MALAYSIA

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1. Introduction

This study was divided into two components of the Japanese Trust Fund (JTF) IV and JTF II. JTF IV was carried out from the year 2005 to 2006, under Component I Activity CI-1: Regional survey of heavy metals in fish and fish product and in the environment, The survey was divided into two areas namely species identification of marine, freshwater and brackish aquacultured fish and marine and freshwater captured fish. Marine, freshwater and brackish aquacultured fish, cockles, green mussel and black tiger prawn. Marine and freshwater captured fish samples were Spanish mackerel, grouper, red snapper, prawns and squid.

However, in 2007, the program previously conducted under JTF IV was moved to JTF II, which excluded aquaculture products. The emphasis was on Component I, Activity CI-1: Regional survey of heavy metals in fish and fish products. The survey focused on fish samples collected from processing establishments.

The survey provided information on the level of heavy metals concentration in seafood consumed by people whether it is for domestic use and/or for export. The survey carried out complemented with our on-going Sanitary and Phytosanitary Monitoring Program (SPS), which has the overall objective to ensure safe fish and fish products for human consumption.

Presently, fish and fish products from Malaysia exported to Europe constitute about 30% of the fish production. The Department of Fisheries (DOF) as one of the Competent Authority (CA) should ensure that fish and fish product exported to EU complies with the requirements of importing country and the European Commision (EC) Regulation 1881/2006 –

setting maximum levels for certain contaminants in foodstuffs.

2. Objective and Goals

The objective of this study is to determine the level of heavy metals namely Cadmium (Cd), Lead (Pb) and total Mercury (Hg). All the data generated will be deposited collected into the Fish and Fish Products Safety Information Network.

3. Survey Methodologies

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

From 10 to 13 May 2005, 160 samples were collected throughout Johor. The samples were collected from east and west Johor especially from Tg. Sedili and Pontian. Spanish mackerel (Scomberomorus sp.), grouper (Epinephelus sp.), red snapper (Lutjanus sp.), shrimp (Metapeneaus sp.), cultured white shrimp (Peneaus vannamei), squid (Loligo sp.) and seabass (Lates calcarifer) were collected from the east and west coast of Johor, while catfish (Clarias batrachus) and tilapia (Oreochromis niloticus) were collected from Kota Tinggi. Green mussel (Perna viridis) were harvested only from aquaculture farms in Masai and Pantai Lido, covering an area from the west and east coasts of Johor. The black tiger prawn (Peneaus monodon) were sampled from Desaru of east coast of Johor and cockles (Anadara granosa) were collected only from Pontian, Johor. The second sampling was carried out from 4 to 8 September 2006, of which 152 samples were collected from the same venues as in the earlier sampling.

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Eight samples per species were randomly taken from each location of sampling plan. All the samples were stored at -18°C in a cold room for four days before being transported to the laboratory for analysis.

In the laboratory, the length and weight of the samples were measured.

From 5 to 6 June 2007, 72 samples of fish products were collected from processing establishments in the states of Perak and Penang. The second sampling was carried out on 28 August 2007, of which 32 samples were collected from other processing establishments in Perak.

Samples comprised of fishcakes, fishballs, frozen shrimps (*P.vannamei*), surimi kintokidai A (from big eye snapper), surimi (from threadfin bream), frozen octopus (*Octopus doflein*), frozen squid (*Sepia inermis*), frozen black tiger prawn (*P. monodon*) and frozen cuttlefish.

Eight samples per species were randomly taken from each location of sampling plan. The samples were kept in cooler boxes filled with ice to maintain the temperature before being transported to the laboratory on the same day.

b. Method of Analysis

Sample Preparation

Frozen samples were thawed at room temperature. Each sample was homogenized using the Seward stomacher 80 Biomaster at 300 rpm for 120 seconds. The homogenized sample was formed into a composite pie and 20g sample was stored in a 50 ml plastic bottle and kept in a freezer for further analysis.

Detection of Heavy Metals

i) Detection of Total Mercury (Hg)

Initially, one layer of additive M was applied onto the surface of a sample boat and about 20-40 mg of sample was placed onto the middle of sample boat before being covered by additive M. This was followed by a layer of additive B and covered again with additive M (Method 2: for a solid or powder sample). The sample was applied to the MA 2000 instrument for detection of total mercury content.

The specifications of MA 2000 instrument are presented in Table 1.

Table 1. The specifications of MA 2000 for total mercury analysis.

Item	Description
1. Method	Gold-amalgam collection
	method with thermal
	decomposition by a ceramic
	tube heater
2. Carrier Gas	Purified dry air
3. Detection Method	Non-dispersion double-
	beam cold atomic absorption
	spectrometry
4. Detection Limit	0.002 ng (weight of sample)
5. Measuring Range	0 – 1,000 ng
6. Wavelength	253.7 nm

ii) Detection of Cadmium (Cd) and Lead (Pb)

Digestion of samples

About 400 mg of sample was prepared into HF100 teflon liner. 4 ml nitric acid (HNO₃) (Merck, Damstard, Germany, 65%) and 2 ml of hydrogen peroxide (H₂O₂) (Merck, Damstard, Germany, 30%) was added into the liner and digested using the USEPA procedure of 3015, 3051 and 3052 wet digestion method (PAAR001H). The digestion was performed using Anton Paar 3000, 16 rotors HF100 teflon vessels at a frequency of 2.45 GHz at 1500 W under operating pressure at 40 bars (580 psi) and maximum temperature of 240°C for 45 minutes. A p/T-sensor was used to monitor the digestion reaction till completion. The sample was topped up to 25 ml prior analysis.

Detection of Lead (Pb) and Cadmium (Cd)Using Atomic Absorption Spectrometry method

Lead (Pb) and cadmium (Cd) were measured by Atomic Absorption Spectrometry (AAS) Perkin Elmer model AA800. The diluted samples were analyzed using an in-house furnace method that was developed by Perkin Elmer, PE TS04. The analytical conditions for the AAS are presented in Table 2.

