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Assessment of Organochlorine and Organophosphorous Pesticides in Groundwater and Agricultural Soils in Mysuru District of Karnataka

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Abstract

Predominance of pesticides use is causing serious and high health risk to mankind through agriculture. This research is carried out to check the persistence of organochlorine and organophosphorous pesticides in ground water and agriculture soil of Heggadadevana kote, Mysore district, India. A total of twenty ground water samples and twenty soil samples were collected from four regions (hoblies). The residues were extracted by using liquid-liquid extraction and solid- liquid extraction. It was analysed using LC-MS and GC-MS.

Introduction

Pesticides have become an integral part of modern agriculture after green revolution. To feed the growing population, the use of pesticides has increased over agriculture crops (Popp et al., 2012). The applied pesticides, reaches the crops and target pests by only 0.1% and the remaining 99% contaminants the soil, water and air (Pimentel and Levitan 1986). Thus, the Pesticides can harm other non target organisms like birds, fish, beneficial insects and non target plants (Aktar et al., 2009). Transport of pesticides from surface soil to ground water, depends on the type of soil like clay soil or sandy soil. The flows of pesticides are maximum when there is a crack or earthworm burrow which provides an easy penetration of pesticides from soil to ground water table (Flury 1996). Accumulation of pesticides in agriculture crops are highly contaminated (Zhang et al., 2011, Anzene et al., 2014, Zhu et al., 2014, Kawano et al., 1992) Bioavailability and bioaccumulation of pesticides depends on the physico-chemical properties of the soil, root-soil inference etc. (Tao et al., 2005). Uptake and translocation of pesticides in plants leads to hazardous to humans and other primary animals, by consuming it (Juraske et al., 2009). Bioaccumulation of pesticides by the aquatic plants increases the toxicity level in the fishes (Li and Macdonald. 2005), which is important food web of terrestrial organisms (Sarkar et al., 2003, Muir 2003). When this is consumed by the raptorial and fish-eating birds, there eggshell reduces in thickness and result in difficult in hatching (Hickey and Anderson 1968, Adams et al., 1949).pesticide exposure is associated with chronic health problems or health symptoms like memory loss, respiratory, cancer, neurologic deficits (McCauley et al. 2006, Corsini et al. 2008, Keifer and Firestone 2007). There are reports on occurrence of pesticides contamination in groundwater, surface water and soil (Shomar et al., 2005, Kumari et al 2008, Kaushik et al.,2008, Domagalski 1992, Mawussi et al,. 2014, Hu et al., 2014, Mahdavian and Somashekar., 2013, Zhang et al., 2011, Adeyemi et al., 2011, Famiglini et al., 2008, Ahad et al., 2000, Zhang et al 2006, Singh 2001, Sankararamakrishna et al.,2005, Gonalves et al 2007, Shukla at al., 2006). The objective of the project work is to check the level of pesticide residues in agricultural soil and ground water samples of Heggadadevana kote taluk, Mysuru, India. As the site is an agricultural prone area.

Material and methods

Study area

The study area, Heggadadevana kote which lies between 12.088°N 76.328°E at the elevation of 694m with an area of 1,94,138 hector in which 63,970 hector is cultivated area. Agriculture is mainly rain-fed and some are river-fed. Red loam, laterite, sandy loam, red clay soil can be seen and paddy, ragi, ginger, cotton, pulses and other commercial vegetables are the main crops (http://www.mysore.nic.in/geography.htm).

Sampling

Samples were collected from the area which was divided into four region (hoblies) hampapura, antharasanthe, kasaba and kandalike. Soil sample were collected from 12-15cm surface and it was stored in the polythene bag. Soil sample were then air dried and sieved in 2 mm mesh and preserved in the zip lock polythene bag until further analysis.

The water samples were collected from the hand pump or electric pump. The starting 2-3 minutes water were left off and then the bottle was rinsed with same water and collected. These bottles were prior washed with potassium dichromate and rinsed with distilled water.

