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Imidacloprid residues in vegetables, soil and water in the southern Punjab, Pakistan

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Imidacloprid [IUPAC name 1-(6-chloro-3-pyridylmethyl)-N-nitroimidazolidin-2-ylideneamine] usage in the Southern Punjab, Pakistan was determined its residues in some summer vegetables, ground water and in the soil. Fresh three seasonal vegetables, field soil samples and groundwater samples collected from Southern Punjab were monitored for determining the magnitude of imidacloprid residue in different samples. Imidacloprid residues were detected with the help of HPLC and their separation carried out on a Supelco LC-18 column (250mm × 4.6mm ID). The tested samples of vegetables showed 21 % contamination with imidacloprid residues. Detections was less frequent in pumpkin samples as compare to other summer vegetables, while only 3 samples of groundwater out of 72 were found to be contaminated with it, while 6 samples out of 40 samples of soil were found to be contaminated with imidacloprid residues.

Key words: HPLC, Groundwater, Imidacloprid Residues, Soil and Vegetables

Introduction

In Pakistan, a large number of studies have been carried out on the biological control of pests, In spite of these studies; the pesticides are still common and reliable source for the farmers to control pests. However, their indiscriminate use may likewise lead to many adverse effects, such as, poisoning of the human beings and animals, induction of the pesticide-resistance, bioaccumulation as well as the pesticide residue problems, and the destruction of environment etc. It has been calculated that over million tones of

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pesticides are being used in the world and this trend is increasing with the passage of time (Fao, 2000, Tariq *et al*., 2007). Before conduction of research a face to face survey was conducted for determining the mostly used pesticides in the research area as well on the vegetable crops of area. On the basis of survey-data, imidacloprid was selected, owing to its popularity in the farmer field.

Imidacloprid [IUPAC name 1-(6-chloro-3- pyridylmethyl)-N-nitroimidazolidin-2- lideneamine] is a common pesticide in Pakistan, belonging to a new chemistry regime (chloronicotinyl insecticide). Imidacloprid is firstly introduced by Bayer Agricultural Products(Daraghmeh *et al.,* 2006). Imidacloprid is marketed as an insecticide that also acts, as an early plant growth enhancer and belongs to a relatively new class of insecticide, known as neonicotinoids, which has a high activity against sucking insects. Imidacloprid has the molecular formula $C_9H_{10}ClN_5O_2$ (Figure 1), with a molecular weight of 255.7 g/mol (Table 1). In appearance, it consists of colorless crystals. The insecticide is quite water soluble even at the lowest solubility value reported (510 mg/L, see Table 1; Krohn, 1989, reviewed in Mulye, 1995) and could potentially leach to groundwater (Cohen et al. 1984, cited in Mulye 1995) or be transported in runoff (Mulye, 1995; Scholz *et al.* (1992) found that imidacloprid degradation was more rapid in soils with cover crops than continual availability for uptake by roots (Mullins, 1993). Thus, imidacloprid can persist in soil depending on soil type, pH, use of organic fertilizers, and presence or absence of ground cover. Available literature showed that in Pakistan, no scientific study was conduct for the determination of residues in groundwater, soil and in vegetables .So a study was planned to determination of the imidacloprid residues in residues in groundwater, soil and in vegetables.

Table 1. Imidacloprid Properties

Physical Proprieties	
Molecular weight	255.7
Water solubility	0.61 g/l (20 ºC)
Vapor pressure	1.00 x 10-7 mm Hg (20ºC)
Hydrolysis half-life Aqueous photolysis	>30 days (25oC at pH 7)
half-life	3.98 x10-2 days (24oC at pH 7)
K_{OW} logP	0.57 (21 ºC

Fig. 1 . Map of Study Area.

A number of methods have been employed to measure residues of imidacloprid, e.g., near-infrared spectroscopy (Pigeon, 2000), Photo chemical-fluorimetric method (Vilchez *et al.* 1998, 2001), electrochemical method, enzyme-linked immunosorbent assays (Wantatable, 2001) capillary electrophoresis, 6gas chromatography-mass spectrometry (GC-MS) and high-performance liquid chromatography (HPLC) (Obana *et al.* 2003). Among these methods GC and LC are the most powerful. However, GC cannot be used directly to determine imidacloprid due to the poor volatility, polarity and/or thermal instability of the imidacloprid. In contrast to GC, HPLC is more effective and appropriate for the residual analysis of imidacloprid, and it has been successfully employed for assaying imidacloprid in the soils, water as well as in the vegetables.

