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Research Article

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To studies organochlorine insecticides in different brands of cold drinks

P. K. Pankaj[1], Prasann Kumar[2*], R. C. Nigam[3] and P. K. Mishra[1]

[1]Department of Soil Science and Agricultural Chemistry, Banaras Hindu University, Varanasi.
[2]Department of Plant Physiology, Institute of Agricultural Sciences, Banaras Hindu University, Varanasi
[3]Department of Soil Science and Agricultural Chemistry, Chandra Shekhar Azad University of Agricultural and Technology, Kanpur.

ABSTRACT

The present endeavour entitled To studies organochlorine insecticides in different brands in cold drinks in different brands in cold drinks was carried out for the fulfilment of aforesaid with nine different brands of samples viz. Pepsi, Mountain Dew, Thums up, Mirinda, Fanta, Sprite, Limka, Coca-Cola and Slice were collected. The total 28 samples including all nine different brands were processed, extracted and clean up by using multi- residues technique, finally the insecticidal residues of chlorinated hydrocarbons and were determined through Gas Liquid Chromatograph (GLC). The result obtained are-Total HCH-100% samples of cold drink of different brands were found contaminated with HCH residues. In maximum samples γ-isomers (Lindane) were detected. The concentration was found below its MRL value (3µg/L). Residues of DDT including pp-DDT, op-DDT, pp-DDE, OP-DDE and pp-DDE were found in 50% of the total samples of different brands of cold drink. pp-DDT and op-DDT were main contaminant. The concentration was found within their safe limit (1µg/L). Residues of dicofol, endosulfan and Chloropyriphos were found in 48%, 60% and 46% of the total cold drink samples of different brands .

Key words: Cold drinks, DDT, Organochlorine, Pesticides, Water, Ground, River, Contamination

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INTRODUCTION

Ground water and river water contamination is a matter of concern, as the contaminants, especially pesticides, may find their way into drinking water. This results in a health hazard to the consumers, as ground water is the main source of potable water in most parts of India. The presence of organochlorine pesticide residues in food commodities water [2], mother's milk, dairy milk and animal feed, human blood. The organochlorine insecticides (DDT, HCH, Aldrin, Endrin) have been banned in most of the developed countries due to their persistent nature or due to the toxicity of one of their metabolites e.g., endrin-epoxide. Endosulfan is the only organochlorine insecticide still used the world over. In India, although DDT has been banned for use in agriculture, it is still used for control of vectors in public health. HCH was banned for use in agriculture in 1998 and linden is recommended in its place. In spite of this there are reports of the presence of these insecticides in various commodities such as milk, animal feed and vegetables. The presence of small amounts of organochlorine insecticides (Mohapatra *et al*., 1995) in agriculture areas in rural India indicates regular use of these compounds. Organochlorines pesticides were found in all the major rivers of India. Some scientist also reported that HCH was found in high concentrations in surface sediments from the river Kaveri, South India, confirming the extensive use of HCH in these areas. There are reports in the literature

on the analysis of pesticide residues in water. Reports are also available on the detection of organochlorines in Malaysian river systems and in samples of water, sediment and fish in Turkey. The threat of pesticides in ground water is that any toxic chemical present will reach the drinking water system, which may prove to be harmful for human consumption. Various organisations and governments all over the world are making efforts to monitor the contamination of water and attempts are being made to check the flow of effluents from agricultural fields into rivers and streams so as to reduce drinking water contamination. This paper presents results from the monitoring for the presence of organochlorine insecticides in drinking water in and around Delhi and in ground water used for irrigation in areas adjoining Delhi.

MATERIALS AND METHODS

The present study entitled "Monitoring of pesticide residues in different brands of cold drink was carried out by adopting the following materials and methods.

1. MATERIALS

a. Equipment: Single pan electric balance, Refrigerator for storage of technical grade pesticides and sample extracts, Rotatory vacuum evaporator with temperature controlled water bath, GLC (Gas liquid Chromatography) GC 17A Ver.3model, Micro Syringe (10 µL capacity),

b. Glass Wares: Separator funnel (1000 ml), Beaker (100 ml), Measuring cylinder (500 ml cap), Glass rod, Absorbent column (2.5 cm ID and 15 cm long), Iodine flask, Graduated test tube (10 ml), Stoppered cylinder (100 ml), Micro pipette (1 ml).

c. Chemical and miscellaneous: Distilled n- Hexane, Methylene Chloride, Sodium Chloride, Activated Anhydrous Sodium Sulphate, Distilled acetone, HPLC grade n-hexane, HPLC grade acetone

2. EXPERIMENTAL PROCEDURE:

(a) Sampling: 500 ml cold drink bottle of different brands of from sites of Kanpur City and its adjoining area were collected and stored in refrigerator for analytical work.

