Review Article

Review: Estimation of Pesticide in Water by Various Analytical Methods

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ABSTRACT

Pesticides have played a serious role in achieving the utmost crop production but maximum usage and accumulation of pesticide residues is very detrimental to aquatic and other ecosystem. Pesticide residues in water have become a major challenge over the last few decades and has been monitored in public water supply resources in national capital territory. Results shows that continuous consumption of contaminated water can pose severe health threats to local parts of this area. Central Pollution Control Board (CPCB), Delhi, shows that α and β isomers of endosulphan residues within the Yamuna river. High concentration levels of γ-HCH (0.259 µg/l) and malathion (2.618 µg/l) were detected within the surface water samples collected from the river Ganga in Kanpur, Uttar Pradesh (UP). High concentration levels of methyl parathion, endosulfan, and DDT were observed in water samples collected from the river at Bhagalpur, Bihar. The Industrial Toxicology Research Centre (ITRC), Lucknow (UP) study shows the result 0.5671 ppb concentrations of endosulfan in the river at Allahabad, UP. Same results were found in other water samples in India.

Keywords: Pesticide residues, Waters, HPLC, HF-LPME, SPE, Gas Chromatography.

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INTRODUCTION

According to WHO, "Pesticides are chemical compounds that are used to kill pests, including insects, rodents, fungi and unwanted plants (weeds). Pesticides are utilized in public health to kill vectors of disease, like mosquitoes, and in agriculture, to kill pests that damage crops. By their nature, pesticides are potentially toxic to other organisms, including humans, and need to be used safely and disposed of properly."

Ecological Effect of Pesticides[1]

The important point is that a lot of those effects are chronic (not lethal), are often not noticed by casual observers, yet have consequences for the whole organic phenomenon.

- Death of the animals.
- Cancers, tumors and lesions on fishes and animals.
- Reproductive inhibition or failure of organs in human as well as in animals. etc

These effects aren't necessarily caused solely by exposure to pesticides or other organic contaminants but could also be related to a mixture of environmental stresses like eutrophication and pathogens. These associated stresses needn't be large to possess a synergistic effect with organic micro pollutants.

Types of Pesticides[2]

These are grouped according to the types of pests which they kill:

Grouped by Types of Pests They Kill

- Insecticides – insects
- Herbicides – plants
- Rodenticides – rodents (rats & mice)
- Bactericides – bacteria
- Fungicides – fungi
- Larvicides – larvae
- Based on how biodegradable they are:

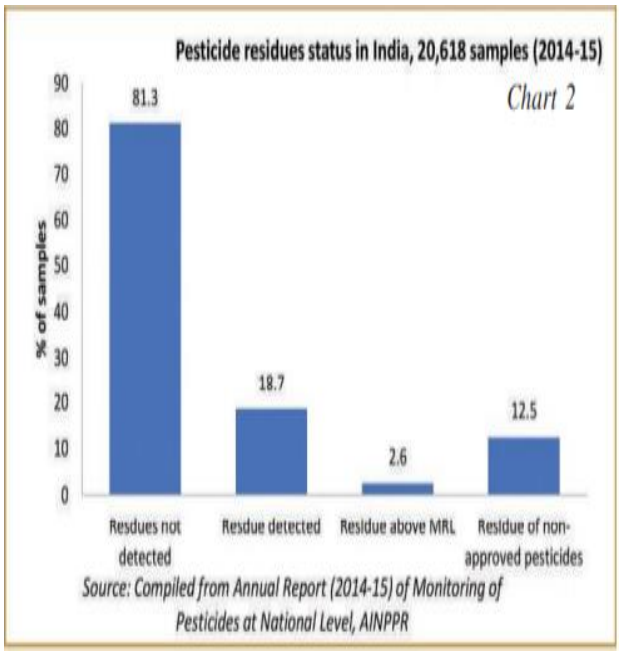
Pesticides can also be considered as in (Table no.1)

Table 1: Types of pesticide and their descriptions

Biodegradable		Chemically-related pesticides	
Name	**Description**	**Name**	**Description**
Persistent	These are types of biodegradables which may take months or years to break down	Organophosphate	Most organophosphates are insecticides; they affect the nervous system by disrupting the enzyme that regulates a neurotransmitter.
		Carbamate	Same as organophosphate but the enzyme effects are reversible.
		Organochlorine insecticides	They were commonly used earlier, but now many countries have been removed Organochlorine insecticides from their market due to their health and environmental effects (e.g., DDT, chlordane, and toxaphene).
		Pyrethroid	These are a synthetic version of pyrethrin, a naturally occurring pesticide, found in chrysanthemums(Flower)
		Sulfonylurea Herbicides	The sulfonylureas herbicides have been commercialized for weed control such as pyrithiobac-sodium, sulfometuron- methyl Sulfosulfuron, etc.
		Biopesticides	The biopesticides are certain types of pesticides derived from such natural materials as animals, plants, bacteria, and certain minerals.