Analysis was performed under room temperature. All the containers and the apparatus were placed in 10% purified acid nitric.

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Table 2. Analytical conditions for AA800.

Item	Description
Argon gas	
- Flow rate	0.9 – 1.5 liters/min
- Operating pressure	50 psig
Air compressor	
- Flow rate	10-28 liters/min
- Operating pressure	60 psig
Lamp/current	Cd & Pb – Hollow cathode lamp/12 mA
Wavelength	Cd – 228.8 nm
	Pb – 283.3 nm

c. Limit of Detection and Limit of Quantification

Element	LOD (mg/kg)	LOQ (mg/kg)
	Year 2005 & 2	2006
Cd	0.0039	0.039
Pb	0.0018	0.018
Total Hg	0.00002	0.0002
	Year 2007	7
Cd	0.0048	0.048
Pb	0.0006	0.006
Total Hg	0.00002	0.0002

d. National Regulatory Limits

Country	Element	Regulatory Limits (mg/kg)
	Cd	1.0
Malaysia	Pb	2.0
Ivialaysia	Hg	0.5
		(1.0 for predator)
	Cd	0.05
EC Dec		(excluding fish species in 3.2.6 & 3.2.7)*
EC. Reg. 1881/2006	Pb	0.3
1001/2000	Hg	0.5
		(excluding fish species in 3.3.2)*

* Refer to Appendix 1

4. Results and Discussion

Year of Participation	Program Name	Analyte Tested	Reported Results (ppb)	True Value (ppb)	z- Score	Remarks
	ASEAN-	Total As	10.1740	10.20	-0.018	_
2004	SEAFDEC Regional Inter-	Total Pb	0.5066	0.319	0.197	_
	Laboratory Proficiency	Total Hg	3.1299	3.37	-0.234	_
Laboratory Proficiency TestingTotalTotal			18.6340	19.40	-0.509	_
	FAPAS	Total As	780.16	1124	-1.900	_
2007	Proficiency Test 0786 Metallic	Cd	55.50	52.4	0.3	_
2007	Contaminants in	Pb	-	-	_	z-score not issued
	canned Fish	Total Hg	597.05	704	-0.9	_

a. Participation in Inter-laboratory Proficiency Testing and Results

b. Survey Results and Discussion

Physical Parameters of Samples Collected in 2005 & 2006

Table 3. Physical parameters of samples collected from east coast of Johor.

Type of system/fish	Location	Weight (g) Average (range)	Length (cm) Average (range)
Aquaculture		Twerage (range)	Average (range)
Catfish	Kota Tinggi/Desaru	380 ± 39.5 (343 - 458)	$40.3 \pm 1.9 \\ (36.5 - 42.1)$
Mussel	Masai/Pasir Gudang	$45.3 \pm 13.4 (22 - 85.4)$	$8.9 \pm 1.0 \\ (7.2 - 40)$
Seabass	Tg. Sedili	450.8 ± 61.0 (319 - 578)	$30.5 \pm 2.3 \\ (24.1 - 33.5)$
Tilapia	Kota Tinggi/Desaru	$491.3 \pm 19.7 (478 - 536)$	$22.2 \pm 2.0 \\ (20.5 - 26.7)$
Tiger prawn	Desaru	27.8 ± 7.6 (17.5 - 38)	N.A*
Shrimp	Desaru	27.5 ± 5.0 (17 - 36)	N.A
Marine captured			
Grouper	Tg. Sedili/Mersing	$206.5 \pm 206.3 (55 - 706)$	$21.7 \pm 6.0 \\ (15.2 - 35.7)$
Red snapper	Tg. Sedili	240.9 ± 276.9 (47 - 903)	$20.8 \pm 8.7 \\ (13 - 39.5)$
Squid	Tg. Sedili/Mersing	97.7 ± 33.5 (57 - 175)	21.5 ± 2.8 (18 - 26)
Spanish Mackerel	Tg. Sedili/Mersing	$ \begin{array}{c} 1131.5 \pm 147.2 \\ (1013 - 1407) \end{array} $	$ \begin{array}{r} 49.5 \pm 1.7 \\ (48 - 52) \end{array} $
Shrimp	Tg. Sedili	$ \begin{array}{r} 14.6 \pm 0.6 \\ (13.6 - 15.4) \end{array} $	$ \begin{array}{c} 4.8 \pm 1.2 \\ (3.3 - 6.5) \end{array} $

*N.A – Not Available

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Table 4. Physical parameters of samples collected from west coast of Johor.

Type of system/fish		Weight (g)	Length (cm)
Aquaculture	Location	Average (range)	Average (range)
Catfish	Senai	$258.6 \pm 205.3 (98 - 767)$	$29.6 \pm 6.3 \\ (22 - 44.2)$
Mussel	Pantai Lido	37.6 ± 26.5 (14.8 - 124)	$7.8 \pm 0.8 \\ (5.6 - 9.0)$
Seabass	Pendas	$\begin{array}{c} 409.9 \pm 96.7 \\ (191 - 535) \end{array}$	$29.2 \pm 2.5 (23 - 32.4)$
Tilapia	Senai	$\begin{array}{c} 454.1 \pm 42.5 \\ (408 - 526) \end{array}$	$21.1 \pm 0.6 \\ (20.5 - 22.4)$
Cockle	Pontian	$7.5 \pm 1.3 \\ (5.9 - 9.5)$	$2.7 \pm 0.2 \\ (2.4 - 3.3)$
Shrimp	Desaru	$11.4 \pm 1.6 \\ (8.1 - 13.9)$	N.A*
Marine captured		,	
Grouper	Pontian	$696 \pm 395.5 (390 - 1509)$	$35.3 \pm 5.9 \\ (30.5 - 46.2)$
Red snapper	Pontian	$895.6 \pm 449.1 (330 - 1608)$	40 ± 4.8 (32 - 47.2)
Squid	Pontian	$105.9 \pm 55.2 \\ (55 - 260)$	$29.1 \pm 12.3 \\ (14 - 54)$
Spanish Mackerel	Pontian	$1131.5 \pm 147.2 \\ (1013 - 1407)$	$ \begin{array}{c} 16.5 \pm 6.3 \\ (11.4 - 31.2) \end{array} $
Shrimp	Pantai Lido	$14.6 \pm 0.6 \\ (13.6 - 15.4)$	N.A*