(b) Extraction and clean up of cold drink: The extraction of cold drink sample was done by the following methods- **(a**-500 ml of cold drink sample was taken in one liter separatory funnel and to this 10 g. of sodium chloride was added. The funnel was shoak to dissolve sodium chloride completely**, (b**- The residues were extracted thrice with methylen chloride (50:25:25 ml), each time shaking vigorously for 1 minute releasing the pressure intermittently**. (c**- Dry lower organic layer by passing through about 1.5^{11} anhydrous sodium sulfate supported on washed clean wool in 4^{11} funnel, **(d**- Combined the organic layers and concentrated to 0.5 ml using vacuum rotatory evaporator at 40 ^{0}C. concentration step was repeated thrice in the presence of distilled hexane to remove all traces of methylene chloride, **(e**- The residues were dissolved in 10 ml graduated test tube and finally make up the volume with HPLC grade n-hexane.

(c**) Estimation:** The multi-residues of pesticides were estimated in accordance with the following analytical process and the specific parameter of GLC for particular insecticide. **Instrument: (**GLC Shimadzo 17 A ver.3 model, Detector =ECD (electron capture detector) equipped with N^{63}). **Column: (**Capillary column D B-1. Film thickness, 0.25 µm. Inner diameter: 0.25 ID, Column length: 30 m). **Temperature:** Oven = 200 ^{0}C, Column = 250 ^{0}C, Detector = 280 ^{0}C. **Carrier gas:** Nitrogen @40mL /minute, Hydrogen @30 mL/minute, O Air @30 mL/ minute, attention 1 for 0°C & sp. and O for OP, Column mode- 2mL/minute, Injection mode-Split, Split ratio-20.

(d) Recovery studies: A representative sample of 50mL cold drink of each brand was fortified at 1-ppb. Level either different standards of Organochlorine, Synthetic pyrethroid and organophosphate. The fortified sample were processed as per the methodology and residues of organochlorine synthetic pyrethroid and organophosphate were estimated by GLC technique (Gas Liquid chromatography)

(e**) Calculation**: Residues level in sample was calculated as follows.

Residues level in $\mathrm{mg\ Kg^{-1}} = \frac{As}{Astd} X \frac{M}{M1} X \frac{V}{V1}$

AS=Peak area of unknown sample
Astd= peak are of the standard
M=µL of standard injected-1µL

$M_{1=\mu L}$ of sample extract injected-2µL
V= volume of the sample extract in mL.
V_1= Volume of the sample in litre.