Materials and methods

Choice of the Insecticide and Study Area

The universe of this study was Punjab, Pakistan; while the fields work/study area was the four districts of the Southern Punjab. The Punjab consists of 37 districts. The patterns of pesticide-use in the Punjab revealed that they are mostly used; on cotton crop as well as on vegetable. According to the crop-zone classification made by the agriculture department, 11 districts have been included in the cotton-zone. Out of these 11 districts, 4 districts (Layyah, Muzaffargarh, Khenewal and Multan) were selected, at random. Selected

districts have extreme climate. The intense temperature of selected areas in summer may increase beyond 50º C in summer, and can reach at 1ºC in winter These selected districts were further divided into tehsils, sub-tehsils and union councils. Owing to the limitations of financial resources as well as of the time, Firstly, two tehsils were selected from an already selected district, while making sure that they were from two different parts of the same district. From the selected tehsils of each distinct sample were collected.

Before collecting the samples, a face to face survey was conducted for determining the nature and frequency of pesticides currently being used on the vegetable crops. On the basis of survey-data, Imidacloprid was selected, owing to its popularity in the farmer fields and different farms were selected, randomly, from already selected districts for the collection of vegetables and water samples.

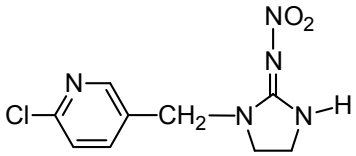

Fig.2. Structure of imidacloprid

Sample Collections and their Extraction

Sampling was carried out, from July 2009 to the last week of August 2009 from, the farmer's fields of four districts in Southern Punjab., i.e., Layyah, D.G. Khan, Muzaffargarh and Multan. Egg plants, okra and pumpkin samples, taken in the ripe stage, were obtained directly from the farmer fields. The site selection, sampling, storage and shipment were done as described by John (2003).

Vegetables have a complex structure, therefore a well plan and sample preparation is required for the isolation of analyltes. Moreover diverse structure of pesticides, increase the importance of "extraction and clean up" procedures, in the pesticide residues analyses, from vegetable samples, which were extracted by following the method of Kadenczki *et al.* (1992) with some modification made by Asi (2005) and other authors. A well-homogenized 50 g vegetable sample was mixed with 20 g of anhydrous sodium sulfate + 0.1 moler Nacl and 50 ml Acetonitrile. The whole mixture was shaken in a GFL shaker (Germany) at high speed for 5 minutes and filtered and then it was transferred onto a glass column, having a 5 ml layer of anhydrous sodium sulfate and 10 g of activated charcoal for bleaching. The mixture was vacuum-filtered through a Whatman no. 6 filter paper. After filtering the solution was shifted into a 250 ml round flask and then, evaporated and concentrated with a rotary evaporator

at 55°C and made a volume upto 10 ml and then, filtered through a 0.45 µm filter, membrane. The collected elute was concentrated just to dryness, under a gentle stream of nitrogen gas and re-dissolved in acetonitrile, volume made upto 500 µl for analyzing with an HPLC.

Due to imidacloprid persistence and mobility, imidacloprid may have a potential to contaminate groundwater. Imidacloprid is currently listed by the DPR as a potential ground water contaminant, based on its high solubility in water, mobility and persistence in soil.

In this experiment, 72 samples were taken from the field, of rural areas, of four districts, while eight samples were taken from the urban areas. All the samples are taken from the ground water with the help of hand pumps, or tap water, which were already present in the field. Samples were selected to determine the Imidacloprid residues with the proposed methods. All the water samples were taken from those sources, which were used for drinking water. Before use, all environmental water samples were filtered through 0.45-µm micropore membranes and stored in a brown glass at low temperature.

