of the soil environmental quality standard of Netherlands published [21, 27], the concentrations of DDTs, chlordane and BHC were greater than the target value for unpolluted soil (2.5 ng/g) causing the ecological risk to the eco-environment and non-target organisms. Besides, for the protection of plants and invertebrates, small birds and mammals [28], the maximum permissible concentrations of DDTs in soil are 10, 11, and 190 ng/g, respectively. In this study, the residual concentrations of DDTs and chlordane from HZ dump site soil particles exceeded 11 ng/g, with the potential to cause ecological harm to plants, invertebrates and small birds.

Human health risk assessment

The incremental lifetime cancer risk (ILCR) technique was employed to estimate the human health risk from interaction with OCPs. This technique highlights that there a potential danger for human beings when exposed to OCPs via three major routes, including ingestion, inhalation, and dermal contact. This model also indicates that an ILCR between 1×10^{-6} and 1×10^{-4} indicated probable danger, an ILCR of 1×10^{-6} or less signified immunity, whereas an increased possibility of a threat to human health was expected by ILCR value of more than 1×10^{-4} [21, 22]. In this study, the average of ILCRs values were 2.54×10^{-8}, 1.68×10^{-8}, and 2.71×10^{-8} for children, adolescents, and adults, respectively. Thus, the result shows that the probability of cancer risk from soil particles containing organochlorine pesticides under this study was acceptable. However, from the elevation difference point of view (Figure 2), obsolete OCPs could be distributed from the point source to the nearby water bodies via runoff. Thus, if the residue analyses encompassing all these environmental matrices (water, sediment, biota) are taken into consideration, the risk might go beyond what we are reporting.

Table 3. Concentrations (ng/g) and cancer risk values on children for different exposure pathways for OCPs in soil collected from HZ dump site.

Compound	Average (n = 3) concentration (SD)	Cancer risk exposure pathways			Cancer risk
		Ingestion	Dermal contact	Inhalation	
p,p'-DDT	14.94 (2.48)	3.40×10^{-9}	1.08×10^{-9}	1.36×10^{-13}	4.48×10^{-9}
p,p'-DDE	24.09 (1.26)	5.48×10^{-9}	1.74×10^{-9}	-	7.22×10^{-9}
p,p'-DDD	2.89 (0.56)	4.64×10^{-10}	1.47×10^{-10}	-	6.11×10^{-10}
α-BHC	2.46 (0.32)	2.63×10^{-9}	1.17×10^{-9}	-	3.80×10^{-9}
β-BHC	ND	-	-	-	-
γ-BHC	3.16 (0.45)	3.38×10^{-9}	1.50×10^{-9}	-	4.88×10^{-9}
δ-BHC	0.29 (0.04)	3.11×10^{-10}	1.38×10^{-10}	-	4.48×10^{-10}
γ-Chlordane	6.15 (1.10)	1.44×10^{-9}	6.40×10^{-10}	5.77×10^{-15}	2.08×10^{-9}
α-Chlordane	5.32 (0.78)	1.25×10^{-9}	5.53×10^{-10}	4.99×10^{-15}	1.80×10^{-9}
α-Endosulfan	ND	-	-	-	-
β-Endosulfan	ND	-	-	-	-
Endosulfan sulfate[a*]	0.44 (0.01)	-	-	-	-
Total average cancer risk					2.54×10^{-8}

SD: Standard deviation; ND: not detected; a*: the parameter values are not given.

Similarly, the cancer risks and total average cancer risk values calculated for adolescence and adults are summarized in Table 4.

Table 4. Cancer risk potential values due to ingestion, dermal contact, and inhalation of OCPs from the exposure to the soil of Hadya Zone dump site.

Age group	Exposure pathway	Mean
Adolescence	Ingestion	9.56×10^{-9}
	Dermal contact	7.26×10^{-9}
	Inhalation	4.13×10^{-13}
	Average cancer risk	1.68×10^{-8}
Adulthood	Ingestion	1.76×10^{-8}
	Dermal contact	9.52×10^{-9}
	Inhalation	7.51×10^{-13}
	Average cancer risk	2.71×10^{-8}

CONCLUSION

According to the findings of this study, the significant concentrations of especially ΣDDT, Σchlordane and ΣBHC reported in this work are potential threats to the local community and ecosystems in proximity of obsolete pesticide dump site in Hadya Zone. These OCPs can be drained from agricultural areas into the nearby water bodies and reach the non-target organisms. The problem might be intensified with poverty, lack of education and awareness about the toxic effect of pesticide exposure and the scarcity of pure drinking water resources in the studied area. Thus, the probability of cancer risk estimated from soil could go beyond what has been reported in this study. Therefore, due attention should be given by the concerned body to avoid similar accumulation in the future and to exhaustively work on the disposal of obsolete chemicals from the existing dumpsites. Additionally, the ability of DDT and DDE to undergo long-range global transport poses an international concern about the residue detected at any corner of the globe. In fact, OCPs contamination is not only a local/national problem but also international, since they

do not recognize any form of border. Therefore, there should be synergy between the national and international efforts to control the use and disposal of such chemicals.

ACKNOWLEDGEMENTS

Ministry of Agriculture and Rural Development of Ethiopia is highly appreciated for allowing GC-MS for residue level analysis. The authors acknowledge Dr. Gudina Legesse (GIS and RS expert at Centre for Environmental Science) for producing the maps of the study area.

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