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Multipesticide Residue Assessment of Agricultural Soil and Water in Major Farming Areas in Benguet, Philippines

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Abstract This study investigated the concentration and presence of pesticide residues in water and soil in Benguet, which is a vegetable producing region in the Philippines. Seventy-eight samples and 49 water samples were taken from different farms covering three municipalities in the province of Benguet and were analyzed using gas chromatography. Meteorological conditions of temperature and humidity were also taken. Thirty-four of the soil samples were found to be positive for pesticide residues. The most significant pesticide type with the highest concentration was technical endosulfan, with a mean concentration of 0.025 mg/kg, followed by endosulfan sulfate (0.015 mg/kg), chlorpyrifos (0.01 mg/kg), profenofos (0.003 mg/kg), chlorothanil, cypermethrin, and cylohathrin (all at 0.002 mg/kg). One water sample was found to be positive for pesticide residue of chlorpyrifos in municipality 2 at a concentration of 0.07 mg/L. The data also showed that endosulfan, which is restricted in the Philippines and banned in other countries, was found to be the most prevalent pesticide used (17.7%) and the second highest in concentration (0.015 mg/kg) in soil samples. The study also showed a relationship between temperature and pesticide concentration in soil. In conclusion, pesticide residues were found in soil and water samples in the farming areas of Benguet.

Agriculture represents one-fifth of the total gross domestic product in the Philippines. Crops also comprise almost 50% of the total agricultural output (BAS 2008). Benguet has an altitude of 6,000 ms above sea level and is commonly referred to as the "vegetable and salad bowl of the Philippines." Benguet is the top producer of vegetables grown in the country, consisting of tubers, roots and bulbs, and leafy vegetables, stems, and flowers. Benguet has about 2,600 km^2 of land area, with a population of 372,500. In 2008, 39,000 ha of land was planted with vegetables. In 2008, the productivity yielded was 20 metric tons per hectare, and the total agricultural productivity was 750,000 metric tons.

To boost production, farmers use pesticides. However, there are many deleterious effects of improper and overuse of pesticides. Research has shown that in certain parts of the developing world, pesticide poisoning causes more deaths than infectious diseases due to poor regulation, easy availability of banned or restricted pesticides, and poor information campaign (Eddleston et al. 2002). Many studies on pesticide residues in the Philippines are mainly on agricultural crops. Traces of DDT, which is a restricted pesticide, were found in the residues of soil and rice crops in the country (Rola 1989). In the vegetable industry, traces of malathion residues were found in corn samples, organophosphorus insecticides in green beans, and carbofuran and 3-OH residues in rice plant and its grains (Iman and Rejesus 1977). In another study in Benguet, various banned pesticides were found in certain vegetable samples (Rola 1989). Endonsulfan was found in string beans at 0.06 mg/kg, which was above the maximum level. DDT, another banned pesticide, was found in string beans, eggplants, onions, carrots, sweet peas, pepper, celery, and mustard. Restricted pesticides such as aldrin, dieldren, chlordane, and heptachlor, otherwise used for termite control, were also found in the vegetable samples (Rola 1989).

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In Central Mindanao in the Philippines, chemicals that are banned from all use in agriculture, particularly endosulfan, thiodan, furadan, bionex, DDT, and restricted chemicals such as azodrin, were still being used by farmers to control major pest and diseases of the crops (Saldivar 1996a, b). In a similar study in the same area, endosulfan was used by 88% of the farmers for control of pests in tomato, 88% in baguio beans, 96% in mango, and 96% in pomelo and guava (Saldivar 1996a, b). This study showed that among the samples submitted, tomato and cabbage contained 0.2 and 1.6 mg/kg residue of metamidophos, levels that were categorically higher than the allowable daily intake of 0.0006 mg/kg body weight.

Methods

Study Area and Sampling

The study area consisted of three municipalities: La Trinidad (municipality 1), Bugias (municipality 2), and Atok (municipality 3). Environmental samples of soil and water were taken from the farms through cluster sampling based on the top three producers of vegetables in the province of Benguet. Seventy-eight soil samples and 49 water samples were taken from different farms (Table 1). The farm areas were plotted using geographic mapping and a geographic positioning system specifying the latitude and the longitude.