History of Pesticides[2].

As on 30th October 2016, 275 pesticides were registered for use in India, of which about 255 are chemical poisons[2]. An analysis by PAN India (Presence across Nation) revealed that more than 115 pesticides out of the 275 are highly hazardous. Highly Hazardous Pesticides are those which have the potential to cause severe health implications such as high acute toxicity, long-term toxic effects like cancers, hormone disorders, reproductive and developmental disorders (Fig :1).



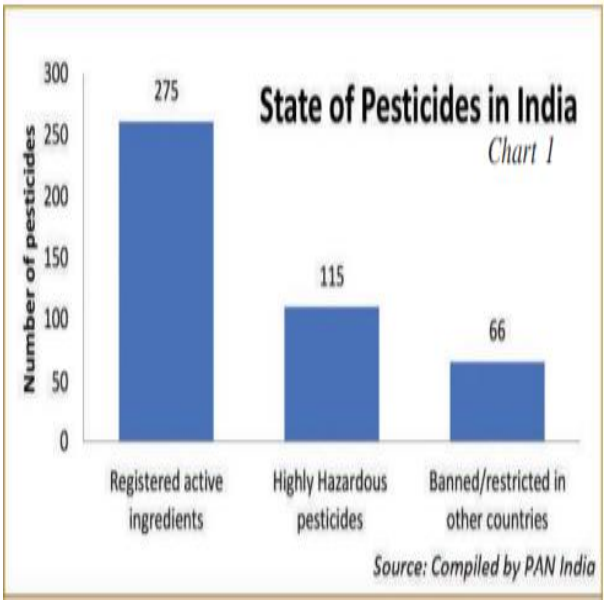


Figure 1: Graph of monitoring of pesticides at national level, AINPPR

Pesticide Use in India

The pesticide industries are successfully created a myth in humans mind that the pesticides are harmless or inevitable. Those pesticides are requirement in modern production systems, even though food production[3].

Thus, farmers started using them carelessly and still use nowadays. Field studies have shown that farmers in India do not use the required protective measures as recommended, affecting their health. Exposure to

pesticides and poisoning is a major problem among farming communities in India. Exposure and poisoning pose risk not only to farmers, but also agricultural workers, women, children, and consumers also.[2]

Problem Due to Pesticide Residue[2]

Groundwater contamination

When pesticides are sprayed on the crops and plant, they were easily flushed and accumulated on the surface of ground, reaching water-bearing aquifers, the pesticides are mixed with groundwater and making them unsuitable for both human and agricultural use.

Marine Life

When pesticides get into water bodies, water animals aren't spare because it can kill animals like fish. For example, if pesticide containing lead or copper get into water and sometimes fishes take them up and when human consume those fishes, they can damage multiple systems in human body that leads to the food chain disruptions.

Indian scenario of pesticide consumption

In India, largest pesticide consumption has been in the state of Uttar Pradesh, according to the data of 1995–1996 and 1999–2000, produced by Central Insecticide Board and Registration Committee, India.

The river Yamuna is that the largest tributary of river Ganga, and about 57 million people have dependencies on Yamuna river water. It is the most municipal drinking water source from Delhi to Agra. The presence of chlorinated pesticides in water of river Yamuna at selected sampling sites has only been reported which is given in (Fig: 2).

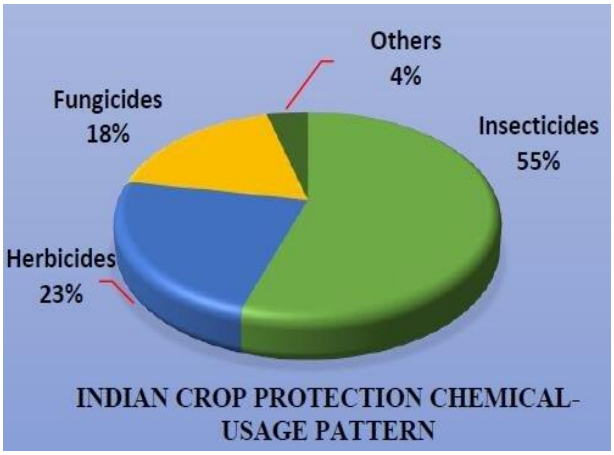


Figure 2: The graph shows the consumption of pesticide in Indian scenario which shows, Insecticide (55%), Herbicide (23%), fungicide (4%) and others (4%).