* N.A – Not Available

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						Min. value of	Max. value of	Average value of		
Type of svstem/fish	Sampling		Fish sample Analysed	Analysed	No. of Samules	results (ppm)	results (ppm)	results (ppm)	Average Recovery (%)	Remarks
	Location	Analyte	Common name	Scientific name		– wet weight basis	– wet weight basis	– wet weight basis		
Aquacultured										
Catfish	Kota Tinggi /	Cd	Ikan keli	Clarias batrachus	16	ND	0.03	0.01		
	Desaru	Pb				0.04	0.88	0.16		
		Hg			,	0.005	0.04	0.02		
Green mussel	Masai / Pasir	Cd	Kupang	Perna viridis	16	0.04	0.12	0.07		
	Gudang	Pb			,	0.01	0.32	0.09	Cd = 97.6	
		Hg				0.005	0.02	0.01	Pb = 102.8	
Seabass	Tg. Sedili	Cd	Siakap	Lates calcarifer	16	ND	0.02	0.01	Hg = 99.8	
		Pb				ND	0.52	0.19		
		Hg				0.01	0.18	0.12		
Tilapia	Kota Tinggi /	Cd	Tilapia	Oreochromis	16	ND	0.02	0.02		
	Desaru	Pb		niloticus		ND	0.18	0.10		
		Hg				0.01	0.02	0.01		

Table 5. Results of Cd, Pb and Hg in marine/freshwater/brackishwater aquacultured fish and marine captured fish from east coast of Johor in 2005 and 2006.

Analytical Results For Heavy Metals for Samples collected in 2005 and 2006

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Note : ND – less than LOD.

Location ed besaru b Desaru Tg. Sedili/ Mersing	alyte	Common	Fish sample Analysed	No. of	value of results (ppm)	value of results (ppm)	value of results (ppm)	Average	Remarks
ltured ser Desaru urimp Desaru d Fish Mersing		name	Scientific name	Samples	– wet weight basis	– wet weight basis	– wet weight basis	vecovery (20)	
ser urimp Desaru d Fish Mersing		-			-				
urimp Desaru d Fish Tg. Sedili/ Mersing		Udang	Penaeus	16	ND	0.07	0.01		
rimp Desaru d Fish Mersing		harimau	monodon		ND	0.40	0.16		
d Fish Mersing					0.02	0.03	0.02		
d Fish Tg. Sedili/ Mersing		Udang	Penaeus	8	0.01	0.10	0.04		
d Fish Tg. Sedili/ Mersing		putih	vannamei		ND	0.18	0.09		
d Fish Tg. Sedili/ Mersing					0.02	0.03	0.02	Cd = 97.6	
Tg. Sedili/ Mersing								$F_{0} = 102.0$ Hg = 99.8	
Mersing		Kerapu	Epinephelus sp.	16	ND	0.06	0.02	0	
					0.01	0.14	0.05		
					0.05	0.27	0.14		
Red snapper Tg. Sedili Cd		Ikan	Lutjanus sp.	16	ND	0.01	0.01		
Pb		merah			ND	0.27	0.08		
Hg					0.02	0.18	0.07		

Table 5. Results of Cd, Pb and Hg in marine/freshwater/brackishwater aquacultured fish and marine captured fish from east coast of Johor in 2005 and 2006 (cont'd).

						Min. value of	Max. value of	Average value of		
Type of svstem/fish	Sampling		Fish sample Analysed	e Analysed	No. of Samules	results (ppm)	results (ppm)	results (ppm)	Average Recovery (%)	Remarks
	Location	Analyte	Common name	Scientific name		– wet weight basis	– wet weight basis	– wet weight basis		
Captured Fish										
Squid	Tg. Sedili/	Cd	Sotong	Loligo sp.	16	0.02	0.21	0.10		
	Mersing	Ъb		,		ND	0.31	0.06		
		Hg		,		0.01	0.03	0.02		
Spanish	Tg. Sedili/	Cd	Tenggiri	Scomberomorus	16	ND	0.01	0.01	Cd = 97.6	
mackerel	Mersing	Pb		sp.		ND	0.15	0.06	Pb = 102.8	
		Hg		<u>.</u>		0.04	0.22	0.12	Hg = 99.8	
Shrimp	Tg. Sedili	Cd	Udang	Metapeneaus sp.	8	ND	0.04	0.02		
		Ъb	laut			ND	0.14	0.05		
		Hg		<u>.</u>		0.02	0.26	0.06		
			Total nu	Total number of samples =	160					
Note : ND – less than LOD.	LOD.									

Table 5. Results of Cd, Pb and Hg in marine/freshwater/brackishwater aquacultured fish and marine captured fish from east coast of Johor in 2005 and 2006 (cont'd).