Sample Collection

Soil and water samples in Benguet were taken in April 2009 during the wet seasons in the Philippines and in the months of June to August. Soil samples were taken from the identified farms. One kilogram of soil was taken from the various plots within the farm to get a representative sample. The various 1-kg soil samples from one farm were mixed together from where the final 1 kg sample of soil was taken for analysis in the laboratory. A soil auger was used to unearthed the soil from the surface to a depth of 1 m. The soil sample was placed in an opaque plastic bag, while the water sample was placed in an opaque container.

Table 1 Distribution of soil and water samples in the municipalities of Benguet, Philippines

Municipality no.	Municipality	No. of soil samples	No. of water samples
1	La Trinidad	16	12
2	Bugias	29	16
3	Atok	33	21

Water samples were taken from various sources such as spring and drinking water within the identified farm locations through the use of surface grab sampling.

Two samples/replicates of the environmental samples were taken from each farm. One field blank was simultaneously collected in each farm together with the samples. All field blanks were analyzed using the same procedure as the environmental samples. Samples were placed in an icebox and delivered to the laboratory within 24 h. The samples were stored in a refrigerator at 5°C.

Meteorological samples were taken using an environmental thermometer (wet bulb globe thermometer) and humidity reading instrument (humidity stick), both manufactured by SKC Inc. The instruments were placed in the center of the farm over a weighted period of 2 h.

The study protocol for collection of environmental samples was approved by the Institutional Review Board of the National Institutes of Health, University of the Philippines, Manila, which is duly accredited by the Forum for Ethical Review Committees in Asia and the Pacific (FERCAP).

Sample Analysis and Quality Control

A standard procedure was used to analyze samples upon receipt (Analytical laboratory Services, BPI 2009). Briefly, pesticide residues were desorbed from the samples and analyzed using gas chromatography (GC) operated in a split mode. Major chromatogram peaks were identified in samples based on a comparison of retention times and mass spectra to peaks from a calibration method. Residues were based on the most commonly used pesticides in Benguet (Lu 2009).

The soil sample underwent three-stage cleanup to remove particulates and impurities in the sample. The first cleanup stage was C18, followed by the use of carbon graphite and, finally, the use of flourisil. The water sample underwent both liquid–liquid extraction and one-phase solid-phase extraction using C18, as water samples are cleaner than soil samples. Gas chromatography (Shimadzu) was used for analysis of multipesticide residues in soil and water. Two detectors were used: nitrogen phosphorous and electron capsule detectors. The elements in the oven program such as the temperature programming, retention time of various pesticides, and temperature of the detector were previously determined, depending on each type of pesticide. During sample transfer, the oven temperature was maintained between 30°C below and 20°C above the solvent's atmospheric boiling point. After the sample had been transferred, the oven temperature was programmed up and chromatography was started. The inlet temperature program consisted of 40°C (4.2 min) and 200°C/min to 320°C (2 min). The oven temperature program included

50°C (6.13 min), 30°C/min to 150°C (2 min), 3°C/min to 205°C (0 min), and 10°C/min to 250°C (20 min).

Spiked calibration standard data were done. Data review was conducted on a single midpoint standard. The midpoint standard was used as a reference to process the remaining five points of the calibration curve. Assessment of all peak assignments, integrations, and calibration curve linearity was done. Analysis of the pesticides exhibited correlation coefficient values of >0.9900.

The recovery was 70–120%. The coefficient of variation was <10%. A blank control matrix was used in the laboratory. Two trials were done for each sample. There was no residue detected at the limit of determination (LOD), which was 0.02 mg/kg for organophosphates and 0.005 mg/kg for organochlorines (OCs) and pyrethroids.