Global scenario of pesticide consumption

The worldwide consumption of pesticides is about two million tons per annum out of which 45 % which there's employed by Europe alone, 25 % which there's consumed within the USA, and 25 % in the rest of the world. India's share of pesticide consumption is just 3.75 %. The usage of pesticides in Korea and Japan is 6.6 and 12.0 kg/ha, respectively, Comparing the worldwide consumption of pesticide, 48% is the share of herbicides, 30% of insecticides, 18 % is that of fungicides, and others account for 7 % only which is given in (Fig: 3).

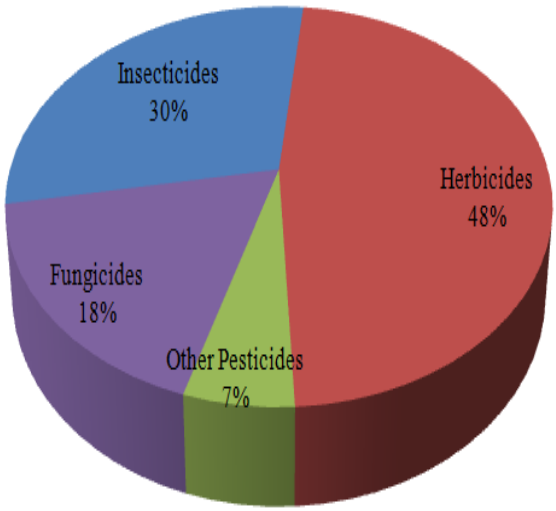


Figure 3: The graph shows the consumption of pesticide in worldwide scenario which shows Insecticide (30%), Herbicide (48%), fungicide (18%) and others (7%).

Different Methods are Used to Estimate Pesticide Residues[3].

To find out the concentration of pesticides several analytical methods have been proposed such methods are, Headspace solid-phase microextraction and gas chromatography-tandem mass spectrometry (HS-SPMC) (GC-MS/MS), High performance liquid chromatography (HPLC), Hollow fiber liquid phase microextraction (HF-LPME), Solid phase extraction (SPE) and solid phase microextraction (SPME), Ultra-High-Performance Liquid Chromatography with mass spectrometry (UHPLC–ESI-MS/MS), Solid Phase Extraction (SPE) with GC/ECD, NPD, Automated on-line trace-enrichment and liquid chromatography (LC), Reverse Phase HPLC, etc.

High performance liquid chromatography (HPLC)

Is a way in analytical chemistry wont to separate, identify, and quantify each component during a mixture .It relies on pumps to pass a pressurized liquid solvent containing the sample mixture through a column crammed with a solid adsorbent material.

Hollow fibre liquid phase microextraction (HF-LPME)

In this technique the extracting phase was placed inside the lumen of a porous extracting phase was placed inside the lumen of a porous polypropylene hollow fiber in which the extraction solvent is stabilized and increases the interfacial area between solvent and aqueous sample ,thus increasing the extraction efficiency.

Solid phase extraction (SPE)

Is an extractive technique by which compounds that are dissolved or suspended during a liquid mixture are separated from other compounds within the mixture consistent with their physical and chemical properties .

High resolution gas chromatography - high resolution mass spectrometry (HRGC-HRMS)

High resolution gas chromatography /mass spectrometry is the most suitable techniques for analysis of environmental pollutants, food products, volatile compounds etc. Methods which are developed are listed below (Table no. 2)

Table 2: Different analytical methods used in estimation of pesticides.