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Type of svetem/fish	Sampling		Fish sample Analysed	Analysed	No. of Samules	Min. value of results (ppm)	Max. value of results	Average value of results (ppm)	Average Rocovery (%)	Remarks
	Location	Analyte	Common name	Scientific name		– wet weight basis	(ppm) – wet weight basis	– wet weight basis		
Aquacultured		_		-	-					
Catfish	Senai	Cd	Ikan keli	Clarias batrachus	16	ND	0.06	0.01		
		Pb			1	ND	0.95	0.24		
		Hg			1	0.004	0.05	0.02		
Green mussel	Pantai Lido	Cd	Kupang	Perna veridis	16	0.01	0.18	0.03		
		Pb				0.01	0.24	0.09	Cd = 97.6	
		Hg			1	0.005	0.03	0.02	Pb = 102.8	
Seabass	Pendas	Cd	Siakap	Lates calcarifer	16	ND	0.06	0.02	Hg = 99.8	
		Pb				ND	0.43	0.11		
		Hg				0.04	0.11	0.07		
Tilapia	Senai	Cd	Tilapia	Oreochromis	8	ND	ND	ND		
		Pb		niloticus		ND	0.18	0.15		
		Hg				0.005	0.01	0.01		
Note : ND – less than LOD.	LOD.									

Table 6. Results of Cd, Pb and Hg in marine/freshwater/brackishwater aquacultured fish and marine captured fish from west coast of Johor in 2005 and 2006.

LocationAnalyte pCommonScientific namewet basisweightwet weightweightwet weightrdPNameCommonScientific nameNoNoNoNoNordPPKerangAnadara granosa160.060.810.330.02rhPPValuePND0.010.050.010.020.01rhPUdangPenaeus vannamei8ND0.020.010.020.01rhPPPND0.020.010.020.010.020.01rhPPPND0.020.010.020.010.020.01rhPPPND0.020.010.020.010.020.01rhPPPND0.020.010.020.010.020.01rhPPPPPPPPPPrhPPPPPPPPPPrhPPPPPPPPPPrhPPPPPPPPPPrhPPPPPPPPPPrhPPPPPPPPPPrh	Type of svetem/fish	Sampling		Fish sample Analysed	Analysed	No. of Samules	Min. value of results (ppm)	Max. value of results	Average value of results (ppm)	Average Recovery (%)	Remarks
ededCdKerangAnadara granosa160.060.810.33PbPbPbPb0.090.580.28PPbPbPbPc0.010.000.02PCdUdangPenaeus vannamei8ND0.020.01PPbPutihPenaeus vannamei8ND0.020.01PPontianCdVdangPenaeus vannamei8ND0.020.01PPbPutihPenaeus vannamei8ND0.020.010.02IshCdKerapuEpinephelus sp.8ND0.020.010.03PontianCdKerapuEpinephelus sp.8ND0.020.010.03IshPontianCdIkanLutjanus sp.16ND0.010.030.03PontianCdIkanLutjanus sp.16ND0.010.050.05PontianPhPutihPutihPutihPutih0.040.010.05PontianPhPutihPutihPutihPutihPutihPutihPutihPontianPhPutihPutihPutihPutihPutihPutihPutihPontianPhPutihPutihPutihPutihPutihPutihPutihPutihPontianPhPutihPutihPutihPutihPutihPutih<		Location	Analyte	Common name	Scientific name	2	– wet weight basis	(ppm) – wet weight basis	– wet weight basis		
$ \left \begin{array}{ccccc} \text{Pottian} & \text{Cd} & \text{Kerang} & Indaca granosa & I6 & 0.06 & 0.81 & 0.33 \\ \hline \text{Pb} & \text{Pb} & \text{Pb} & 0.09 & 0.58 & 0.28 \\ \hline \text{Hg} & \text{Cd} & \text{Udang} & \text{Penaeus vanuanei} & 8 & \text{ND} & 0.06 & 0.02 \\ \hline \text{Pb} & \text{Putih} & \text{Penaeus vanuanei} & 8 & \text{ND} & 0.09 & 0.05 \\ \hline \text{Hg} & \text{Pottian} & 0.01 & 0.004 & 0.03 & 0.05 \\ \hline \text{Ish} & \text{Pottian} & \text{Cd} & \text{Verapu} & \text{Penaeus vanuanei} & 8 & \text{ND} & 0.09 & 0.05 \\ \hline \text{Hg} & \text{Pottian} & \text{Cd} & \text{Verapu} & \text{Penaeus vanuanei} & 8 & \text{ND} & 0.09 & 0.05 \\ \hline \text{Ish} & \text{Pottian} & \text{Penaeus vanuanei} & 8 & \text{ND} & 0.09 & 0.05 \\ \hline \text{Pottian} & \text{Cd} & \text{Kerapu} & \text{Epinephelus sp.} & 8 & \text{ND} & 0.03 & 0.02 \\ \hline \text{Pottian} & \text{Pottian} & \text{Cd} & \text{Kerapu} & \text{Epinephelus sp.} & 8 & \text{ND} & 0.07 & 0.03 \\ \hline \text{Pottian} & \text{Pottian} & \text{Cd} & \text{Rapu} & \text{Lujanus sp.} & 8 & \text{ND} & 0.07 & 0.03 & 0.03 \\ \hline \text{Pottian} & \text{Cd} & \text{Rapu} & \text{Lujanus sp.} & 16 & \text{ND} & 0.01 & 0.005 \\ \hline \text{Pottian} & \text{Cd} & \text{Reabu} & \text{Lujanus sp.} & 16 & \text{ND} & 0.01 & 0.05 \\ \hline \text{Pottian} & \text{Cd} & \text{Reabu} & \text{Lujanus sp.} & 16 & \text{ND} & 0.01 & 0.005 \\ \hline \text{Pottian} & \text{Pottian} & \text{Lujanus sp.} & 16 & \text{ND} & 0.01 & 0.01 & 0.05 \\ \hline \text{Pottian} & \text{Hg} & \text{Reabu} & \text{Lujanus sp.} & 16 & \text{ND} & 0.01 & 0.01 & 0.05 \\ \hline \text{Pottian} & \text{Hg} & \text{Pottian} & 0.04 & 0.02 & 0.01 & 0.05 \\ \hline \end{array} \right $	Aquacultured										
$ \left \begin{array}{c c c c c c c c c c c c c c c c c c c $	Cockles	Pontian	Cd	Kerang		16	0.06	0.81	0.33		
			Pb				0.09	0.58	0.28		
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$			Hg				0.01	0.06	0.02		
$ \left(\begin{array}{c c c c c c c c c c c c c c c c c c c $	White shrimp	Pontian	Cd	Udang	Penaeus vannamei	8	ND	0.02	0.01		
HgHg0.040.030.02ish I I I I I I I PontianCdKerapuEpinephelus sp.8ND I I PontianCdKerapuEpinephelus sp.8ND I I I PontianCdKerapuEpinephelus sp.8ND I I I PontianCdKerapuEpinephelus sp.8ND I I I PontianCdKanuLutjanus sp.16 I I I I I PontianCdIkanLutjanus sp.16 I I I I I I I HgHgHg I			Pb	putih			ND	0.09	0.05		
			Hg				0.004	0.03	0.02	Cd = 97.6	
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	Captured Fish									FU = 102.0 Hg = 99.8	
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	Grouper	Pontian	Cd	Kerapu	Epinephelus sp.	8	ND	0.02	0.01	0	
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			Pb				ND	0.07	0.03		
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $			Hg				0.05	0.28	0.13		
merah ND 0.12 0.04 0.32	Red snapper	Pontian	Cd	Ikan		16	ND	0.01	0.005		
0.04 0.32			Pb	merah			ND	0.12	0.06		
			Hg				0.04	0.32	0.12		