Data were analyzed using descriptive statistics for pesticide residue concentration in samples, and linear regression for relationship between meteorological conditions and pesticide residues. Data were plotted using ArcView, which is a geographic information system (GIS) software for visualizing, managing, creating, and analyzing geographic data. ArcView shows the geographic context of data, allowing the researcher to have a geospatial map of environmental samples gathered. The program used was Arcview GIS 3.2, manufactured by ESRI.

Results

A total of 127 samples was taken, 78 from soil and 49 from water. Of the 78 soil samples, 34 were found to be positive for pesticide residues. Of the 49 water samples, 1 sample was found to be positive for pesticide chlorpyrifos, in municipality 2, at a concentration of 0.07 mg/L.

For the 34 soil samples positive for pesticide residues, the specific pesticide residue types are shown in Fig. 1. The most prevalent types of pesticide in all municipalities were endosulfan sulfate and chlorpyrifos (17.7%), followed by profenofos (8.9%), cyhalothrin (5.1%), cypermethrin (3.8%), technical (t) endosulfan (0.25%), and then chlorothalonil (0.13%). The most prevalent type was chlorpyrifos for municipality 1 (27.4%), endosulfan sulfate for municipality 2 (13.8%), and both endosulfan sulfate and profenofos for municipality 3 (15.6%) (see Fig. 2).

As for pesticide concentration, the most significant pesticide with the highest concentration was t endosulfan, with a mean concentration of 0.025 mg/kg, followed by endosulfan sulfate (0.015 mg/kg), chlorpyrifos (0.01 mg/kg), profenofos (0.003 mg/kg), chlorothanil, cypermethrin, and cylohathrin (all at 0.002 mg/kg) (see Fig. 3). The samples were positive for cyhalothrin (5.1% of soil samples, at a

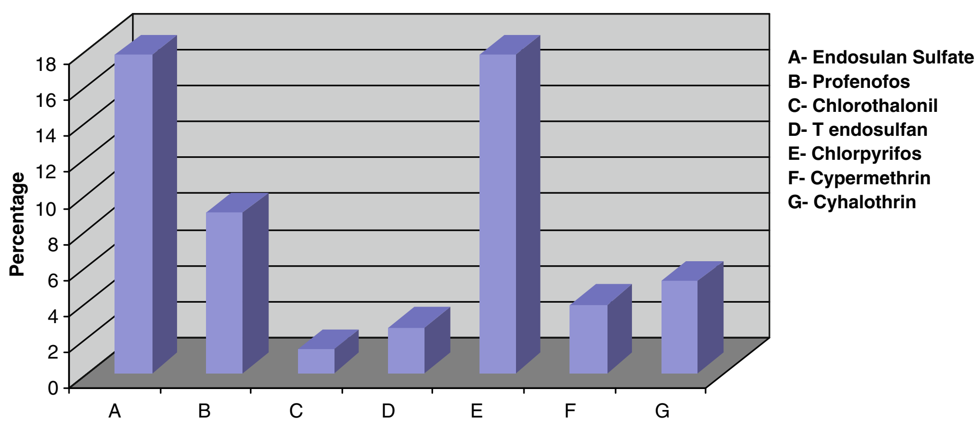

Fig. 1 Pesticide type in soil samples in municipalities of Benguet, Philippines

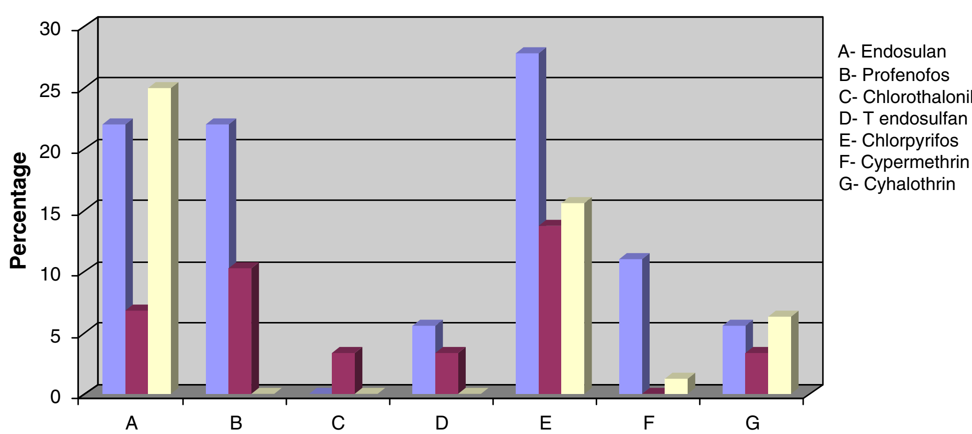

Fig. 2 Distribution of residue per pesticide type in municipalities of Benguet, Philippines