Chemicals:

The solvents used for the extraction were obtained from Merck India Pvt. Ltd. (AR and HPLC grade). Organochlorine pesticides mix standards (α,β,γ,δ – HCH, Heptachlor, Aldrin, Heptachlor epoxide, Endosulfan I, 4,4'-DDE, Dieldrin, Endrin, 4,4'-DDD, Endosulfan II, 4,4'DDT, Endrin aldehyde, endosulfan sulphate and methoxychlor) and Organophosphates pesticide standard (Dichlorvos, Mocap, Disulfoton, Methyl parathion, Ronnel, Dursban, Tokuthion, Guthion) were procured from, Sigma-Aldrich Co.USA.

Extraction clean up

In Soil

Pesticides were extracted from the soils according to the following method. Each sample was weighed (10 g) into Erlenmayer flask with 50 ml of petroleum ether and acetone solvent (1:1) in a mechanical shaker for one hour. The mixture was filtered by using filter paper (solid-liquid extraction). The filtrate was collected in a beaker and was directly evaporated to dryness.

In water

A known amount of water (25 ml) was taken in a separating funnel followed by 25 ml of petroleum ether solvent. The mixture was hand shaked, the water is left out and the solvent was collected in a beaker. Then, to the beaker, a gram of anhydrous sodium sulphate is added to absorb the moisture content. The organic solvent is collected in another beaker and was evaporated to dryness.

The dry residues obtained in beaker of both soil and water samples were redissolved in 1ml of acetonitrile (HPLC grade) and the residue was collected in an eppendorf tube and stored at -10°C for the further analysis.

Thin layer chromatography

A cut piece of silica coated aluminium sheet was taken. Extracted sample was spotted on the base of the sheet by using capillary tube and was dried. In the glass chamber containing mobile phase solvent of (8:1:1) ratio of n-hexane: acetone: ethyl acetate. The sheet was dipped into the TLC glass chamber and allowed the solvent to reach 95%. Then the sheet was removed and air dried components were detected under UV light and later dipped in KM_nO_4 solution.

Liquid Chromatography-Mass Spectroscopy (LC-MS) Analysis

Chlorpyrifos and its residues were analyzed by Liquid Chromatography-Mass Spectroscopy (LC-MS). (Acquity Waters, USA). The LC-MS was equipped with a BEHC 181.7 µm column (10 x 50 mm) with auto injector. The cartridges were conditioned with acetonitrile and washed with deionized water containing 0.1 % formic acid. Mass spectroscopy (MS) was performed using a Synapt G2 HPMS MS (Waters, USA) equipped with Electron spray ionization (ESI) detector. The operating condition was Capillary (kV)-3.00, Sampling Cone-40.00, Extraction Cone-4.00, Source Temperature (ºC)-100, Desolvation Temperature (°C)-200, and Desolvation Gas flow (L/Hr)-500.0.

Gas Chromatography-Mass Spectroscopy analysis (GC-MS)

GC-MS analysis was carried out on 7890A GC systems (Agilent Technologies, inc) linked to Mass spectroscopy of Synapt G2 HPMS MS (Waters, USA) equipped with Electron spray ionization (ESI) detector. The GC column consisted with HP-5MS capillary column of 30m x 0.250mm (Agilent Technologies, Inc). The GC-MS was programmed according to Kamei et al. 2011. The oven temperature was programmed to increase from 120ºC to 320ºC at 10ºC min^{-1}. Mass spectra were recorded at 70 ev using full scan mode. The qualitative analysis of endosulfan present in the samples was monitored by comparing retention time with respect to internal standard. Recovery study was carried out to evaluate the efficiency of above said procedure. The average recovery for both endosulfan isomers is found to be 93% in average.

Results and Discussion

Organochlorine Pesticides and their residues in Ground Water:

The concentration of OCPs in the ground water from the Heggadadevana kote is shown in the table. The most abundant pesticides detected were sum of heptachlor *m/z* 371.81 (Chlordene *m/z* 337.86), DDT *m/z* 353.91 (DDE *m/z* 317.94, 6-oxo-2-hydroxy-7-3,8,8,8-tetrachloroocta- 2z,4z-dienotae *m/z* 417.48) , g-HCH *m/z* 289.86 (g-PCCH *m/z* 253.88) , Methoxychlor *m/z* 344.01 (methoxychlor olefin *m/z* 308.04), endosulfan *m/z* 405.81 (endosulfan sulphate *m/z* 421.85) and aldrin *m/z* 363.87. Detection of heptachlor epoxide, an oxidized and dechlorinated metabolite of heptachlor is because, the natural micro biota present in soil, oxidizes heptachlor resulting in formation epoxide and dechlorination to Chlordene (Xiao 2011).