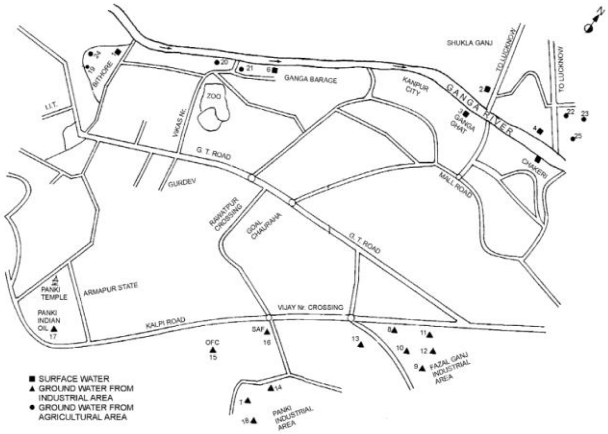
Methods	Types of Pesticides	Water samples
Headspace solid phase microextraction and gas chromatography-tandem mass spectrometry (HS-SPMC) (GC-MS/MS)	Organochlorine (OCP)	Deionized water from Milli-Q purification system (Millipore, Milford, MA, USA)
High performance liquid chromatography (HPLC)	Polycyclic aromatic hydrocarbons (PAHs)	Rain water ,during pre-monsoon and post monsoon (Yamuna river bank)(Delhi)
Hollow fiber liquid phase microextraction (HF-LPME)	Parathion-Methyl, Chlorpyrifos, Captan, Procymidone, α-Endosulfan, Prothiofos, Cyproconazole, Ethion, Triazophos, Phosmet	Surface water in the region of crops and coffee,eucalyptus. And tomatoes.
Solid phase extraction (SPE) And solid phase microextraction (SPME)	Chlorinated, Organophosphorus, Triazines, Pyretroids And Chloroacetamides	Ground water source from squalia river basin (argentina)
Ultra-High-Performance Liquid Chromatography with mass spectrometry (UHPLC–ESI-MS/MS)	Glyphosate And AMPA	Runoff water sources from field of po-valley(north east- italy)
Solid Phase Extraction (SPE) with GC/ECD, NPD.	Alachlor, Captan, Chlorpyrifos, Ciazinon, Profenophos And Oxyfluorfen	Environmental water (Sri Lanka)
Automated on-line trace-enrichment and liquid chromatography (LC)	Simazine, Atrazine.	Ground water and fresh water.
Reverse Phase HPLC	Organo-Phousphorus(Op)	Environmental water
High performance liquid chromatography with fluorescence detection (HPLC-FLD)	Glyphosate and AMPA	Tap water and rain water sample, lake water and river water. (Jiulong River)

MATERIALS AND METHODS

Pesticide Residue in Ground Water

Collection and extraction of sample

In Jaipur[15], Rajasthan, total 50 samples were collected during the study period. For the collection of water samples, Out of these five zones central, east and south zones get water supply from driven well and therefore the north and west zones are supplied from Ramgarh water reservoir, which is about 35 km far away from the city/town. The water samples were extracted within 48 hrs of collection. All the glassware were properly washed with soap water followed by distilled water and finally rinsed with acetone and heated at 220°C in an oven to avoid any contamination of pesticides. In Kanpur[6], A total of seven sampling locations were identified between Bithore and Jajmau for ground water samples from agricultural areas. They were Bithore, Hawatpur, Kalupurwa, Rautapur, Nikarhara, Mardankhara and Jajmau (Fig : 8).



Surface water: 1 Bithore, 2 Gangaghat (shuklaganj), 3 Ghangaghat (Kanpur City), 4 Jajmau, 5 Chakeri, 6 Gangabaraj, **Industrial area:** 7 Chandan factory, 8 Singhasani chemical, 9 Inder themit, 10 Bengal Chemical Pharma, 11 J.K. Jute Mill, 12 Bharat Petroleum, 13, Aliga Rubber, 14 Auto Fasteners, 15 OFC Kanpur, 16 Vijay Nagar (SAF), 17 Panki Indian Oil, 18 Tata Salt Factory, **Agricultural Area:** 19 Hawatpur, 20 Kulupurwa, 21, Rautapur, 22 Nikarkhera, 23 Mardahkhera, 24 Bithore, 25 Mansa Kher.

Figure 8: Sampling area of Kanpur

● - Shows ground water from agricultural area

▲ - Shows ground water from industrial area

For the ground water samples, around 30–40 l of water was flushed out of the hand pumps before the gathering.

Duplicate samples for pesticide measurement were collected from each area of sampling location. In Unnao district, total 96 samples (42 samples from dug wells and 54 samples from bore wells), were they collected in the month of October-November 2003, using high quality brown bottles, the collection of groundwater is in the region occurs at shallow depth of less than 10 m below ground level (bgl) (Fig: 9). In Kolkata[1], the groundwater samples are collected from regions were hand pumps and tube wells are located tapping aquifers between 40 m and 180 m bgl. The sample have been collected from the outlets after flushing water for 10 minutes just in case of hand pump, and 5 minutes just in case of bore well, in order to obtain fresh aquifer water (Fig : 10)