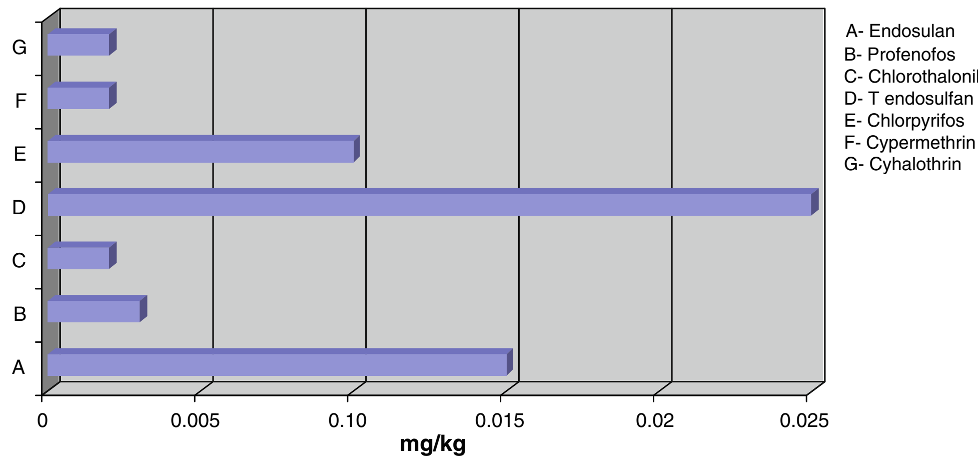

Fig. 3 Concentration of residue per pesticide type in municipalities of Benguet, Philippines

concentration of 0.002 mg/kg) and cypermethrin (3.8%, at 0.002 mg/kg). Both are synthetic pyrethroid insecticides. Lambda cyhalothrin is moderately persistent in soil. It is less adsorbed in sandy soils or those with va ery low organic content. Both these pyrethroids are unlikely to contaminate groundwater by leaching. In our study, chlorpyrifos was found only in soil, and not in water, samples.

Soil samples were positive for chlorothalonil (0.13% of soil samples, at a concentration of 0.002 mg/kg). Chlorothalonil is a chloronitrile which is a broad-spectrum OC fungicide. It has trade names of Rover, Shield, Daconel, and Pillarich. It is degraded more rapidly at higher temperatures and when soil moisture is increased. It has a low water solubility, 0.6 mg/L, at 25°C. There was no water sample with chlorothalonil because of its low water solubility.

Chlorpyrifos, which is an organophosphate, was found in 27.4% of soil samples at 0.01 mg/kg concentration. It was also found in one water sample at 0.07 mg/L, which is above the threshold limit value for both acute and chronic effects on aquatic and marine life.

Temperature and humidity at the farms were measured. There was no statistical association between pesticide residue concentration and humidity in this study. However, an inverse relationship between temperature and pesticide residue concentrations was observed (Fig. 4). Municipality 3, with the lowest temperature, had the highest pesticide concentration. In summary, Table 2 reports the pesticide concentration and fate of residues in relation to pesticide characteristics.

