Thailand

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1. Introduction

Organochlorine pesticides were used widely from the 1940s to the 1960s for agricultural pest control and for malaria programs. Since the 1960s their use has been reduced greatly due to their health and environmental effects and persistence. Fish and shellfish accumulate pesticides from the environment in which they live in, but the extent of accumulation depends on such factors as geographical location, fish species feeding patterns, solubility and lipophilicity of the pesticides. These pesticides may accumulate in fish at levels that can cause illness. Therefore the maximum levels for some organochlorine pesticides in fish products are set in many countries. To manage the safety of fish and fish products, the residues level should be known.

2. Objectives And Goals

To set up the laboratory analysis method for organochlorine pesticides and obtain an understanding of levels of organochlorine pesticides in fish and fish products.

3. Survey Methodologies

a. Sampling Method, Location, Species, Number of Samples and Sampling Size

Frozen vannamei shrimp (*Penaeus vannamei*), frozen nile tilapia (*Tilapia nilotica*) and dried yellowstripe trevally (*Selaroides leptolepis*) from the factories in Samutsakorn province were collected. After sampling, samples were packed in plastic bag and sent to laboratory. The samples if not analyzed immediately were kept at –18°C.

b. Method of Analysis

About 300 g of the edible part of the sample was blended and extracted according to the method as attached in Appendix 1 at Samutsakorn Fish Inspection and Research Center and injected into Hewlett Packard Gas Chromatograph-Electron Capture Detector (GC-ECD) at the Central Laboratory (Thailand) Co. Ltd.

c. Limit of Detection and Limit of Quantification

Limit of Detection is 0.01 ppm.
Limit of Quantification is 0.05 ppm.

d. National Regulatory Limits

The regulatory limits of each organochlorine pesticides depend on products and importing countries. For Thailand's regulatory limits of pesticide residues in fish and fish products:

- Aldrin, Dieldrin, Heptachlor - 0.02 ppm
- Chlordane, Endrin - 0.05 ppm
- DDT - 1 ppm

4. Results And Discussion

a. Participation of Inter-laboratory Proficiency Testing and Results

Thailand did not participate in any Inter-Laboratory Proficiency Testing Program.

b. Survey Results and Discussion

Nineteen samples of frozen vannamei shrimp (*Penaeus vannamei*), 18 samples of frozen nile

tilapia (*Tilapia nilotica*) and 11 samples of dried yellowstripe trevally (*Selaroides leptolepis*) were collected from the factories in Samutsakorn province. The samples were analysed for the following pesticide residues and no pesticide residues was detected in any samples.

S/N	Compounds	Chemical Name
1	α-HCH	alpha-Hexachlorocyclohexane
2	γ-HCH	gamma-Hexachlorocyclohexane
3	δ-HCH	delta-Hexachlorocyclohexane
4	HCB	Hexachlorobenzene
5	Heptachlor	Heptachlor
6	2,4'-DDE	ortho-para- Dichlorodiphenyldichloroethylene
7	4,4'-DDE	para-para- Dichlorodiphenyldichloroethylene
8	2,4'-DDD	ortho-para-Dichlorodiphenyldichloroethane
9	4,4'-DDD	para-para-Dichlorodiphenyldichloroethane
10	2,4'-DDT	ortho-para-Dichlorodiphenyltrichloroethane
11	4,4'-DDT	para-para-Dichlorodiphenyltrichloroethane
12	Aldrin	Aldrin
13	Dieldrin	Dieldrin
14	Endrin	Endrin

Year of analysis & Sampling location	Analyte	Fish sample analysed		No. of samples analysed	Min. value of results (ppm) – wet weight basis	Max. value of result (ppm) – wet weight basis	Average value of result (ppm) – wet weight basis	Average Recovery (%)
		Common name	Scientific name					
2008 Samutsakorn province	Aldrin	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	85
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	91
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	46
	4,4′-DDE	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	87
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	93
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	48
	2,4′-DDE	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	80
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	89
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	46
	4,4′-DDD	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	88
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	101
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	50
	2,4′-DDD	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	90
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	97
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	50
	4,4′-DDT	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	69
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	88
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	49

