DETECTION OF ORGANOCHLORINE COMPOUND IN PUYU, SEPAT AND HARUAN FISH CAUGHT FROM IRRIGATION CANALS IN PADDY FIELDS IN SELANGOR AND PERAK, MALAYSIA

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Abstract

A case study was conducted to investigate the concentration of organochlorine pesticide residues in fish samples collected from irrigation canals in paddy fields in Tanjong Karang and Sekinchan, Selangor and Seberang Perak, Perak, Malaysia. The samples comprised of three fish species, namely puyu (*Anabas testudineus),* haruan (*Channa striatus*) and sepat (*Tricogaster pectoralis).* Samples were chopped and homogenized and later digested and extracted using QuEChERS method and characterised for organochlorine compounds using gas chromatography. Seven organochlorine compounds, namely aldrin, dieldrin, endrin, endosulfan, o,p-DDT, o,p-DDD and p,p-DDT were identified and quantified. The water samples taken from the irrigation canals were analysed only for endosulfan at 0.04 mg/kg, which exceeded the maximum residue level for endosulfan pesticide residue for water sewage by Environment Protection Administration of the Republic of China on Taiwan (2000) at 0.03 mg/kg. However, the concentrations of endosulfan in all fish samples were below the Codex Alimentary Maximum Residue Limit of 0.2 mg/kg for meat.

Keywords: organochlorine pesticide residues, gas chromatography, paddy-field fish, maximum residue limit

INTRODUCTION

Paddy cultivation is one of the major agricultural activities in Malaysia, mainly concentrated in the major granary areas in Kedah, Kelantan, Perak and Selangor which are served with a system of water ways including irrigation canals, drains, rivers and dams. A variety of fish can be found to inhabit these water ways and these fish contribute a common protein source, apart from marine fish, chicken, meat and eggs, to the general populace (Chen *et al*., 1984). Among the common fish caught from irrigation canals in paddy fields are puyu (*Anabas testudineus*), haruan (*Channa striatus*) and sepat (*Tricogaster pectoralis*).

Pesticides have been used extensively in paddy planting activities in Malaysia and many parts of the world to control the infestation of major pests such as brown plant hoppers (*Nilaparvata lugens*) and golden apple snails (*Pomacea canaliculata*). Among the common pesticides used are those belonging to the two major pesticide categories: organochlorine and organophosphate - extremely hazardous Class I and II chemicals -

which are either banned or severely restricted for use in some countries even when used with high levels of protection. The use of organochlorines in farming activities has caused concern due to their effect on human health and aquatic ecosystem, particularly so in view of their persistent and bioaccumulative properties (George and Frear, 1966). These chemicals have also been implicated in a broad range of adverse human health effects including reproductive failures, birth defects, immune system malfunction, endocrine disruptions, and cancers (Garabrant *et al*., 1992)

The residue levels of pesticides found in paddy field fish were moderately high, considering the short exposure period during the fish growing process. These levels generally do not cause toxic effects to the fish. However the residue can accumulate to alarming levels in human subjects, particularly when contaminated fish are consumed frequently and in great quantities (Chen *et al*., 1984). There is little scientific research been carried out to address the safety of use of these pesticides by paddy farmers. This study was conducted to investigate the concentrations of organochlorine compound residues in sepat (*Tricogaster pectoralis*), haruan (*Anabas testudineus*) and puyu (*Channa striatus*) fish from paddy fields in Sekinchan, Tanjong Karang and Seberang Perak.

MATERIALS AND METHODS

Fish sampling

Fish samples used in the present study were caught live from irrigation canals in paddy fields in three rice growing areas, namely Tanjong Karang and Sekinchan in Selangor and Seberang Perak in Perak, Malaysia during the month of December 2011. The fish were of three common species: sepat (*Tricogaster pectoralis*), puyu (*Anabas testudineus*) and haruan (*Channa striatus*). The samples comprised of 5 fish of each species (*Tricogaster pectoralis, Anabas testudineus and Channa striatus*) caught in Tanjong Karang, 3 fish each of *Channa striatus, Tricogaster pectoralis* and *Anabas testudineus* obtained from Seberang Perak and Sekinchan. The fish samples were kept in thermal insulator boxes and transported to the MARDI Central Laboratory at Serdang, Selangor, Malaysia the following day after collection.

Sample storage

Upon arrival at the laboratory the samples were washed several times with deionized water to remove sediments and other entangled materials. Each fish sample was given an individual identification code. Fish samples were wrapped in aluminum foil and stored at 4°C prior to sample preparation.

Sample preparation

Haruan samples from Tanjong Karang were thawed at room temperature (25°C) for an hour, cut and blended as whole samples to include skin, internal organs and head. Other puyu, sepat and haruan samples from Sekinchan and Seberang Perak were blended and homogenised separately according to internal organs and fillet.