Table 6. Results of Cd, Pb and Hg in marine/freshwater/brackishwater aquacultured fish and marine captured fish from west coast of Johor in 2005 and 2006 (cont'd).

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Type of	Complete		L'ah comu		No. of	Min. value of results	Max. value of	Average value of results	Average	Domodia
svstem/fish	Sampung		risn sample Analysed	e Analyseu	Samples	(mdd)	results	(mdd)	Recovery (%)	Kemarks
	Location	Analyte	Common name	Scientific name		– wet weight basis	(ppm) – wet weight basis	– wet weight basis		
Captured Fish										
Squid	Pontian	Cd	Sotong	Loligo sp.	16	0.03	0.30	0.08		
		Pb				ND	0.33	0.07		
		Hg				0.02	0.07	0.05		
Spanish	Pontian	Cd	Tenggiri	Scomberomorus	16	ND	0.08	0.02	Cd = 97.6	
mackerel		Pb		sp.		ND	0.74	0.11	Pb = 102.8	
		Hg				0.02	0.14	0.05	Hg = 99.8	
Shrimp	Pontian	Cd	Udang	Metapeneaus sp.	16	ND	0.03	0.01		
		Pb	laut			ND	0.23	0.08		
		Hg				0.01	0.04	0.02		
			Total nu	Total number of samples =	152					
Note : ND – less than LOD.	LOD.									

Table 6. Results of Cd, Pb and Hg in marine/freshwater/brackishwater aquacultured fish and marine captured fish from west coast of Johor in 2005 and 2006 (cont'd).

Regional Survey of Heavy Metals in Fish and Fish Products (2004-2008)

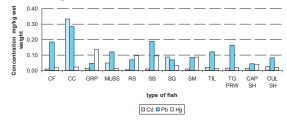
30

Discussion

Mean Concentration of Heavy Metals in various fishes in Johor

The mean concentration of Cadmium (Cd), Lead (Pb) and Mercury (Hg) of all the species of fish, green mussel and prawn in Johor was much lower than the standard of Commission Regulation $(EC)^1$ No. 1881/2006; Food Act of Malaysia² (1983) (amendment, 1993) and Malaysia Food Regulation (1985).

Graph 1. Mean Concentrations of Cd, Pb and Hg in various species of fish in Johor.



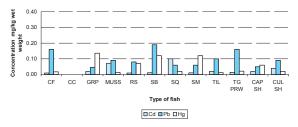
CF - Catfish; CC – Cockles; GRP – Grouper; MUSS – Mussel; RS – Red Snapper; SB – Seabass; SQ – Squid; SM – Spanish Mackerel; TIL – Tilapia; TG PRW – Tiger Prawn; CAP SH – Captured Shrimp; CUL SH – Cultured Shrimp

Graph 1 shows the mean concentration of Cd and Pb were less than 0.20 ppm except for cockles. The highest concentrations of Cd and Pb were found in cockles collected from Pontian, west coast of Johor in 2005, with a concentration of 0.81 ppm for Cd and 0.58 ppm for Pb. The mean concentration of Hg for all samples was less than 0.20 ppm. The highest concentration of Hg was detected in red snapper from Pontian, west coast of Johor in 2005 at a concentration of 0.32 ppm.

Mean Concentration of Heavy Metals in various fishes from east coast of Johor

The mean concentration of Cadmium, Lead and Mercury of all species of fish, green mussel and prawn from east coast of Johor was also lower than the regulatory standards.

Graph 2. Mean Concentrations of Cd, Pb and Hg from east coast of Johor.



CF - Catfish; CC – Cockles; GRP – Grouper; MUSS – Mussel; RS – Red Snapper; SB – Seabass; SQ – Squid; SM – Spanish Mackerel; TIL – Tilapia; TG PRW – Tiger Prawn; CAP SH – Captured Shrimp; CUL SH – Cultured Shrimp

Regional Survey of Heavy Metals in Fish and Fish Products (2004-2008)

Marine, brackish water and freshwater Aquacultured Fish

Graph 2 shows the mean concentrations of Cd, Pb and Hg in various species of aquacultured fish from east coast of Johor were less than 0.20 ppm. The concentration of Cd was found to be highest in mussel from Masai at 0.12 ppm in 2005. For Pb, it was found that catfish from Kota Tinggi in 2005 had the highest concentration at 0.88 ppm. The highest concentration of Hg was observed in seabass from Tg. Sedili in 2005 at 0.18 ppm. Details are shown in Table 5.