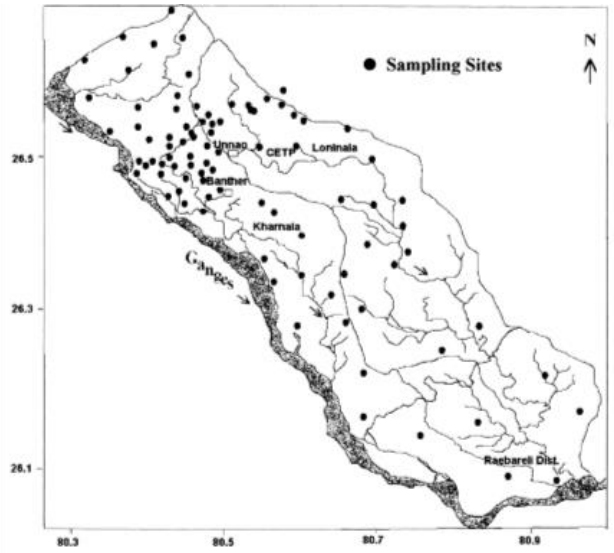


Figure 9: Sampling area of Unnao district in the Gangetic alluvial plains in northern part of India.

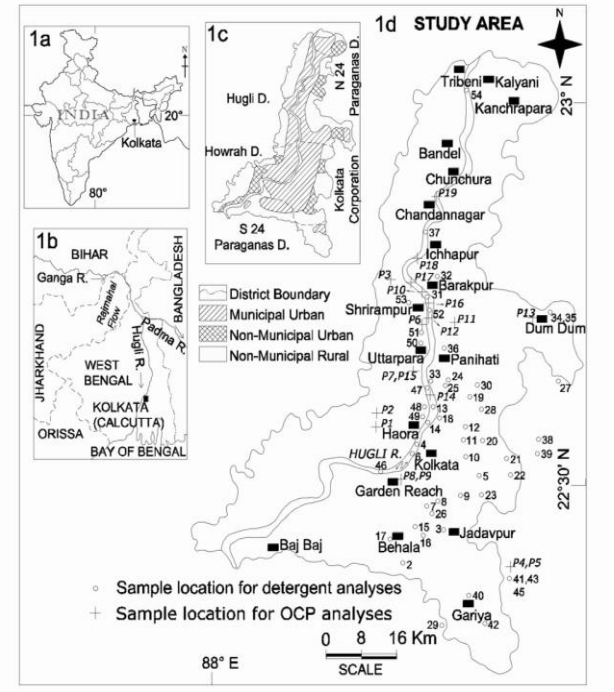


Figure 10: Sampling area of Sonapur, greater Kolkata and Palta, greater Kolkata

Pesticide Residues in Surface Water

Collection and extraction of sample

In Kanpur[6], Surface water was sampled from the River Ganges along the 35-km stretch of Kanpur Seven sampling locations were chosen along this stretch, which include Bithore, Gangabharaj, Gangaghat (Kanpur city), Gangaghat (Shuklaghanj), Chakeri and Jajmau. Around 30–40 l of water was flushed out of the hand pumps before the gathering. Duplicate samples for pesticide measurement were gathered from each sampling location.

In Unnao district[13], total 86 samples were collected in deferent areas at the month of October-November 2003, using high quality brown bottles. For the collection of surface water. In Hisar district, Haryana[2]. A total of 38 samples, in triplicate, were gathered on two occasions, i.e. June and October 1999. In Haryana state, monsoon usually extends from July to September. The samples collected in the first and second week of June represented the pre-monsoon/summer season, while the samples of October represented the post monsoon period (Fig: 11).Surface and ground water areas of Vidarbha region of Maharashtra state – Bhandara (3717 sq km), Amravati (12212 sq km) and Yavatmal (13584 sq km) Collection of water samples were performed out from September 2011 to July 2012. Total numbers of 156 water samples were collected from different parts of Maharashtra. Grab sampling was done (Fig: 12). Real samples of surface water were collected in a part of rural area of the state of Minas Gerais, Brazil[11]. In these areas the main crops are coffee, eucalyptus, and tomatoes. Samples of surface water were gathered from 2 km downstream of these crops.

