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Original Research Paper

Pesticide Residues Monitoring in Some Agricultural Soil Samples of Taybad, Eastern Iran

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ABSTRACT

The main objective of this study was focused on determining the likely presence of pesticide residues in agricultural soil samples in Taybad district, Khorasan Razavi Province, Iran. Ten agricultural soil samples were taken from different farms were analysed using gas chromatography (GC) method consisting of electron capture detector (ECD) for organochlorine pesticides (OCPs). Seven soil samples were eventually found to be positively polluted by pesticide residues (70 % of the samples) including dieldrin, α-HCH, β-HCH, endosulfan-1, p, p'-DDT, and o,p-DDT. Although endosulfan-1 occurred with the most frequency among the pesticide residues in the soil samples (3 times) and o,p-DDT showed the highest concentration compared to other residues (0.0014 ppm), all the detected pesticide residues were quietly in low concentrations.

INTRODUCTION

Pesticides have been a major environmental issue, attracting both scientific and public concern because of their nature of toxicity, persistence, health problems and endocrine disrupting effects (Al-Wabel et al. 2010). These materials may remain long in the environmental media, and especially in soil their final behaviour depends upon physical, chemical and biological factors, which are often complex and dynamic (Singh 2001). There is a growing environmental concern due to continuous rise in production of pesticides. The agricultural trends over the past 40 years showing global production of pesticides, use of fertilizers and cereal production are shown in Fig. 1 (Tomlin 2002).

Pesticides are able to remain for a long time in the environmental media (soil and water bodies). The physical, chemical and biological factors, which govern the fate of pesticides include sorption-desorption, volatilization, chemical and biological degradation, plant uptake, surface runoff and leaching (Singh 2001). It also depends on the environmental conditions at the time of application. Pesticides may reach the soil through direct application to the soil surface, incorporation in the top few inches of soil, or during application to crops (McEwen & Stephenson 1979). The possible impacts of pesticide residues on soil health include (1) risk of injury to crops and non-target species, (2) development of weed resistance, (3) effect on soil biota and associated processes e.g., nitrogen fixation, (4) incidence and severity of root diseases, and (5) interference in nutrient uptake and utilization by plants (Ferris 1993, McLaughlin et al. 1998).

A research conducted in Burkina Faso indicated that endosulfan was one of the most important pollutants in soil samples collected from cotton farmlands (Norbert et al. 2011). In the Philippines, another study showed that endosulfan, which is restricted in the country and banned in other countries, was found to be the second highest pesticide in concentration in soil samples (Jinky Leilanie 2010). In southern Iran, atrazine residues were found in different soils in Shiraz (Dehghani et al. 2010). In Ethiopia, low concentrations or non-detectable levels of persistent organic pollutants (POPs) such as aldrin, dieldrin, endrin, heptachlor and HCHs were found in few soils (Westbom et al. 2008). A study on agricultural soils in Australia showed the contamination of soil samples by triazine herbicides (Ying et al. 2004).

In Taybad, although agricultural pesticides use is continuously increasing, but no information is available concerning the environmental impacts of those residues in the agricultural soils. Some farmers who work with 2,4-D suffer from neurotic illness, moreover digestive disease and prostate cancer related to 2,4-D have been registered based on the previous studies (Shahsavari et al. 2012, Sierra Club of Canada 2005). 2,4-D causes slight decrease in testosterone release and significant increases in estrogen release from testicular cells (Liu et al. 1996).

MATERIALS AND METHODS

The study area was divided into two half parts including southern and northern parts. Selection of the sampling sites occurred randomly from different agricultural crop fields around Taybad district. Ten soil samples were evenly collected, each of which was a representative of 5 subsamples collected from four corners of the site and one from the approximate center. Soil samples were collected from the 5-30 cm layer from the soil surface, air dried and sieved through a No. 20 brass soil sieve and refrigerated at 4°C until the analysis. Pesticide standards were purchased from Chem Service and soil samples were screened for a total of 18 pesticides (Table 1). The sample codes along with cultivated crops are given in Table 2.

Sample extraction: Fifty g of each soil sample was weighed into a glass jar and added ammonium chloride solution (NH_4Cl) followed by 50 mL distilled acetone. The mixture was shaken for 20 minutes and then, 50 mL of distilled petroleum ether was added and kept in a shaker for overnight. The sample mixture was filtered through Whatman No. 1 filter paper and followed by adding 50 mL of acetone to each soil sample and filtered again. The eluate was collected and concentrated to 1 mL using a rotary flash evaporator. The soil extract was then subjected to additional clean up steps. Activated florisil was placed in the chromatographic column above the glass wool and then added with about 2-4 cm sodium sulphate to absorb any residual moisture from the extract. The column was pre-wetted with 50 mL petroleum ether and was not allowed to dry up. K-D with volumetric or graduated receiving flask was placed under column to receive eluate. The extract was diluted with 25 mL petroleum ether (22.5 mL) and acetone (2.5 mL). The solution was transferred to florisil column, letting it to pass through at about 5 mL/min. Column was eluted at about 5 mL/min with 160 mL petroleum ether mixed with diethyl ether. Florisil eluate was evaporated to near dryness. The final volume was reached to 5 mL with GC grade solvent.