Discussion

Environmental effects of pesticides, specifically organophosphates and OCs, are widely documented such as death of fish alongside rice fields or aquatic systems through soil

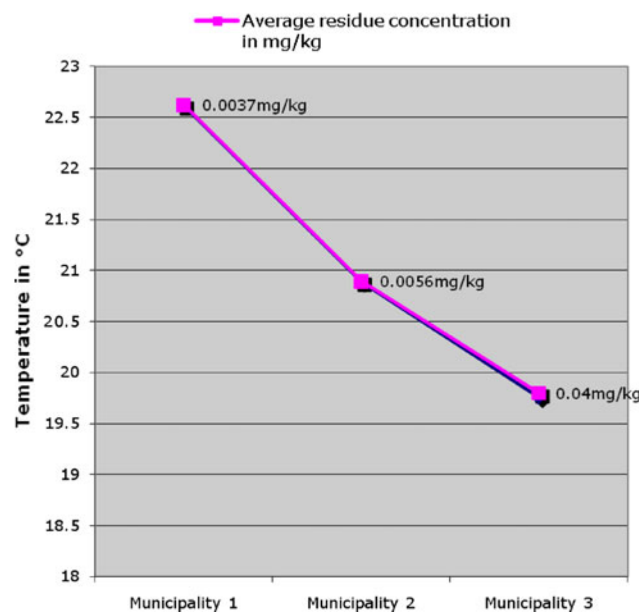

Fig. 4 Relationship between temperature and pesticide residue in soil samples from municipalities of Benguet, Philippines

erosion or water runoff and poisoning or death of farm animals fed contaminated vegetables or grasses (Ishii-Eiteman and Ardhianie 2002). Many studies have also been conducted to determine the health effects of pesticide to humans. In one study, 92% of the farmers complained of health-related problems immediately after applying pesticides (Ishii-Eiteman and Ardhianie 2002). These symptoms included tiredness, weakness, dizziness, nausea, vomiting, blurred vision, rashes, itchy skin, burning sensations in the throat, chest pain, and difficulty breathing. In another study, respiratory complaints such as restrictive respiratory impairments and bronchial obstruction were documented (Rastogi et al. 1989). In Costa Rica, 7% of 100 banana workers exposed to pesticides were found to have skin and eye irritation (Wesseling 2001).

Table 2 Pesticide concentration and fate of residues in relation to pesticide characteristics

Pesticide residues found in soil samples	Characteristics of pesticide	Findings in this study
Endosulfan sulfate	It is degraded by fungi and bacteria from technical (t) endosulfan to endosulfan sulfate	Endosulfan sulfate was found in more soil samples than t endosulfan
	It is not prone to leaching, but transport is likely if it is adsorbed to soil particles in runoff	
	Endosulfan does not easily dissolve in water. It has a water solubility of 0.32 mg/L at 22°C	Endosulfan was not found in water samples
	Its capacity to adhere to soil is moderate. It is absorbed more in soil rich in organic matter compared to sandy soil	The soil type in Benguet is rich in organic matter for vegetation. Endosulfan was the most prevalent type of pesticide found in soil samples
Profenofos	The sorption coefficient of (KoC) is 2000, a high coefficient. A higher sorption coefficient means higher retention in soil and lesser degradation in soil	Profenofos was prevalently found at higher levels, 8.9%, in soil samples compared to other types. It was the third most prevalent pesticide type in soil samples
Chlorothanil	It is degraded more rapidly at higher temperatures and when soil moisture is increased. It is highly adsorbed by silty loam and silty clay-loam soils	Chlorathonil was found in only a few soil samples, at a low concentration, because of its volatility with temperature and its adsorption in silty loam, which is not the soil type in Benguet
T Endosulfan	It has a low water solubility, 0.6 mg/L, at 25°C	This is why no chlorathonil was found in water samples
	It is a regulated pesticide and its use has been restricted	
Chlorpyrifos	Its capacity to adhere to soil is moderate. It is absorbed more in soil rich in organic matter compared to sandy soil	The soil type in Benguet is rich in organic matter for vegetation. The concentration of t endosulfan was the highest of all the pesticides found in soil samples
Cypermethrin	It has a strong adsorption in soil	Together with endosulfan sulfate, chlorpyrifos was the most prevalent pesticide in soil samples and the only pesticide found in water samples
	Cypermethrin is moderately persistent in soil and degrades more rapidly in soils low in organic material	Cypermethrin was found in soil samples at 3.8% at a 0.002 mg/kg concentration
Cyhalothrin	It is not soluble in water and is strongly adsorbed by soil particles (PIP, EXTOXNET 2010)	There was no cypermethrin found in water samples
	This unlikely to contaminates groundwater by leaching	There was no cyhalothrin found in water samples