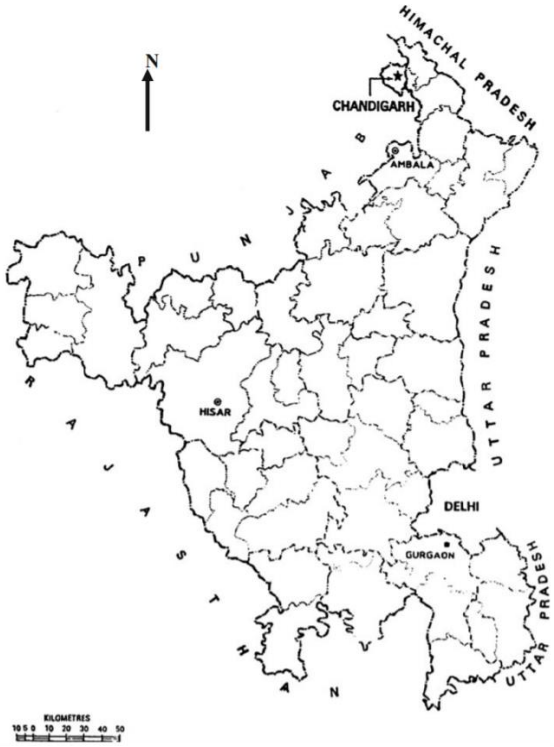


Figure 11: Sampling location of Hisar, Haryana

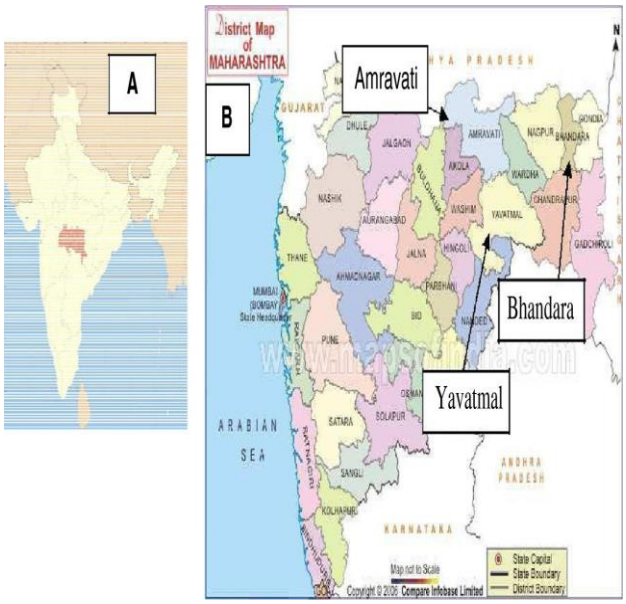


Figure 12: (A): Map of India highlighting Vidarbha region, (B): Sampling location of Amravati, Yavatmal and Bhandara.

Pesticide Residues in River And Sea Water

Collection and extraction of sample

Seawater samples were gathered from Chennai harbour and Cuddalore fishing harbour. Sediment samples were gathered from all the six locations between 0.5 and 10 nautical miles (nmi) from the shore using the grab sampler in the Department of Ocean Development (DOD, India) coastal research vessel. The samples were place in clean polyethylene bags and brought to the laboratory under cold condition (48℃) and frozen at 208℃ until chemical analysis (Fig: 13). In Kunao river, UP[7]. The water samples were gathered in pre-monsoon and post-monsoon period (June, 2004 to May 2006) in between 8.0 to 10.0 AM in first week of each month from two different spots areas (S1 and S2 as well as along the banks of river B1 and B2). In Champanala, Ganga and Mond ghat, Ganga river, Bhagalpur, Bihar[9] (Fig: 14). The water sample of the river was gathered in sterilized plastic container. The containers were carefully filled just to overflowing, without passing air bubbles through sample or trapping air bubbles in sealed container. In Yamuna River, Wazirabad and Yamuna River, Okhla, Delh[12]. The water sample of the river was collected in sterilized plastic container. The solvents used were of Laboratory grade and were purchased locally and were purified by glass distillation Different types of aqueous samples were gathered and analysed according to the procedure described above. Tap water and rain water were gathered and measured directly without further treatment. Lake water was gathered from the lake in Xiamen University[10] and river water was gathered from upstream of the Jiulong River. Seawater of salinity 35, used as sample matrix, was gathered using Niskin bottles during a cruise in the Western Pacific in April 2015.