RESULTS AND DISCUSSION

1. **HCH residues:-** Twenty eight cold drink samples were collected from various locations and monitored for residues of various isomers of hexa- chlorocyclohexane. Results are summarized Table -6 shows that all the samples of different brands of cold drink were found to be contaminated with HCH. The total HCH contamination including α, β, ϒ and $^{\delta}$ isomer varied from 0.016 to 0.212 µg/L in pepsi brand of cold drink. Similarly, in Mountain –dew cold drink samples contained the residues of various isomers of HCH in the range of 0.181 to 0.331 µg/L. In thums up cold drink samples contained the total HCH residues from 0.092 to 0.232 µg/L. While in Miranda cold drink samples contain the total concentration of HCH varied from 0.107 to 0.261 µg/L. The total HCH including all isomers in Fanta cold drinks samples contained the residues in the range of 0.105 to 0.312µg/L. In Sprite cold drink samples total HCH Varied from 0.112 to o.285 µg/L. While in case of Limca cold drink samples contained different isomers of HCH in range of 0.083 to 0.125 µg/L. In Coca-cola drinks samples were also found contaminated with α, β, ϒ and $^{\delta}$ HCH residues varied from 0.022 to 0.0243 µg/L. In Slice cold drink samples contained HCH residues in range of 0.103 to 0.0346 µg/L. Kumar et al. [1998] surved to lakes at Jalmahal and Mohalon around Jaipur during 1985-86 [7]. These lakes were characterized by high fish productivity and were selected because of their importance as recreational areas. These lakes were found contaminated with moderate to high levels of residues of different insecticides. In Jalmohal lakes, the total organochlorine insecticides were in the range from 0.17 to 6.6 ppb. In both lakes, residues of DDT exceeded the recommended limit of water quality criteria. Mahapatra et al. [1995] found that the ground water from rural area near Farrukhabad in the vicinity of Ganga rivers was contaminated with organochlorine insecticides [8]. ϒ-HCH concentrations were well below the MRL value. Total DDT (ND-1.657 ppb). Aldrin and dieldrin (ND-0.908 ppb) and heptachlor (ND-0.129 ppb) were higher than their respective MRL values in some samples of drinking water. Kulshreshtha [1989] reported average residues of total HCH (272 ppb.) and DDT (21.9 ppb.) in Kshipra river whereas average residues of total DDT (14.47 ppb.) were found to be in Chambal river [6]. Baker et al. [1990] monitored the organochlorine insecticides in 92 water samples of the Mahala reservoir, situated at Jaipur [4]. All water samples were found to be contaminated with organochlorine insecticides which were in the range from 1.07 to 81.23 ppb. ϒ-HCH was most frequently (91%) distributed pesticides with concentration ranging from 0.10 to 44.72 ppb followed by a –HCH (88%) ranging from 0.11 to 20.12 ppb.

2. **DDT Residues**:- Table-1 reveals that Pepsi cold drink samples were found to be contaminated with total DDT in range of ND to 0.062 µg/L. Out of 4 Pepsi samples only two samples were contaminated with pp-DDT and op-DDT isomers. Six mountain dew cold drink samples was also found contaminated in range of ND to 0.081 µg/L. Out of six samples only two samples mainly contained op-DDT and pp-DDT isomers (table-1).In Thums up cold drink samples also found contaminated with DDT isomers in range of ND to 0.073 µg/L. Only two cold drink samples out of four analysed, showed contaminated with only op-DDT isomers. The Mirinda cold drink samples the total DDT concentration was ND to 0.064 µg/L and one samples showed only pp DDT and op-DDT isomer. While Sprite cold drink samples the total DDT concentration was found in range of ND to 0.042 µg/L. In Limca cold drink samples the total DDT contamination was recorded in range of ND to 0.063 µg/ L including op-DDT, pp-DDT and pp-DDE isomers. In Coca cola cold drink samples out of two samples only one sample was found contain total DDT from ND to 0.068µg/L. In Slice cold drink samples were also found contaminated with total DDT in range of ND to 0.085 µg/L including pp-DDT and op-DDT isomers in one samples. Rao et al. [1986] reported the DDT residues in Yamuna river to the extent of 2.15 ppb [10]. Water samples from Yamuna River showed DDT residues ranging from 0.062-0.963 ppb [1].

Endosulfan residues:- Summary table-1 reveals that Pepsi, Mountain Dew ,Thumsup, Mirinda, Fanta, Sprite, Limca, Coca cola and Slice cold drink samples also found contaminated with total endosulfan including endodulfan-α and endosulfan-β isomers, Endosulfan isomers contains only one samples of Mountain Dew and one samples of Mirinda cold drink, out of twenty eight samples of different cold drinks. Summary table-1 also shows that quantitatively Mirinda, Thumsup and Mountain Dew had more quantity of total endosulfan (ND to 0.371 µg/L) in comparision other brands of cold drink samples (ND to 0.008 µg/L) in comparision other brands of cold drink samples (ND to 0.008 µg/L). Prabhaker et.al. [2003] reported that out of the 322 samples of ground and surface water, 105 samples were contaminated with DDT residues, 149 samples with the HCH, 88 samples with endosulfan and 93 samples with other commonly used organochlorine and organophosphorus pesticide [9]. Kathpal et.al

[2004)] analysed water sample from Keoladeo National Park Bharatpur and found that in the all water sample, total residues of HCH, DDT and endosulfan ranged from 0.0065-0.199, 0.014-0.560 and 0.00020-0.039 ppb respectively [5].