Neuromuscular function of humans including weakness can also be affected by pesticide. Pesticide applicators were two times more likely to develop reduced muscular strength as compared to the control group (Cole et al. 1998). Pesticides, namely, monocrotophos, chlorpyrifos, profenofos, and acephate, were also found to be inhibitors of acetylcholinesterase (AChE), leading to muscle weakness and paralysis (Das et al. 2006). Moreover, neuroparalysis and myopathy were seen among those with acute organophosphate poisoning (Venkatesh et al. 2006).

Some chronic effects of pesticide cited in the studies by Karlsson (2004) and Lee et al. (2002) included carcinogenic effects, poor reproductive outcomes, neurologic and respiratory disorders, impairments of the immune system, and birth defects. Also, women who reported using pesticides in their homes or yards were two times more likely to have neural tube defect (NTD)-affected pregnancies than women without these reported exposures (Brender et al. 2009).

In this study, OC pesticides, namely, endosulfan, t endosulfan, and chlorothalonil, were found in the soil samples. Other types of OCs were also found in 17 topsoils in China, namely, hexachlorohexanes, dichlorodiphenyltrichloroethane (DDT), and hexachlorobenzene (Wang et al. 2009). Pesticide residues are dependent on pesticide application history and dissipation rates (Harner et al. 1999). As such, source identification analysis in the study in China revealed that all OCs found in soil samples were due to historical use of these chemicals or from other source regions through long- and short-range atmospheric transport. In Benguet, both current and historical use of pesticides accounted for the residues found in the soil samples. Benguet has always engaged in commercial farming.

Bartha et al. (1967) also found that some pesticides have a significant effect in soil. Carbamates, cyclodienes, phenylureas, and thiolcarbamates can depress respiration and nitrification, thus persisting in soils, while amides, anilides, organophosphates, phenylcarbamates, and triazines display toxicity levels.

Other studies have also shown persistence of pesticides in the environment. In the Arctic, levels of selected current use pesticides (CUPs) have been identified and reported in media (air, water, sediment, and biota) since the year 2000. Almost all of the 10 pesticides (chlorothalonil, chlorpyrifos, dacthal, diazinon, dicofol, lindane, methoxychlor, pentachloronitrobenzene, pentachlorophenol, and trifluralin) examined were high-production volume chemicals (Hoferkamp et al. 2009). In a national park in Turkey, 16 different organochlorine pesticides were detected, with more pesticides detected in sediments than in water. The most prevalent pesticides were DDT (69.5% of samples), heptachlor (62.3%), a-endosulfan (55% of samples), and endrin (37%) (Turgut et al. 2009). A high concentration of endosulfan was also found in the study in Benguet. These pesticides could be a long-term contamination source that enters the food web.

The data showed that endosulfan sulfate, commonly known by its trade name Thiodan, was the most prevalent pesticide used (17.7%), and ranked second in pesticide concentration (0.015 mg/kg) in soil samples. This is still lower, however, compared to residue levels of t endosulfan in agricultural soils in the United States, at 1 mg/kg, and Italy, 0.23–3.88 mg/kg (INCHEM 2010).

Endosulfan is included on the list of restricted pesticides by the Fertilizer and Pesticide Authority in the Philippines and should not be used near aquatic resources (FPA, 2009). At the sampling sites, the farms were situated near or alongside river systems, creeks, springs, and streams.

Chlorpyrifos residue is emulsifiable. As such, it was the only residue found in the water sample. There is no threshold level set for pesticides in drinking water but its ambient water criterion for acute effects in freshwater is 0.083 μg/L, and the chronic effect is set at 0.041 μg/L (Hamilton et al. 2003). The actual chlorpyrifos residue in the water sample was 0.07 mg/L, which is above the threshold limit value for both acute and chronic effects on aquatic and marine life.