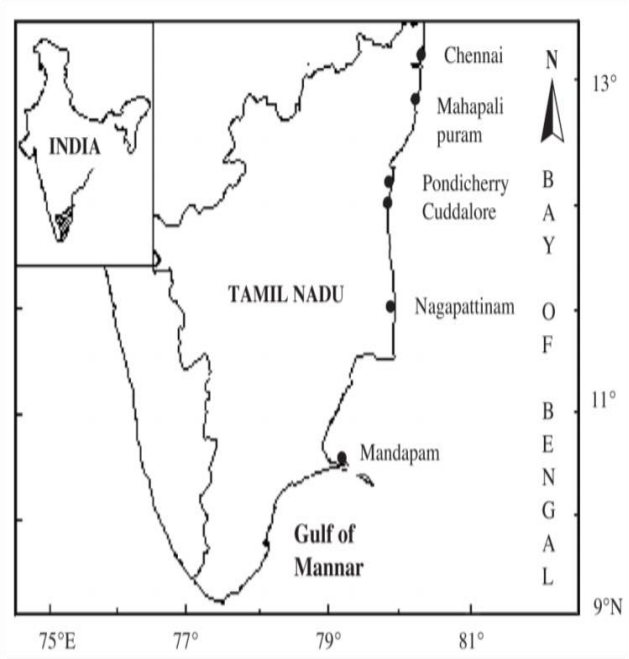


Figure 14: Sampling location of Chennai harbour and Cuddalore fishing harbour

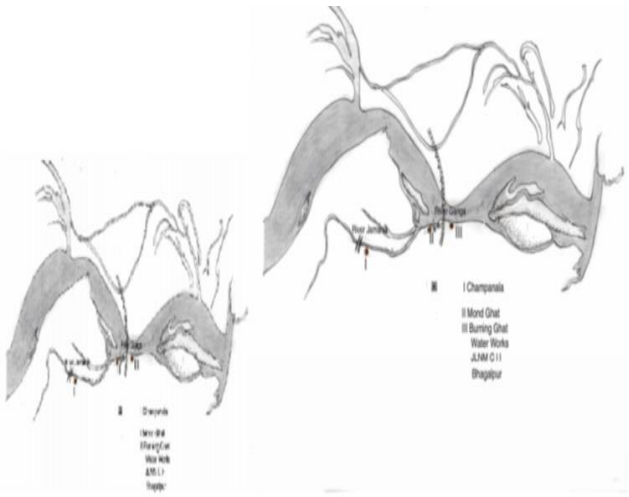


Figure 15: Sampling location of River Ganga in Nathnagar-Bhagalpur Stretch (Bihar)

RESULTS

Pesticide Residue in Ground Water, Surface Water, River and Sea Water

The concentrations of total heptachlor epoxide ,OCP residues in Ramgadh water were observed, OCP residues shows seasonal variations maximum concentration were observe during the rainy season followed by winter season, in winter seasons, adrin and hepatochlor epoxide were observe during rainy season. In unnao district, ground water of kolkata and adrin residues below limit were observed. The results are mentioned in (Table no. 3, 4 and 5).

Table 3: Detected pesticide residue in ground water.

S.N	Place/state	Pesticides detected	Concentration of pesticides detected	Method used
1	Ramgarh water reservoir, north and west zone of Jaipur, Rajasthan,	Heptachlor epoxide	1.121 µg/l	GC-ECD
		Aldrin	0.336 µg/l	
		Heptachlor	0.231 µg/l	
	Central, east and south zone of Jaipur, Rajasthan, water source tube well	Heptachlor	0.694 µg/l	
		Aldrin	0.136 µg/l	
		Heptachlor epoxide	0.657 µg/l	GC-ECD
	Summer season Jaipur, Rajasthan	Heptachlor epoxide	0.609 µg/l	
	Winter season Jaipur, Rajasthan	Aldrin	0.350 µg/l	
	Rainy season Jaipur, Rajasthan	Heptachlor epoxide	0.943 µg/l	
2	Dug wells, Unnao (UP)	Aldrin	BDL—1355.2 µg/l	
		Chlordane	BDL—7.2 µg/l	
		Endosulfan	BDL—54.4 µg/l	
3	Agricultural areas, Kanpur, UP	α-HCH	0.189 µg/l	GC-ECD & GC-MS
	Industrial areas, Kanpur, UP	γ-HCH	0.145–0.915 µg/l	
4	Sonapur, greater Kolkata	Aldrin/dieldrin	0.01–0.03 µg/l	GLC
		Dicifol	0.02–0.03 µg/l	
	Palta, greater Kolkata	Heptachlor	0.01–0.01 µg/l	
		DDT	0.30–0.50 µg/l	
European Economic Commission Standards (EEC Council Directive 1980/778/EEC) for ground water: the total pesticide level should not exceed 0.620 µg/l and, individual pesticide should not be greater than 0.10 µg/l				

Table 4: Detected pesticide residue in Surface water.