Solid phase extraction was a rapid and effective method for the water extraction for the determination of imidacloprid (Baskaran *et al.* 1997). SPE is the most popular current technique for extracting pesticides from water. Early comparisons of SPE with liquid–liquid extraction, showed SPE to be as much as 20% better than accepted traditional extraction procedures (Krynitsky and Lehotay, 2003). Solid phase extraction (SPE) was used for the cleanup of the samples. Each sample was processed, in triplicate, to check reproducibility of results. After filtering 500 mL water sample. It was passed through preconditioned solid phase extraction cartridges (Spulelco), at a flow rate of 5 mL/min. After this, cartridge was washed with 10 mL of distilled water, and the cartridge was dried, with air suction, at a pressure of 80 mmHg, for one hour. In order to check the dryness of pre-weight cartridges, they were weighed again. The target compounds, collected on the cartridges, were eluted successively with 3 mL acetone; 3 mL Methanol and 3 mL ethyl acetate with a negative pressure. The elute was concentrated with help of nitrogen gas and then, re-dissolved in 1 ml of acetonitrile (Asi, 2005, Zahu, 2006). Several advantages were obtained from the use of the Solid phase extraction (SPE) such as a reduced cost, higher sample throughput, low use of solvent and safer to use etc.

Soil works as a filter buffer and has a degradation potential for pesticides having the property storage of pollutant, owing to the presence of organic carbon (Burauel and Bassmann, 2005). It is recognized that the soil is a pathway for the pesticide transportation to water, air and food through a runoff and leaching. Pesticides are also transferred from soil to plants and animal and

ultimately to the human beings. Pesticides, which are very persistent in soil, slowly break down and result as a source of contamination.

Soil samples were collected from the study areas, during September 2008. A total of 40 soil samples from the surface of cotton and vegetable farms, were collected from the agricultural region of the Southern Punjab. The sampling locations evenly covered four districts. For each sample, five subsamples (from the depth of 0-15cm) were collected randomly, from the desired agricultural fields and mixed together to form almost one kg sample; the soil was collected from a layer of 0-15cm using as Oakfield soil auger. After the collection of each soil sample, the soil auger, bucket, sieve and mixing tool were cleaned and then, rinsed with the tap water and dried before the subsequent use. Separate sampling equipment (bucket, soil auger) was used for the control samples. The samples were transferred into polythene bags, transported to the laboratory and were preserved at 4C, till further processing. In the laboratory the samples were dried in the dark, and the leaves, twigs, roots and stones removed, after words. The samples were, and then mixed thoroughly to make a composite sample. After homogenization, the soil samples were sieved, through a 2mm sieve. Representative samples were, however obtained after quartering and coning.

Soil samples were homogenized, passed through a 2mm sieve and 5 g of each transferred into a glass tube. Twenty milliliters of acetonitrile was added and the mixture mixed in a vortex bottle for 1 min. The samples were put into a sonication bath, left for 10 min, and the solvent phase collected. The extraction was repeated twice with 15 mL of acetonitrile. The extracts were then centrifuged, passed through funnels, filled with sodium sulphate, rotary-evaporated to 5mL, and concentrated to 0.5 mL in a nitrogen stream

Chemicals

EthylAcetate (HPLC, Grade,) Methanol (HPLC, Grade), Sodium chloride (HPLC grade) Acetonitrile (Hplc grade), potassium permanganate (GR 99%) were all obtained from Merck, Germany) grade were obtained from Merck. Distal Water was obtained with the help of glass-distill and further purified with the help of a Millipore Milli-Q water purifier. Germany. Analytical standard of imidacloprid (purity 99.9%) was obtained from Dr Ehrenstorfer Ltd (Augsburg, Germany). A standard solution (1mg/mlg/mL) stock was prepared by dissolving imidacloprid standard in acetonitrile and stored in the dark, at 4°C.

Apparatus and HPLC conditions

HPLC conditions were optimized, using a Shimadzu LC-6A apparatus, equipped with UV/vis detector and CR-4A processor. HPLC coupled to UV detection, is particularly suitable for the detection of imidacloprid, due to their strong absorbance between 230 and 270 nm (Martınez *et al.,* 1998). Separation was carried out on a Supelco LC-18 column (250mm× 4.6mm ID, 5µm) (Supelco Park, Bellefonte, USA). Normally, a mixture of water and acetonitrile is commonly used for the HPLC separation of imidacloprid, on a reverse phase column (Fernandez *et al.,* 1996).But this mobile phase, produced a asymmetrical and broad peak. Imidacloprid is more stable in an acidic medium (Zheng and liu, 1999), so, for the experiment. The mobile phase was acetonitrile / 0.01 *M* phosphate buffer (pH 3.0) (25/75 (v/v)). Flow rate was 1 ml/mim at 25 °C temperature.