Gas chromatograph: A Shimadzu 14B GC unit, OV 17 column (Japan) consisting of electron capture detector (ECD) and flame photometric detector (FPD) was used to analyse the organochlorine and organophosphorus pesticide residues respectively. All extracts and standards were injected manually. Pure analytical grade pesticide standards were used for GC analysis as reference standard. The injector temperature was 230°C and the detector temperature was 240°C. The temperature program on the capillary column was 220°C. Ultra pure nitrogen gas was used as carrier gas (flow rate 40 mL/min) and zero air and ultra pure hydrogen was used as flame source for FPD detector (flow rate was 60 mL/min). CR-6 chromatographic data processor was used to record the chromatograms and peak areas to calculate the pesticide residues in samples, comparing with the technical standard pesticides.

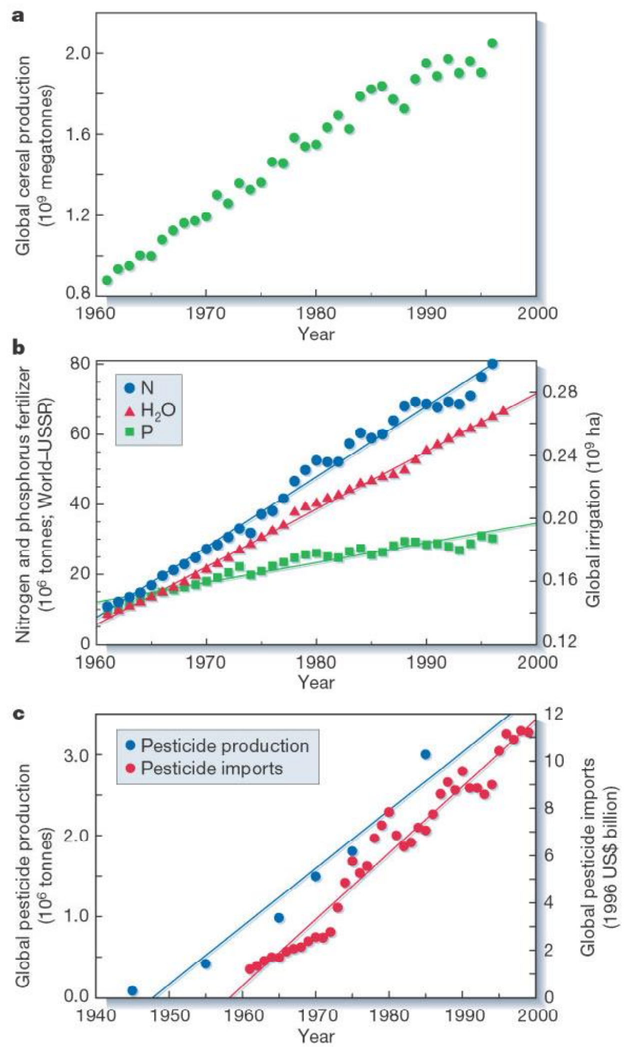

Fig. 1: Agricultural trends over the past 40 years (Tomlin 2002).

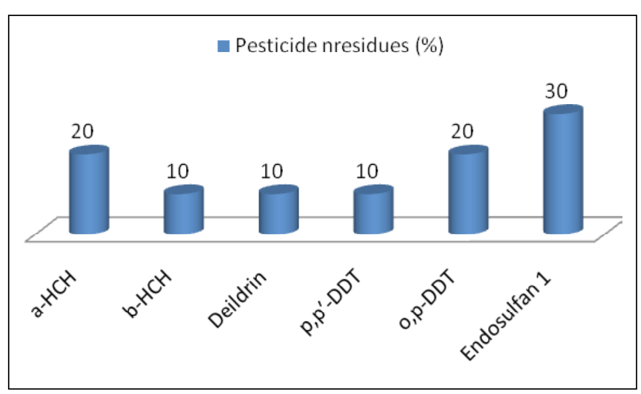

Fig. 2: Frequency of pesticide residues detected in soil samples of Taybad district (%)

Table 1: Standards used for assessing pesticides in soil samples of Taybad district.

Standards	Pesticide(s)
Organochlorine Pesticides	Aldrin, α-HCH, β-HCH, Dieldrin, γ-HCH, 2,4-DDD, 2,4-DDT, 4,4-DDE, 4,4-DDT, Endosulfan-1, Endosulfan-2

Table 2: Sample codes along with cultivated crops in Taybad district.