Chloriphos residues:- Summary table -1 reveals that Pepsi cold drink samples were found to contain Chlorpyriphos in the range of ND to 0.126 µg/L. In Pepsi only one sample was contaminated out of four samples analysed. Mountain Dew cold drink also found contaminated in range of ND to 0.21 µg/L in all three samples except one samples not detected chlorpyriphos insecticide. In Thums up brand of cold drink samples showed chlorpyriphos residues in range of ND to 0.081 µg/l. Only one sample out of four showed their presence. Mirinda, Fanta, Sprite, Limca, Coca-cola and Slice cold drink samples also found to be contaminated with chlorpyriphos in very low concentration i.e. ND to 0.08 µg/L in comparison to other brands of cold drink. The report published by CSE on the presence of pesticides in packaged water and soft drinks created not only awareness but also over reaction by public considering the seriousness of the matter, the government constituted a joint parliamentary committee. Committee recorded that the findings of CSE are correct on the presence of pesticides in carbonated water in respect of three samples each of 12 brand products of Pepsi and Coca Cola. CSE analysed 36 samples of different brands products of Pepsi and Coca-Cola and tested for 32 most used pesticides of India (16 OC_s, 12, OP_s and 4 SP_s). CSE detected lindane and chlorpyrifos in all 36 samples, malathion in 35 and DDT and its metabolites in 29. Synthetic Pyrethroids were not found in any of the 36 samples. Some of the samples were also independently analysed at CETRI mysore and CFL Kolkata and the results were confirmed by the use of GC-MS [3].

Dicofol residues:- Summary table-6 reveals that all the brands of cold drink also contaminated with dicofol insecticide. Their contamination in Pepsi cold drink was in range of ND-0.08µg/L. Two samples not were free from of dicofol residue (Table-1). In Mountain Dew cold drink also indicate same quantity of residues i.e. ND to 0.08µg/L and and found 50% contamination in all six samples of mountaindew. In Thums up brand of cold drink, contamination of dicofol was also found in the range of ND to 0.08 µg/L in only two samples, out of four analysed. Mirinda, Fanta, Sprite and Limca brand of cold drink samples showed low amount of concentration i.e. ND to 0.06, 0.051, 0.062 and 0.013µg/L respectively. While Coca Cola brand of cold drink found free from dicofol residues. Two Slice cold drink samples was analysed and one sample was found contaminated (0.08µg/L) with dicofol and others free from dicofol residues. Many scientists reported the residues of DDT, HCH, Aldrin, Dieldrin and Dicofol in water samples from various sources in various concentrations [2]. The contamination of water was highest in samples collected from tanks followed by open wells>tube wells>canal river>ponds/lakes. Residues of DDT and HCH have also found to be reported from various centers of AICRP on pesticide residues.

Table: 1 Conc. Of organochlorine insecticides in cold drinks (µg/L)

Name of Brands	Total HCH (Mean Value)	Total DDT (Mean Value)	Total Endosulfan (Mean Value)	Chlorpyriphos	Dicofol
Pepsi	0.016-0.212	ND-0.062	ND-0.026	ND-0.126	ND-0.08
Mountain Dew	0.181-0.331	ND-0.081	ND-0.136	ND-0. 21	ND-0.08
Thumps UP	0.092-0.232	ND-0.073	ND-0.371	ND-0.081	ND-0.08
Mirinda	0.107-0.261	ND-0.075	ND-0. 215	ND-0.04	ND-0.06
Fanta	0.105-0.312	ND-0.064	ND-0.081	ND-0.06	ND-0.051
Sprite	0.112-0.285	ND-0.042	ND-0.042	ND-0.06	ND-0.062
Limca	0.083-0.125	ND-0.063	ND-0.038	ND-0.02	ND-0.013
Coca Cola	0.022-0.243	ND-0.068	ND-0.008	ND-0.08	ND
Slice	0.103-0.346	ND-0.085	ND-0.073	ND-0.032	ND-0.08

CONCLUSION

From the forgoing study it is concluded that cold drink of different brands was found to be contaminated with above mentioned pesticides. HCH was found to be dominant contaminant followed by DDT, Dicofol, Endosulfan and chlorpyriphos. Residues of DDT including pp-DDT, op-DDT, pp-DDE, OP-DDE and pp-DDE were found in 50% of the total samples of different brands of cold drink. Pp-DDT and op-DDT were main contaminant. The concentration was found within their safe limit (1µg/L). Residues of dicofol, endosulfan and Chloropyriphos were found in 48%, 60% and 46% of the total cold drink samples of different brands

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