A linear range of UV response, at 270nm, was observed over the concentration range 0.2–50ml/1imidacloprid.A linear line is developed, which showed the accuracy of the method.

The validity of an analytical method is determined by the agreement between the true value of analyte, in the sample and the value obtained by analysis. Accuracy is usually expressed as the recovery of known, added amounts of analyte by the assay (Francotte *et al.*1996). The average recoveries determined for imidacloprid, at all concentrations, were found between 82 to 92 %, while for the water samples, it was more than 89%.

These values were quite satisfactory and meet the requirements of the European Commission (SANCO/3103/2000), indicating that a method can be considered accurate and precise, when the accuracy of data is between 70 and 110%, with relative standard deviations (RSDs) not higher than 20% The validity of method is checked by the addition of different quantities of imidacloprid in different type of samples (Table-2)

Table 2. Recovery of imidacloprid from spiked materials as determined by HPLC

Adding amount (mg/liter) water	Recovery Percentage (n=3)	RSD	Adding amount (mg/kg) soil	Recovery Percentage (n=3)	RSD	Adding amount (mg/kg) vegetable	Recovery Percentage (n=3)	RSD
.50	90	5	.50	83	7.2	.5	83	7
1.5	85	4.1	1.5	86	3	1.5	89	8.4
2.6	92	3.2	2.6	82	6	3	86	7.3

Results and discussions

The residue concentrations of imidacloprid in vegetables, soil, and water samples are given below.

Vegetables

The concentrations of imidacloprid residues of, in vegetables, are summarized in Table 3. It was observed that 23 samples of vegetables, out of 108 vegetable samples were found to be contaminated with imidacloprid residues. Of them nine samples contained pesticide residues above maximum residues limits (MRLs). The highest average concentration of the pesticide was found to be in the eggplants (.81 mg/kg), followed by (okra .49 mg /kg), and then in (pumpkins.45 mg/kg). On the whole, Pesticides residues were detected in less number in pumpkin samples as compared to other two vegetables. Most probably due to the less number of pesticide sprays on pumpkins as compared to other two vegetables. During the survey, it had also been observed that Pumpkins required less number of pespticide sprays, even in most of the cases, these were being produced without spraying of any pesticide. Market forces such as price of vegetables and its demand have also some impact on the pesticide residue quantity (Parveen *et al.* 2005).As the pumpkins had less price as compared to other two vegetables at that time, so the numbers of pesticides application was also less. 70 percent samples of Pumpkins were free from pesticide residues. It had been observed that 19 % egg plants, had pesticide residues more than the MRLs, while 11 % of Okra samples, had pesticide residues more than the MRLs. A maximum concentration of 1.4 mg/kg of imidacloprid was detected in egg plant. It was also observed that the farmers were unaware of the concept of "with holding period". All the samples, which were brought in the market, after the very next day of pesticide spraying, were contaminated with the pesticide residues.

Sahi *et al. (*2005) stated that the egg plant is fit for the human consumption of even after third day of spraying of imidacloprid, as MRL adopted by different countries for eggplant was ranged from 0.2 to 1 ppm. The potential hazards of these pesticide residues to the human beings, could not be taken severely, as the selected vegetables, were normally, used after trimming, washing, peeling and cooking. It has also been observed that the contaminated samples of okra were obtained, from cotton field, which is common practice in the field.

Table 3. Imidacloprid Residues in Vegetables of Selected Districts

Districts/ Vegetables	Number of Sample Analyzed				Number of Sample contaminated				Average Residues	MRLS
	Layyah	Muzaffargarh	Multan	Khanewal	Layyah	Muzafer Garh	Multan	Khanewal		
Okra(36)	9	9	9	9	2	3	3	3	0.49 mg/kg	.5[a]
Eggplant(32)	9	9	9	9	3	2	2	2	0.81 mg/kg	.5[b]
Pumpkin(36)	9	9	9	9	1	1	1	0	0.45 mg/kg	1[c]

a = Australian MRLs, b = UK MRLs, c = Japanese MRLs d= EU maximum residual limit for individual pesticide