Sample Code	Cultivated Crop
S1	Cotton
S2	Wheat
S3	Melon
S4	Wheat
S5	Barley
S6	Saffron
S7	Water melon
S8	Sugar beet
S9	Cumin
S10	Barley

Recovery experiment: Recovery experiments were conducted to ascertain the exact quantity of pesticide residues found in soil and water samples. Soil and water samples totally free from pesticide residues were collected and sprayed with the known concentration of pesticide standards in triplicate. The difference between the sprayed concentration and the concentration obtained after standard procedure of extraction and clean up was calculated. The result was expressed as percent recovery and the correction factor was included while calculating and expressing the results of pesticide residues in soil and water samples. The recovery obtained varied from 80 to 85 %.

RESULTS

The results of the analysis of soil samples for pesticide residues are given in Table 3. The frequency of pesticide residues detected in soil samples of Taybad district are shown in Fig. 2. Analysis of soil samples by gas chromatography indicated the presence of organochlorine pesticide residues in seven out of ten soil samples. Out of eighteen injected pesticide standards, six pesticides and their metabolites were detected including α-HCH, β-HCH, dieldrin, endosulfan-1, p,p'-DDT and its breakdown product o,p-DDT. These residues were found in 70 % of the total samples. Multiresidual contaminations occurred in a few soil samples of Taybad district.

The most common pesticide residues detected in the study area was endosulfan-1 isomer, constituting 30 % of the samples followed by o,p-DDT (20 %) and α-HCH (20%). Although, endosulfan-1 exhibited generally the highest frequency of residues in the soil samples, the maximum amount of a single residue detected was found for o,p-DDT (0.0014 ppm) in the soil sample No. 2 collected from south of the district.

Four pesticide residues were found in the samples collected from southern half of the study area including endosulfan-1, α-HCH, p,p'-DDT and o,p-DDT. Southern half of the district showed the highest frequency of pesticides detected with a total 5 contaminated soil samples out of ten samples with maximum concentrations of 0.0014 ppm. It means that all of the soil samples collected from this area have been contaminated by pesticide residues.

Among the soil samples collected from northern half of the district, three samples showed the presence of these residues including endosulfan-1, β-HCH and dieldrin. Multiresidual pesticides occurrence was found in 3 soil samples. The occurrence of other pesticides was quite low. Residues of aldrin, 2,4-DDD, γ-HCH, 4,4-DDE and endosulfan-2 pesticides were not detected in any of the samples.

Table 3: Amount of pesticide residues (ppm) detected in agricultural soil samples of Taybad district.

Sample No.	Endosulfan-1	o, p-DDT	p, p'-DDT	Dieldrin	β-HCH	α-HCH
S1	ND	ND	ND	0.0003	ND	0.0001
S2	0.0010	0.0014	ND	ND	ND	ND
S3	ND	ND	ND	ND	ND	ND
S4	ND	ND	ND	ND	0.0002	ND
S5	0.0003	ND	ND	ND	ND	ND
S6	ND	0.0002	0.0001	ND	ND	ND
S7	ND	ND	ND	ND	ND	0.0001
S8	ND	ND	ND	ND	ND	ND
S9	ND	ND	ND	ND	ND	ND
S10	0.0001	ND	ND	ND	ND	ND

DISCUSSION

The results of the present study showed the occurrence of organochlorine persistent (like p,p'-DDT, endosulfan-1) and moderately persistent pesticide residues (such as dieldrin) at low concentrations in the soil samples collected from growing areas of Taybad district. High occurrence of pesticide residues in southern part of the district might have happened because of the intensive use of agrochemicals in this area. Recent nonagricultural application of p,p'-DDT along with the historical usage of this persistent pesticide may be considered as the other likely reasons for the present detections.

Concentration of organochlorine pesticide residues in the agricultural soil samples of Taybad district identified quite low in comparison with the majority of investigations done in other countries (Al-Wabel et al. 2010, Dehghani et al. 2010, Mo et al. 2008, Li et al. 2008, Westbom et al. 2008, Shegunova et al. 2007).

It should be taken account that there is not a confirmed MRL's list for pesticide residues detected from agricultural soil systems issued by valid national or international authorities.

CONCLUSION

This research work showed that the contamination by pesticide residues in agricultural soil system of Taybad district is not significantly high. Almost all the contaminated samples were lightly polluted by pesticide residues. Obviously, the identified pesticide residues would be harmful when their concentrations increase. The present research project was restricted to the samples collected from a few number of fields in a particular phase of the growing season. More pesticide residue studies are needed in this area in order to understand the actual levels of these residues in different soil systems.~

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