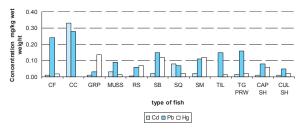
This site is considered to be not suitable for cockles culture due to high water current and soil profile (sandy area), therefore there is no reported data on cockle from this area.

Marine Captured Fish

Graph 2 also shows the mean concentrations of Cd, Pb and Hg in various species of marine captured fish from east coast of Johor were less than 0.20 ppm. The concentration of Cd was found to be highest in squid from Mersing at 0.21 ppm in 2006. In addition, the concentration of Pb was found in squid from Tg. Sedili in 2005 at 0.31 ppm. The highest concentration of Hg was observed in grouper from Pontian in 2005 at 0.27 ppm. Details are shown in Table 5.

Mean concentration of heavy metals in various fishes from west coast of Johor

Graph 3. Mean Concentrations of Cd, Pb and Hg from west coast of Johor.



CF - Catfish; CC – Cockles; GRP – Grouper; MUSS – Mussel; RS – Red Snapper; SB - Seabass; SQ – Squid; SM – Spanish Mackerel; TIL – Tilapia; TG PRW – Tiger Prawn; CAP SH – Captured Shrimp; CUL SH – Cultured Shrimp

Marine, brackish water, freshwater Aquacultured Fish

Graph 3 shows the mean concentrations of Cd, Pb and Hg in various species of aquacultured fish from west coast of Johor were lower than 0.20 ppm except for Cd and Pb in cockles and also for Pb in catfish. The highest concentrations of Cd and Pb in cockles were found from Air Baloi, Pontian at 0.81 ppm and 0.58 ppm respectively. The highest concentration of Cd from cockles collected in Desaru showed lower concentration at 0.19 ppm as compared to those collected in Pontian. Meanwhile, the concentration of Pb in catfish was found to be highest in Senai at 0.95 ppm. The concentration of Hg was observed at 0.11 ppm. Details are shown in Table 6.

Marine Captured Fish

Graph 3 shows the mean concentrations of Cd, Pb and Hg in various species of marine captured fish

from west coast of Johor were also lower than 0.20 ppm. The highest concentration of Cd was found in squid collected from Pontian in 2005 at 0.30 ppm. The concentration of Pb was found to be highest in Spanish Mackerel from Pontian at 0.74 ppm. In addition, the highest concentration of Hg was found in red snapper collected from Pontian at 0.32 ppm. Details are shown in Table 6.

Analytical Results For Heavy Metals for Samples collected in 2007

Table 7. Mean concentrations of Cd, Pb and Hg in the various fish products.

Fish Product	Analyte	No. of Samples	Min. value of results (ppm) – wet weight basis	Max. value of results (ppm) – wet weight basis	Average value of results (ppm) – wet weight basis	Average Recovery (%)	Remarks
Fishcake	Cd	16	ND	0.02	0.008		
TISHCake	Pb	- 10	ND	0.02	0.000	-	
	Hg	_	0.018	0.024	0.02	-	
Fishball	Cd	16	ND	ND	ND	-	
1 Ionoun	Pb		ND	0.25	0.05	-	
	Hg	_	0.01	0.03	0.02	-	
Frozen shrimps	Cd	8	ND	ND	ND	-	
(Penaeus vannamei)	Pb	-	ND	0.11	0.06	-	
	Hg	_	0.01	0.02	0.01	-	
Surimi Kintokidai A	Cd	16	ND	0.08	0.08	-	
(big eye snapper)	Pb	-	ND	0.38*	0.32		
	Hg	-	0.02	0.05	0.03	-	
Surimi (threadfin	Cd	8	ND	0.005	0.005	-	
bream)	Pb	-	ND	0.06	0.04	-	
	Hg		0.03	0.04	0.03		
Frozen Octopus	Cd	8	0.03	0.47	0.18	Cd = 99.5	
(Octopus dofleini)	Pb		0.01	0.44	0.18	Pb = 101.1	ND for 14
	Hg		0.01	0.02	0.02	Hg =100.4	samples
Frozen squid	Cd	8	0.12	0.45	0.26		
(Loligo sp.)	Pb		ND	0.64	0.31		
	Hg		0.007	0.02	0.01		
Frozen black tiger	Cd	8	0.002	0.03	0.01		
prawn	Pb	1	ND	ND	ND	1	
(Penaeus monodon)	Hg	1	0.01	0.02	0.02		
Frozen cuttlefish	Cd	8	0.01	0.07	0.04		
(Sepia inermis)	Pb	1	ND	ND	ND	1	
	Hg	1	0.007	0.01	0.01	1	
Total number of	-	96					

Note : ND - less than LOD., * reanalyzed

Table 8. Mean concentrations of Cd, Pb and Hg in the various fish products from Hutan Melintang, Perak.