Dichlorodiphenyltrichloroethane (DDT) dechlorinated to DDD and dehydrochlorinated to DDE. This DDE is dechlorinated to DDMU by methanogenic and sulfidogenic microbes.

6-oxo-2-hydroxy-7-3-,8,8,8-tetrachloroocta-2z,4z-dienotae which is degraded by series of dehydroxide and deoxidation by soil microbes. (http://eawag-bbd.ethz.ch/ddt/ddt_map.html)

1, 3(R), 4(S), 5(S), 6(R)-Pentachlorocyclohexene (g-PCCH) , a dehydrochlorinated metabolite of g-HCH is because of microbial or enzymatic degradation, dehydrochlorination results in the formation of g-PCCH (http://eawag-bbd.ethz.ch/ghch/ghch_map.html). Methoxychlor olefin, a dehydrochlorinated metabolite of methoxychlor is because of microbial or enzymatic degradation, dehydrochlorination results in the formation of methoxychlor olefin (Hirai 2004). Endosulfan sulphate, an oxidized metabolite of endosulfan is because, the natural micro biota present in soil, oxidizes endosulfan resulting in formation of endosulfan sulphate (shivaramaiah 2006, http://eawag-bbd.ethz.ch/end/end_map.html).

From the data on levels of OCPs in the study area were compared with the similar results reported in and around India. **Jayashree and Vasudevan** (2007) found γ-HCH (0-11.6µg/l), DDE (0-0.7µg/l), endosulfan (0.1-11.6µg/l) and Endosulfan sulphate (1.1-19.2µg/l) residues in ground water of Thiruvallur district, India. **Adeyemi et al** (2011) analyzed OCPs in water of Lagos Lagoon and found chlordane (0.006-0.950µg/l), heptachlor (0-0.67µg/l), methoxychlor (0-0.123µg/l), endosulfan (0.015-0.996µg/l), DDE (0.005-0.477µg/l) and aldrin (0.080-0.790µg/l). **Mahdavian and Somashekar** (2013) analyzed OCPs in water samples, around Chamarajanagar district, India and found γ-HCH (0.3-8), endosulfan (0-97), and DDE (0-17). **Mawussi et al** (2014) found heptachlor epoxide (0-0.012µg/L), endosulfan (0-0.009µg/L) and endosulfan sulphate (0- 0.116µg/L) in soil (vegetable production) of coastal areas in Togo. **Singh** (2001) found ∑ DDT (202-812 ng/l), ∑ BHC (46-733 ng/l), endosulfan (0-56 ng/l), aldrin (12-104 ng/l), dieldrin (91-471 ng/l) and heptachlor (90-112 ng/l). **Sankararamakrishnan et al** (2005) analysed γ-HCH (0.303-0.471µg/l) and ∑ DDT, endosulfan, aldrin were not detected in the samples. **Kumari et al** (2008) found γ-HCH (0 -1.674 µg/l), ∑ DDT (0.090-0.865 µg/l), ∑ endosulfan (0-0.141 µg/l) and heptachlor, aldrin, chlordane were not detected in the samples. **Shomar et al** (2005) found ∑ DDT (0.3-1 ± 1 µg/l), Aldrin (0.1-6 ±1µg/l µg/l), heptachlor (0.4±1µg/l).

Organochlorine Pesticides and their residues in agriculture soil:

The concentration of OCPs in the agriculture soil from the Heggadadevana kote is shown in the table. The most abundant pesticides detected were sum of g-HCH (g-PCCH), heptachlor (heptachlor epoxide and Chlordene), Methoxychlor (methoxychlor olefin), DDT (DDE, DDMU *m/z* 281.98, 6-oxo-2-hydroxy-7-3,8,8,8-tetrachloroocta- 2z,4z-dienotae and endosulfan.