S.N	Place/state	Pesticides detected	Concentration of pesticides detected	Method used
1	Surface water, Kanpur, UP	γ-HCH	0.259 µg/l	GC-ECD and GC-MS
		α-HCH	0.190 µg/l	
		Dieldrin	1.671 µg/l	
		Malathion	2.618 µg/l	
2	Unnao (UP)	Aldrin	BDL—1.88 µg/l	GC-ECD
		Chlordane	BDL— 0.04 µg/l	
		DDT	BDL— 0.23 µg/l	
		HCH	1.88–1.95 µg/l	
		Heptachlor	BDL—0.11 µg/l	
3	Agriculture intensive areas, Bhandara region, Maharashtra	α-HCH	0.06 µg/l	GC-ECD & GC-MS
		Endosulphan	0.08 µg/l	
		Dichlorvos	0.20 µg/l	
	Agriculture intensive areas, Amravati region, Maharashtra	Phorate	0.19 µg/l	
		Chlorpyrifos	0.26 µg/l	
		Parathion-methyl	0.15 µg/l	
	Agriculture intensive areas, Yavatmal region, Maharashtra	Chlorpyrifos	0.44 µg/l	
		Parathion-methyl	0.17 µg/l	
4	Hisar, Haryana	DDT	50.1–332.2 µg/l	GC-ECD
		HCH	2.3–560.6 µg/l	

5	Minas Gerais, Brazil	Parathion-methyl	0.04 -0.14 µg/l	HF-LPME
		Chlorpyrifos	0.44 -1.46 µg/l	
		Captan	0.20 -0.67 µg/l	
		Procymidone	0.17- 0.57 µg/l	
		α-Endosulfan	0.12 -1.69 µg/l	
		Prothiofos	0.35 -1.16 µg/l	
		Cyproconazole	0.14 -0.48 µg/l	
		Ethion	0.13- 0.42 µg/l	
		Triazophos	0.09- 0.31 µg/l	
		Phosmet	0.23 -0.76 µg/l	
European Economic Commission Standards (EEC Council Directive 1980/778/EEC) for surface water: the total pesticide level should not exceed 0.60 µg/l, and individual pesticide should not be greater than 0.10 µg/l				

Table 5: Detected pesticide residue in River and Sea water.

S.N	Place/state	Pesticides detected	Concentration of pesticides detected	Method used
1	Seawater, Chennai harbor, Bay of Bangal	PCB	0.0458 µg/l	HRGC-HRMS
		DDT	0.235 µg/l	
	Seawater, Cuddalore fishing harbor, Bay of Bangal	PCB	0.0109 µg/l	
		DDT	0.0543 µg/l	
2	Pre monsoon, Kuano river, UP	α-HCH	0.013–0.019 µg/l	TLC METHOD
		β-HCH	0.016–0.027 µg/l	
		γ-HCH	0.0006–0.015 µg/l	
		DDT	0.0002–0.0009 µg/l	
	Post monsoon, Kuano river, UP	α-HCH	0.016–0.020 µg/l	
		β-HCH	0.010–0.029 µg/l	
		γ-HCH	0.0008–0.007 µg/l	
		DDT	0.0003–0.003 µg/l	
3	Champanala, Ganga river, Bhagalpur, Bihar	α-Endosulfan	0.0456 µg/l	GC-ECD & GC-MS
		o,p-DDT	0.0342 µg/l	
	Mond ghat, Ganga river, Bhagalpur, Bihar	Lindane	0.0657 µg/l	
		α-Endosulfan	0.073µg/l	
		β-Endosulfan	0.130 µg/l	
	Burning ghat, Ganga river, Bhagalpur, Bihar	α-Endosulfan	0.012 µg/l	
		o,p-DDT	0.054 µg/l	
		p,p'-DDT	0.123 µg/l	
4	Yamuna river, Wazirabad, Delhi	DDT	0.11 µg/l	GLC
		α-Endosulfan	0.219 µg/l	
	Yamuna river, Okhla, Delhi	β-Endosulfan	0.236 µg/l	
		α-Endosulfan	0.324 µg/l	
		p,p'-DDT	0.13 µg/l	
5	Jiulong River, Western Pacific, Fujian, China			
	Pure water	GLYP	0.24 -0.80 µg/l	HPLC-FLD
		AMPA	0.06 - 0.20 µg/l	
	Artificial sea water	GLYP	0.60 -2.00 µg/l	
		AMPA	0.30 -1.00 µg/l	
European Economic Commission Standards (EEC Council Directive 1980/778/EEC) for river and sea water: the total pesticide level should not exceed 0.50 µg/l, and individual pesticide should not be greater than 0.10 µg/l				

CONCLUSION

Pesticide residues are found in soil, air, surface water, and groundwater. The important pesticides found in water from different sources are OCPs, OPPs, and their derivatives as they persist long in the environment. The rivers get seriously polluted due to discharge of toxic heavy metals and pesticides. The present study reports the contamination status of OCPs, OPPs, POPs, and SPs in ground, surface, river, and drinking water in India. The conclusion of the present study is the better understanding of cause of deterioration of water quality due to pesticide residues and developing strategies to minimize the losses caused by residues.

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