Fish Product	Analyte	No. of Samples	Min. value of results (ppm) – wet weight basis	Max. value of results (ppm) – wet weight basis	Average value of results (ppm) – wet weight basis	Average Recovery (%)	Remarks
Fishcake	Cd	8	0.004	0.01	0.007		
	Pb		ND	0.02	0.07		
	Hg		0.019	0.02	0.02		
Fishball	Cd	8	ND	ND	ND		
	Pb		ND	0.07	0.05		
	Hg		0.017	0.03	0.02		
Surimi (Kintokidai A;	Cd	8	ND	ND	ND		
big eye snapper)	Pb		ND	ND	ND		
	Hg		0.03	0.04	0.03		
Frozen shrimp	Cd	8	ND	ND	ND		
(Penaeus vannamei)	Pb		ND	0.06	0.06		
	Hg		0.01	0.02	0.01		
Frozen Octopus	Cd	8	0.03	0.47	0.18		
(Octopus dofleini)	Pb		0.01	0.44	0.18	-Cd = 99.5	
	Hg		0.01	0.02	0.02	- Pb =101.1 - Hg =100.4	
Frozen squid	Cd	8	0.12	0.45	0.26	_ ng =100.4	
(Loligo sp.)	Pb		ND	0.64	0.31	_	
	Hg		0.007	0.02	0.01		
Frozen black tiger prawn	Cd	8	0.002	0.03	0.01		
(Penaeus monodon)	Pb		ND	ND	ND	_	
	Hg		0.01	0.02	0.02	_	
Frozen cuttlefish	Cd	8	0.01	0.07	0.04		
(Sepia inermis)	Pb		ND	ND	ND		
	Hg		0.007	0.01	0.01	-	
Surimi (threadfin bream)	Cd	8	ND	0.005	0.005		
	Pb		ND	0.06	0.04	-	
	Hg		0.03	0.04	0.03	-	
Total number of	-	72	1	1	1	-	

Note : ND – less than LOD.

Table 9. Mean concentrations of	Cd Pb and Hg in the va	rious fish products from	Bukit Mertaiam Penang
ruble 9. mean concentrations (Cu, i o unu ing in the vu	nous non products nom	Dukit Morajani, i onang.

Fish Product	Analyte	No. of Samples	Min. value of results (ppm) – wet weight basis	Max. value of results (ppm) – wet weight basis	Average value of results (ppm) – wet weight basis	Average Recovery (%)	Remarks
Fishcake	Cd	8	ND	0.02	0.01		
	Pb		0.02	0.24	0.11		
	Hg		0.02	0.022	0.02		
Fishball	Cd	8	ND	ND	ND	Cd = 99.5	
	Pb		0.02	0.25	0.06	Pb =101.1	
	Hg		0.01	0.02	0.02	Hg =100.4	
Surimi Kintokidai A	Cd	8	ND	0.08	0.08	1	
(big eye snapper)	Pb	1	ND	0.15	0.12]	
	Hg]	0.02	0.03	0.03]	
Total number of	of samples =	24					

Note : ND – less than LOD.

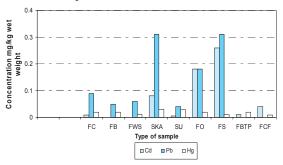
Discussion

<u>Mean Concentrations of Heavy Metals in various</u> <u>Fish Products</u>

All samples of fish products showed the mean concentration of Cadmium, Lead and Mercury were much lower than the standard of Commission Regulation (EC) No. 1881/2006, Food Act of Malaysia, 1983 (amendment 1993) and Food Regulation, 1985.

The mean concentrations of heavy metals in Table 7, was actually the overall result from Tables 8 and 9. However, some of the samples were collected from different states, where frozen shrimp, octopus, squid, black tiger prawn and cuttlefish can only be found in Perak. In Penang, only samples of fishcake, fishball and surimi kintokidai A were collected.

Graph 4. Mean Concentrations of Cd, Pb and Hg in various fish products.



FC - Fishcake; FB – Fishball; FWS – Frozen Shrimp (*P.vannamei*); SKA – Surimi Kintokidai A (big eye snapper); SU – Surimi (threadfin bream); FO – Frozen Octopus; FS – Frozen Squid; FBTP – Frozen Black Tiger Prawn; FCF – Frozen Cuttlefish

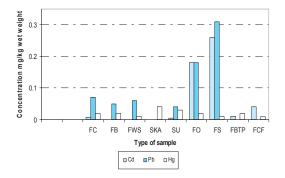
Graph 4 shows the mean concentration of Cd in fish products was less than 0.20 ppm except for frozen squid. Meanwhile, the concentration of Pb in surimi kintokidai A (big eye snapper) and frozen squid were higher than 0.30 ppm. For Hg, the concentration was observed to be lower than 0.1 ppm. Surimi kintokidai A showed concentrations of Cd and Pb exceeding the standard limits set by EC standard 1881/2006. However, only 1 sample out of 16 samples was analyzed to be more than this standard with their concentration of 0.08 for Cd and 0.37 ppm for Pb. However the rest of the samples had very low levels and not detected. Cd and Pb were not detected in the samples collected from processing establishments from Perak.

Frozen octopus and squids collected from Perak showed a higher concentration but not exceeding the EC standard for Cd, Pb and Hg. The highest concentration of Cd and Pb in frozen octopus was 0.47 and 0.44 ppm respectively. Frozen squid also had the highest concentration of Cd and Pb of 0.45 and 0.64 ppm. It is lower than the EC standard for Cd and Pb with their concentration of 0.5 and 1.0 ppm for crustaceans and cephalopods.

All the samples collected for Hg analysis showed that the concentration did not exceed the EC and National standards.

Mean concentration of heavy metals in various fish products from Perak

Graph 5. Mean Concentrations of Cd, Pb and Hg in various fish products from Perak.

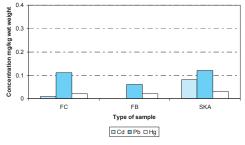


FC - Fishcake; FB – Fishball; FWS – Frozen Shrimp (*P.vannamei*); SKA – Surimi Kintokidai A (big eye snapper); SU – Surimi (threadfin bream); FO – Frozen Octopus; FS – Frozen Squid; FBTP – Frozen Black Tiger Prawn; FCF – Frozen Cuttlefish

Graph 5 shows the mean concentrations of Cd, Pb and Hg in the various fish products in Perak as stated in Table 8. The concentration of heavy metals in these products were less than 0.1 ppm except for frozen octopus and frozen squid.