From the data on levels of OCPs in the study area were compared with the similar results reported in and around India. **Zhao et al** (2013) and found γ-HCH (0.07-0.99 ng/g), heptachlor epoxide (2.56-21.72 ng/g), chlordane (0.09-2.70 ng/g) and endosulfan (0-18.12 ng/g) residues in the mountain soils of Tajikistan. **Mahdavian and Somashekar** (2013) analyzed OCPs in agricultural soil around Chamarajanagar district, India and found γ-HCH (0-37 µg/kg), endosulfan (1-54 µg/kg), and DDE (0-121 µg/kg). **Mawussi et al** (2014) found heptachlor epoxide (0-6.837 µg/kg), DDE (0-2.288 µg/kg), endosulfan (0-0.737 µg/kg) and endosulfan sulphate (0- 0.943). **Rajanna and Belagali** (2014) found γ-HCH (bdl), heptachlor (bdl-0.0020 µg/kg), aldrin (bdl), heptachlor epoxide (bdl) and DDE (bdl-0.0004 µg/kg) in soil samples of Nanjangud region. **Zhang** (2006) analysed the residues of organochlorine pesticides in Hong Kong soils and found ∑ DDT (0.24 ± 0.20), ∑ HCH (6.32 ± 1.52), Endrin (0.02 ± 0.02) and α-endosulfan (0.0047). **Tao** (2005) analysed OCPs in agricultural soil from Tianjin, China and found ∑ DDT (157.55 ng/g) and ∑ HCH (52.8 ng/g).

Organophosphorous pesticides and their residues in ground water

The concentration of OPPs in the ground water from the Heggadadevana kote is shown in the table. The most abundant pesticides detected were sum of Azinphos methyl *m/z* 317.01 (Azinphos methyl oxon *m/z* 301.03), Prothiofos *m/z* 343.96 (Prothiofos oxon *m/z* 327.99) and chlorpyrifos *m/z* 350.92.

Detection of Azinphos methyl oxon, an oxidized and metabolite of Azinphos methyl is because, the natural micro biota present in soil (Jauregui 2003).

Prothiofos oxon, an oxidized and metabolite of Prothiofos is because, the natural micro biota present in soil and it biotransforms (Gawad 2008, 2010).

From the data on levels of OPPs in the study area were compared with the similar results reported in and around India. **Rahmanikhah et.al** (2010) and found the concentration of chlorpyrifos and were 0.016 µg/l. **Lari et al** (2014) found dichlorvos (0-0.09) and chlorpyrifos (0-0.25) in ground water, Maharashtra. **Pan** (2017) analyzed groundwater samples from Yangtze River Basin and found Chlorpyrifos (309.67–336.92 ng/l) and ∑OPPs (1738.8–2194.3 ng/l). **Fosu-Mensah** (2016) found chlorpyrifos (0.04 ± 0.01µ/l) in cocoa farms in Dormaa West District, Ghana.

Organophosphorous pesticides and their residues in soil

The concentration of OPPs in the soil from the Heggadadevana kote is shown in the table. The most abundant pesticides detected were sum of Azinphos methyl (Azinphos methyl oxon), Prothiofos (Prothiofos oxon) chlorpyrifos, dichlorvos and Disulfoton (Disulfoton sulfoxide). Detection of Disulfoton sulfoxide, an oxidized and metabolite of Disulfoton is because, the natural micro biota present in soil (Szeto 1983). From the data on levels of OPPs in the study area were compared with the similar results reported in and around India. **Pan** (2017) analyzed groundwater samples from Yangtze River Basin and found Chlorpyrifos 0.40 – 5.58ng/g.

Water Sample collected site	**Organochlorine pesticides Compound**	**Metabolite group**	**Retention time**	**Ionization %**
Antharasanthe	DDE	DDT	2.77	4.20
	6-oxo-2-hydroxy-7-3,8,8,8-tetrachloroocta-2z,4z-dienotae	DDT	3.16	31.75

	Chlordene	Heptachlor	3.76	2.72
	Endosulfan	Endosulfan	4.68	10.37
	Methoxychlor olefin	Methoxychlor	4.81	17.30
	Aldrin	Aldrin	5.412	2.63
Hampura	DDE	DDT	2.766	2.29
	6-oxo-2-hydroxy-7-3,8,8,8-tetrachloroocta-2z,4z-dienotae	DDT	3.141	10.04
	Chlordene	Heptachlor	3.773	2.77
	Endosulfan sulphate	Endosulfan	4.593	1.68
	Endosulfan	Endosulfan	4.678	22.73
	Methoxychlor olefin	Methoxychlor	4.815	12.59
	Aldrin	Aldrin	5.40	3.32
Kandalike	Chlordene	Heptachlor	3.77	5.30
Kasaba	DDE	DDT	2.77	6.56
	6-oxo-2-hydroxy-7-3,8,8,8-tetrachloroocta-2z,4z-dienotae	DDT	3.16	45.71
	Chlordene	Heptachlor	3.76	5.04
	Methoxychlor	Methoxychlor	4.23	2.95
	Endosulfan sulphate	Endosulfan	4.32	3.11
	g-PCCH	g-HCH	4.40	6.24