Samples were positive for cyhalothrin (5.1% of soil samples, at a concentration of 0.002 mg/kg) and cypermethrin (3.8%, at 0.002 mg/kg). Both are synthetic pyrethroid insecticides. Lambda cyhalothrin is moderately persistent in the soil. It is less adsorbed in sandy soils or those with a very low organic content. Both pyrethroids are unlikely to contaminate groundwater by leaching. Meanwhile, cypermethrin is moderately persistent in soil and degrades more rapidly in soils low in organic material under controlled conditions (PIP, EXTOXNET 2010). Since the characteristic soil in Benguet is high in organic matter, t endosulfan and endosulfan sulfate were most prevalent at higher concentrations compared to cypermethrin and cyhalothrin. These pesticides can enter the food web and thus affect humans. In one study, cypermethrin and chlorpyrifos were found in the diet of 12 Atlanta adults over two cycles (2005–2006) (Riederer et al. 2009).

Soil samples were also positive for chlorothalonil (1.3% of soil samples, with a concentration of 0.002 mg/kg). Chlorothalonil is a chloronitrile which is a broad-spectrum OC fungicide. It has trade names of Rover, Shield, Daconel, and Pillarich. It is degraded more rapidly at higher temperatures and with greater soil moisture (PIP, EXTOXNET 2010). None of the water samples was found to contain chlorothalonil because of its low water solubility.

Soil characteristics may also increase the degree of absorption of pesticides in soil. Pesticide concentrations were found to be highest in temperate mountain soils that were rich in organic matter and received large amounts of cold precipitation (Daly et al. 2007). The samples taken in Benguet had a soil type rich in organic matter.

The processes of adsorption, diffusion, and hydrolysis also tend to influence the behavior of pesticide residues in water sediment systems (Katagi 2006). A study in China showed that there is a higher affinity of OC compounds in sediments than in water because these organic hydrophobic pollutants tend to stay more in sediments (Zhang et al. 2003). In our study, the OCs, consisting of endosulfan sulfate and t endosulfan, were the most prevalent pesticides in soil samples.

The study showed that there was an inverse relationship between temperature and pesticide residue concentrations. Aulagnier and Poissant (2005) found that pesticide residues in air were dependent on temperature.

This is the first comprehensive environmental monitoring of soil and water in the province of Benguet. Background surveillance is a critical public health tool for control of pesticide poisoning. The research showed that endosulfan, which is restricted in the Philippines and banned in many other developed countries, is still being used by farmers. As such, the data gathered in this study can serve as a basis for pesticide regulation. Exposures occur not only on farms, but also in environmental media such as water and soil systems, as found in a study where children were exposed to pesticides from residues found in soil and water (Tolosana et al. 2009). Environmental monitoring is thus warranted. However, health investigation of farmers should be correlated with the presence and concentration of pesticides in the environment.

This study did not distinguish between historical and current use of pesticides. There are pesticides identified in the study that may not be currently used by farmers but have remained in the environment because of their persistence and long half-life.

Conclusion and Recommendations

This study has shown that pesticide residues were found in soil and water samples collected from the top producers of vegetables in Benguet. The most prevalent pesticides in all municipalities were endosulfan sulfate, chlorpyrifos, and profenofos. The highest pesticide residue concentration included total endosulfan, endosulfan sulfate, and chlorpyrifos. The persistence of pesticides that are restricted in the country shows weak regulatory mechanisms.

Mere reporting of pesticides by farmers may not be very accurate or sufficient. The culture of monitoring based on report writing must change to one of surveillance that leads to intervention, although the latter deals with greater resource allocation (London and Bailie 2001). The effort to increase awareness of pesticide poisoning and health effects on humans can be complemented by evidence of surveillance of residues in the environment.

The monitoring done in this research showed active ingredients of pesticides used in the area. There are specific symptoms associated with each type of pesticide. Thus, information on actual pesticides used and those that remain in the environment can assist medical personnel in their toxicological assessment of patients.

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