Mean concentration of heavy metals in various fish products from Penang

Graph 6. Mean Concentrations of Cd, Pb and Hg in various fish products from Penang.



FC - Fishcake; FB – Fishball; SKA – Surimi Kintokidai A (big eye snapper);

Graph 6 also shows the mean concentrations of Cd, Pb and Hg in the various fish products in Penang which were stated in Table 9. The concentration of heavy metals in these products were less than 0.1 ppm for Cd and Hg and 0.2 ppm for Pb respectively.

<u>Findings</u>

The mean concentrations of heavy metals for all samples such as in fish and fish products were found to be not exceeding the food safety standard under the national standards. This implies that fishes from Johor east and west coasts and the fish products collected from Perak and Penang are within food safety limits for heavy metals contaminants. However, a more comprehensive study needs to be carried out to ensure seafood safety in Malaysia for heavy metals and other chemical contaminants.

c. Corrective Actions

Only one of the surimi kintokidai A sample from Perak exceeded the Pb level set by EC standard. The sample extraction and analysis was conducted again. No heavy metals was detected for this sample.

5. Problems and challenges encountered

- Method validation
- Proficiency testing
- Accreditation

6. Recommendations and Suggestions for **Future Follow up Action**

- The JTF programs should be continued among the • ASEAN country to collect data which can support regional trade opportunities and benefits.
- The proficiency testing (PT) should be extended • to another scope of analysis such as antibiotics, pesticides and histamine.
- Other elements such as Copper (Cu) and Arsenic (As) should be included in the survey.
- This survey should also cover canned products.
- SEAFDEC should consider the possibility of training for methyl Mercury and inorganic Arsenic for member countries because of the possibility that these analytes might be part of the requirement for food safety for products exported to EU country in the next few years.

Appendix 1

	Foodstuff	Maximum level (mg/ kg wet weight)	Performance criteria for sampling and for methods of analysis
	3.1.5. Muscle meat of fish $(^{1})(^{2})$	0,30	
3.1 LEAD	3.1.6. Crustacean, excluding brown meat of crab and excluding head and thorax meat of lobster and similar large crustaceans (Nephropidae and Palinuridae) (³)	0,50	Reg 333/2007
(Pb)	3.1.7. Bivalve molluscs (³)	1,50	
	3.1.8. Cephalopods (without viscera) (³)	1,00	
	3.2.5. muscle meat of fish $(^{1})(^{2})$, excluding fish species in 3.2.6 and 3.2.7	0,05	
3.2 CADMIUM (Cd)	 3.2.6. Muscle meat of the following fish (¹) (²): [1] anchovy (Engraulis species) [2] bonito (Sarda sarda) [3] common two-banded seabream (Diplodus vulgaris) [4] eel (Anguilla anguilla) [5] grey mullet (Mugil labrosus labrosus) [6] horse mackerel or scad (Trachurus trachurus) [7] louvar or luvar (Luvarus imperialis) [8] sardine (Sardina pilchardus) [9] sardinops (Sardinops species) [10] tuna (Thunnus, Euthynnys species, Katsuwonus pelamis) [11] wedge sole (Dicologoglossa cuneata) 	0,10	Reg 333/2007
	3.2.7. Muscle meat of swordfish (<i>Xiphias gladius</i>) (¹) (²)	0,30	
	3.2.8. Crustaceans, excluding brown meat crab and excluding head and thorax meat of lobster and similar large crustaceans (<i>Nephropidae</i> and <i>Palinuridae</i>) (³)	0,50	
	3.2.9. Bivalve molluscs (³)	1,00	
	3.2.10. Cephalopods (without viscera) (³)	1,00	

COM.REG. 1881/2006 - setting maximum levels for certain contaminants in foodstuff

	3.3.1. <u>Fishery products</u> (³) and <u>muscle meat of fish</u> (¹) (²), excluding species listed in 3.3.2. The maximum level applies to <u>crustaceans</u> , excluding the brown meat of crab and excluding head and thorax meat of lobster and similar large crustaceans (<i>Nephropidae</i> and <i>Palinuridae</i>)	0,50	
3.3 MERCURY (Hg)	 3.3.2. Muscle meat of the following fish (¹) (²) [1] anglerfish (Lophius spp.) / Baudroie [2] atlantic catfish (Anarhichas lupus) / Loup [3] bonito (Sarda sarda) [4] eel (Anguilla spp.) / anguille [5] emperor, orange roughly, rosy soldierfish (Hoplostethus atlanticus) [6] grenadier (Coryphaenoides rupestris) [7] halibut (Hippoglossus hippoglossus) /flétan [8] marlin (Makaira spp.) [9] megrim (Lepidorhombus species) [10] mullet (Mullus species) [11] pike (Esox lucius)/ Brochet [12] plain bonito (Orcynopsis unicolor) / palomète [13] poor cod (Tricopterus minutes) [14] Portuguese dogfish (Centroscymnes coelolepis) [15] rays (Raja spp.) [16] redfish (Sebastes marinus, S. mentella, S. viviparus) [17] sail fish (Istiophorus platypterus) [18] scabbard fish (Lepidopus caudatus, Aphanopus carbo) [19] sea bream, Pandora (Pagellus species) [20] shark (all species) [21] snake mackerel or butterfish (Lepidocybium flavobrunneum, Ruvettus pretiosus, Gempylus serpens) [22] sturgeon (Acipenser spp.) / esturgeon [23] swordfish (Xiphias gladius) / espadon [24] tuna (Thunnus spp., Euthynnus species, Katsuwonus pelamis) 	1,00	Reg 333/2007
3.4 TIN (inorganic) (Sn)	Canned foods other than beverages	200	Reg 333/2007 LOD<5 mg/kg LOQ<10 mg/kg

Reg 333/2007: for HM, Detection limit (LOD): 1/10 maximum level Quantification limit (LOQ): 1/5 maximum level