Detection of pesticide residue

The extraction method used in this study was QuEChERS extraction which involved single-step buffered acetonitrile (MeCN) and salting out liquid-liquid partitioning from the water in the sample with $MgSO_4$. Dispersive solid phase extraction clean-up was done to remove organic acids and excess water from the samples. Then the extracts were analyzed by mass spectrometry after a chromatographic analytical separation. Organochlorine compound residue analysis was done for endosulfan, aldrin, o,p-DDT, p,p-DDT, o,p-DDD, dieldrin and endrin. A well-chopped 15-g fish sample along with 1 mL 1% acetic acid (HOAc) in MeCN and 0.5 g anhydrous $MgSO_4$ per g sample were centrifuged for 5 minutes followed by the addition of a portion of the MeCN extract to anhydrous $MgSO_4$ sorbent, and then mixed and recentrifuged. This final extract was transferred to auto-sampler vials for analysis by gas chromatography (Agilent 7890N) and mass spectrometry to determine the concentration of the specific organochlorine residues.

RESULTS AND DISCUSSION

Pesticide residues in water of irrigation canals in paddy fields

Table 1 shows the organochlorine compound residue concentration in water samples taken from irrigation canals in Tanjong Karang and Sekinchan paddy fields. These samples were taken to ascertain the pesticide residue status in water from the sampling areas. The water samples were analyzed for organochlorine compounds, mainly endosulfan, aldrin, dieldrin, o,p-DDT, o,p-DDT, and p,p-DDT. The residue levels of organochlorine compound in the water samples taken from Tanjong Karang and Sekinchan indicated endosulfan was present at a concentration of 0.04 mg/kg. According to Environment Protection Administration of the Republic of China on Taiwan (2000), endosulfan pesticide residue levels in sewage water should not exceed 0.03 mg/kg. The present study showed that the endosulfan pesticide residue exceed the minimum level, which is something of concern. This result showed that endosulfan was still being used in the paddy fields in the two sampled areas. Although organochlorine endosulfan pesticide has been banned since 2008 in many countries including Malaysia for use in crop cultivation, it is still being used because of availability in the Malaysia market.

Pesticide residues in fish samples

Tables 2 and 3 show the levels of organochlorine compounds in puyu, haruan and sepat fish samples from Tanjong Karang, Sekinchan and Seberang Perak. Fish sampled from these three areas had pesticide residue concentration of 0.02 mg/kg or less. This concentration is below the Codex Alimentary minimum residue level for meat. The organochlorine residual analysis used in the present study has been programmed to detect residue concentration of 0.02 mg/kg or less. Thus concentration of less than 0.02 mg/kg was indicated as non-traceable. The concentration of organochlorine compound residue of less than 0.02 mg/kg was considered to be very low and the fish samples were not free of organochlorine compound residue since the concentration of organochlorine compound residue in the water of irrigation canals from the same areas was 0.04 mg/kg. The residue detection method adopted by the MARDI Laboratory was meant to

differentiate samples exceeding 0.02 mg/kg in organochlorine compound residue from those with less than 0.02 mg/kg for gross classification of samples.

Table 1. Organochlorine (OC) pesticide residue in water samples from irrigation canals in Tanjung Karang and Sekinchan paddy fields

Area	OC residue (mg/kg)
Tanjong Karang	0.04
Sekinchan	0.04

Table 2. Organochlorine compound residues in whole samples of three fish species caught in irrigation canals in paddy fields in Tanjung Karang

Fish species	No. of samples detected above BDL[1]	BDL[1](mg/kg)
Sepat	Nil	<0.02
Puyu	Nil	<0.02
Haruan	Nil	<0.02

[1]Below detection limit

Table 3. Organochlorine compound residues of different parts of fish sampled from irrigation canals in paddy fields in Seberang Perak (S.Perak) and Sekinchan areas

Fish species	Samples Type	Sampling area	No. of samples detected above BDL[1]	BDL[1] (mg/kg)
Haruan	Organ	S. Perak	Nil	<0.02
		Sekinchan	Nil	<0.02
	Meat	S. Perak	Nil	<0.02
		Sekinchan	Nil	<0.02
Sepat	Meat	Sekinchan	Nil	<0.02
Puyu	Meat	Sekinchan	Nil	<0.02

[1]Below detection limit

Pesticide application was carried out at several stages in the paddy cultivation process and many paddy farmers applied “cocktail pesticide”, a combination of pesticides made up of endosulfan, organophosphate chemicals and cypermethrine. In this “cocktail pesticide”, the endosulfan concentration was low. This explained the low endosulfan compound residue level in the water samples. However, because endosulfan is a hydrophobic compound, it might be higher in the soil than in water. The low endosulfan concentration in water samples from irrigation canals would lead to low exposure of this organochlorine pesticide to the fish. Thus, this situation has led to low

bioaccumulation of pesticide residues in the fish. This study shows that there is a need to determine biometabolism of endosulfan in future researches.

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