Water Sample collected site	**Organophosphorous pesticides Compound**	**Metabolite group**	**Retention time**	**Ionization %**
Antharasanthe	Azinphos methyl oxon	Azinphos methyl	3.84	7.91
Hampura	Azinphos methyl oxon	Azinphos methyl	3.824	2.65
	Prothiofos oxon	Prothiofos	4.063	0.84
	Chlorpyrifos	Chlorpyrifos	4.371	18.46
Kandalike	Azinphos methyl oxon	Azinphos methyl	3.84	5.33
Kasaba	Azinphos methyl oxon	Azinphos methyl	3.84	16.87

Soil Sample collected sites	**Organochlorine pesticides Compound**	**Metabolite group**	**Retention time**	**Ionization %**
Kasaba	g-PCCH	g-HCH	3.756	2.64
	Methoxychlor olefin	Methoxychlor	4.814	7.25
	Chlordene	Heptachlor	5.139	3.75
Hampura	DDE	DDT	2.76	4.78
	6-oxo-2-hydroxy-7-3,8,8,8-tetrachloroocta-2z,4z-dienotae	DDT	3.12	25.78
	Endosulfan	Endosulfan	3.483	4.50
	g-PCCH	g-HCH	3.77	358
	DDMU	DDT	4.473	2.79
	Methoxychlor olefin	Methoxychlor	4.80	17.17
	Chlordene	Heptachlor	5.17	1.84
Antharasanthe	Endosulfan	Endosulfan	2.766	0.58
	6-oxo-2-hydroxy-7-3,8,8,8-tetrachloroocta-2z,4z-dienotae	DDT	3.141	10.42
	g-PCCH	g-HCH	3773	1.51
	Heptachlor epoxide	Heptachlor	3.927	2.09
	DDMU	DDT	4.490	2.85
	Methoxychlor olefin	Methoxychlor	4.815	22.78
	Chlordene	Heptachlor	5.156	3.40
Kandalike	Methoxychlor olefin	Methoxychlor	4.78	5.64
	Chlordene	Heptachlor	5.14	5.70

Soil Sample samples collected site	**Organophosphorous pesticides Compound**	**Metabolite group**	**Retention time**	**Ionization %**
Kasaba	Dichlorvos	Dichlorvos	3.517	22.51
	Azinphos methyl oxon	Azinphos methyl	3.841	6.35

	Prothiofos oxon	Prothiofos	4.097	12.0
Hampura	p-nitrophenol	Methyl parathion	286	4.83
	Chlorpyrifos	Chlorpyrifos	309	8.07
	Disulfoton sulfoxide	Disulfoton	363	1.29
	Azinphos methyl oxon	Azinphos methyl	3.84	3.09
	Prothiofos oxon	Prothiofos	4.35	1.48
	Chlorpyrifos	Chlorpyrifos	2.134	0.44
	Azinphos methyl oxon	Azinphos methyl	3.858	4.80
	Prothiofos oxon	Prothiofos	4.097	2.59
Kandalike	Azinphos methyl oxon	Azinphos methyl	3.84	5.80
	Prothiofos oxon	Prothiofos	4.10	3.61

Conclusion

Organochlorine pesticides and organophosphorous pesticides were detected in all the samples from HD kote, Karnataka. With DDT and heptachlor were predominate OCPs and azinophos methyl were predominate OPPs in water and soil samples. Some of the forbidden pesticides were persisted which may due to historic usage and remaining are from anthropogenic active use. Pesticides bioaccumulate in food chain and poses for longer duration in human and cause high health risk. However there should be regular monitoring of pesticide level and should be eliminate. Use of pesticides should be stopped and encourage to use bio pesticides.

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