Year of analysis & Sampling location	Analyte	Fish sample analysed		No. of samples analysed	Min. value of results (ppm) – wet weight basis	Max. value of result (ppm) – wet weight basis	Average value of result (ppm) – wet weight basis	Average Recovery (%)
		Common name	Scientific name					
2008 Samutsakorn province	2,4′-DDT	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	74
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	86
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	48
	Dieldrin	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	88
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	94
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	50
	Endrin	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	108
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	126
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	75
	α-HCH	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	81
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	94
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	51
	γ-HCH	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	78
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	91
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	49
	δ-HCH	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	88
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	99
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	51

Year of analysis & Sampling location	Analyte	Fish sample analysed		No. of samples analysed	Min. value of results (ppm) – wet weight basis	Max. value of result (ppm) – wet weight basis	Average value of result (ppm) – wet weight basis	Average Recovery (%)
		Common name	Scientific name					
2008 Samutsakorn province	Heptachlor	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	81
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	96
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	51
	HCB	Frozen vannamei shrimp	*Penaeus vannamei*	19	Not detected	Not detected	Not detected	78
		Frozen nile tilapia	*Tilapia nilotica*	18	Not detected	Not detected	Not detected	91
		Dried yellowstripe trevally	*Selaroides leptolepis*	11	Not detected	Not detected	Not detected	49

a. Corrective Actions

No correction action was taken as no pesticide residues were detected. In cases of exceeding the importing country regulation, that lot of product will be rejected for export. The processor will be required to identify any other products that were produced from the same lot of raw material and provide a corrective action plan. A follow-up at plant may be deemed necessary in some cases. If the processor could not bring its system to comply with the safety requirement, the processor will be suspended for export. The processor will be withdrawn from the Department of Fisheries list of approved fishery establishment when the offence is repeated.

5. Problems and Challenges Encountered

Equipment

The existing GC-ECD in the laboratory is old and less sensitive. Hence, the laboratory had to use the GC-ECD at Central Laboratory (Thailand) Co. Ltd. for the analysis. This resulted in the high expenditure.

Method

The method taught in the regional training course uses a lot of solvent, has many steps and takes a long time to complete the extraction. So another method was adopted in order to reduce extraction time and solvent usage.

This method gave good recovery for testing of fresh samples. The average recovery is 89%. For dried samples, however, the recovery was poor at 51% on average. This method thus needs to be improved further.

6. Recommendations and Suggestions for Future Follow up Action

- Organophosphate should be surveyed as there is little problem with organochlorine pesticide residues now due to the ban of organochlorine pesticides for agriculture use in Thailand.
- The fund should be given at the beginning of the year for easier planning and conducting of activities.

Method of Analysis

Weigh 2 g sample in 50 ml polypropylene screw cap centrifuge tube
↓
Add about 2 spoonful of sodium sulfate
↓
Mix with stirring rod
↓
Add 20 ml petroleum ether
↓
Homogenize for 1 min
↓
Decant petroleum ether extracted through about 2 spoonful of sodium sulfate
(which was placed on whatman no. 1 filter paper in funnel) into evaporation flask
↓
Extract another 3 times by using 10 ml petroleum ether
↓
Evaporate with vacuum rotary evaporator at room temperature until nearly dry
↓
Add about 1ml petroleum ether
↓
Load into Florisil SPE
(which was pre-conditioned with 5ml petroleum ether)
↓
Elute with 10 ml of 6% diethyl ether in petroleum ether
Follow by 10 ml of 15% diethyl ether in petroleum ether
↓
Evaporate with vacuum rotary evaporator at room temperature
↓
Dissolve the residues with 2 ml n-hexane
↓
Inject into GC-ECD

Calculation

Organochlorine pesticides (ppm) = C x 2 / W where,
C = concentration of organochlorine pesticides from calibration curve (ug/ml)
W = sample weight (g)

Reference

Official Methods of Analysis of AOAC International 17th edition 2000. Chapter 10. Pesticide and industrial chemical residues. 10.2.01. AOAC Official method 983.21. p. 12E-13.