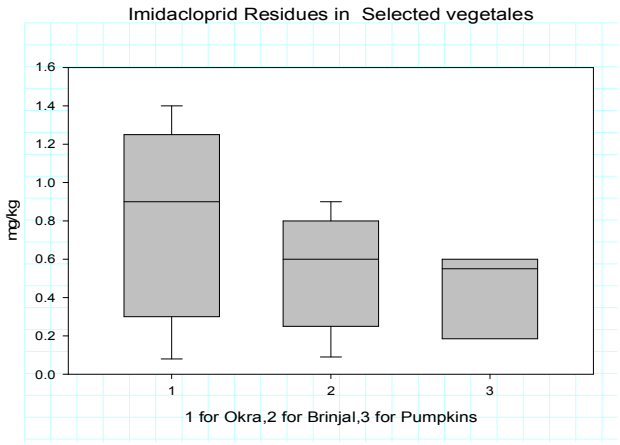

Fig. 3. Occurrence of pesticide residues in the vegetables samples

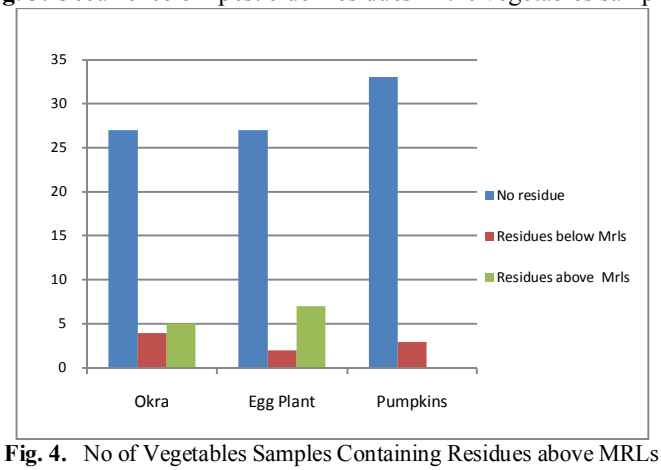

Fig. 4. No of Vegetables Samples Containing Residues above MRLs

Groundwater

In order to determine the residues of imidacloprid, in ground-water, 70 ground-water samples from the hand-pumps, rotary Pumps and tube wells of both rural and peri-urban areas, were collected and analyzed. Only 3 of rural areas samples were seen to be contaminated with imidacloprid residues, but the residue level, was very low. The detection of imidacloprid residues in groundwater were also verified by the study of Mulye 1995. He concluded that imidacloprid could potentially leach to groundwater. Another probable reason for the detection of pesticide, in the groundwater samples from rural areas of Layyah and Muzaffargarh, could be a higher level of the water table because the contaminated samples were collected from those areas of the district which are situated near the river Indus. The water table was reported high in these areas. The results of this study were also verified by Sohamer *et al*. (2006) concluded that the deeper the water table, the less likely the occurrence of pesticides at detectable concentrations. The farmers were also not well aware of best management practices, related to the pesticide usage, mixing and storage.

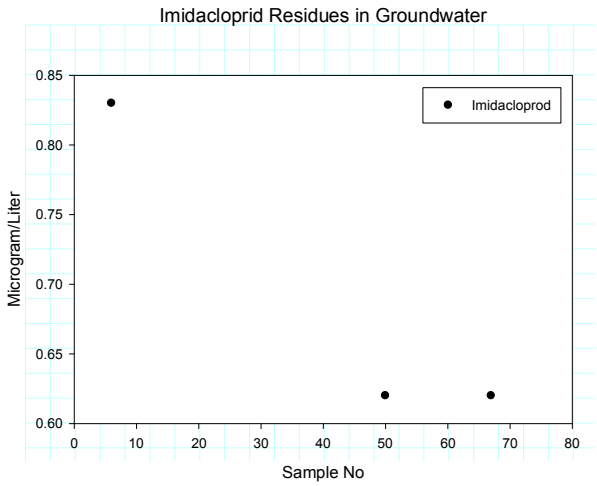

Fig. 5. Imidacloprid Residues in Ground-water

A model based on the daily intake of pesticides in the study district utilizing the average pesticide residues and highest pesticide residues in drinking water as shown in Table 4. The primary concept of this model comes from the study of Dalvie *et al.* (2003). In this model, two conditions were used; a worst case condition, in which the highest pesticide residues, detected in drinking water, was used and another condition, where an average value was

used. These two conditions were used to calculate the total daily intake of pesticide through drinking water and to compare it with the published acceptable daily intakes (ADIs). Before estimating the percentage of ADI were taken by an adult through water consumption. The average intake of pesticide was determining on the assumption that an average person (weighs 60 kg) consumes 2 liters of water, per day. Drinking water intake is thought to pose health risks, if it exceeds 1 to 10% of ADI. On using, a model of acceptable daily intake for the study population, Table 4 shows that the Percentage ADI was not exceeded from 1% to 10%.So it is reasonable to be concluded that the Imidacloprid residues in the water is not of immediate concern. However, it should be noted that the calculations in Table 4 do not take account of vulnerable groups such as children who have a higher consumption of water per kg body weight.

Table 4. Modeling of Acceptable Daily Intake (ADI) for Imidacloprid using Average Pesticide Residues (APR) and Maximum Pesticide Residues (MPR) in selected area for Drinking Water (µg/L)

Sr.No.	Pesticide	ADI	MPR (µg/L)	Daily Intake (µg/kg)	% ADI	APR (µg/L)	Daily intake (µg/kg)	% ADI
1	Imidacloprid	6	0.83	0.0276	0.4611	0.63	0.023	0.3833

Soil

Imidacloprid was also detected the soil. This was due to its higher usage, in the sampled field. 15% (6) of the soil samples were contaminated with the residues of imidacloprid. The residues of imidacloprid were found in soil-samples, of all four selected districts, to be within a range of 0.8 to 2.3 mg/kg. Previously imidacloprid residues were also reported in the different soil samples. Daraghmeh et *al*. (2007) reported that imidacloprid was persistent in soil (decay time for 50% of imidacloprid (DT50) is two years), with a high potential for carry-over and build-up of chemical residues. It was investigated that imidacloprid was used in the contaminated-soil fields within last three months, before the sample collection. It might be a reason of presence of imidacloprid residues in soil, which is verified by Rouchard *et al.* (1994) and Sarkar *et al.* (2001).That the imidacloprid and its major metabolites (6-chloronicotinic acid and CAN) persist in the soil for three months, after its application. Persistence of imidacloprid in soil is affected by various reasons, including, organic matter of the soil, temperature and whether the field is cropped or not. The time required for 50% of the field-applied imidacloprid to

dissipate (DT_{50}) can range anywhere from approximately 80 days to 2 years. Assuming typical DT_{50}s of 1 to 2 years, PMRA has classified imidacloprid as persistent in soil based on the classification scheme of Goring *et al*.(1975)

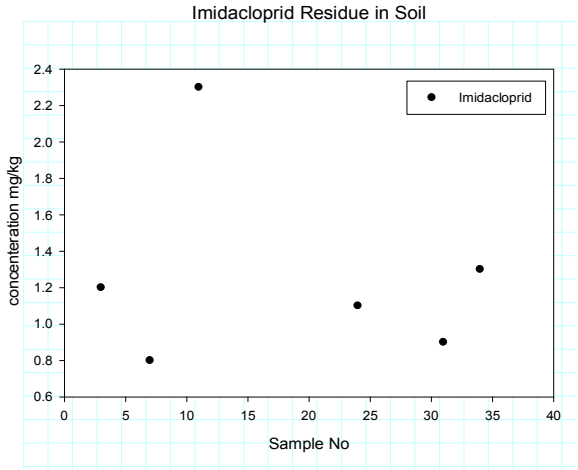

Fig. 6. Imidacloprid Residues in Soil

Conclusion

Qualitative methods were devoted to the finding of imidacloprid in vegetables, soil and groundwater. Imidacloprid residues were found in all three types of samples (Ground water, soil, and vegetables), whether this is due to leaching and/or other groundwater transport processes, or whether this is due to irresponsible insecticide handling practice, is not known. We recommended that pesticide residues, especially currently used pesticides should be monitored, regularly, for determining pesticide residues in primary products as well as in water. We also recommended educating the farmers regarding the pesticide usage, handling and storage and also communicating them the concept of Minimum Waiting Periods and Good Agriculture Practices regarding the pesticide usage.

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