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Determination of Cd and Pb in Fruit Juice, Bottled Tea, Condiments and Dried Fish Samples Using ICP-MS

Riza A. Magbitang[1] , Melanie A. Bucsit[2],
Eugene S.F. Tia[2], Rowena Grace Rumbaoa[3],
Lowela Lou M. Cervas[3], Danica Angeline Dimaya[2]
and Irene B. Rodriguez [1,2]*
[1]Natural Sciences Research Institute, College of Science
[2]Institute of Chemistry, College of Science
[3]Department of Food Science and Nutrition, College of Home Economics
University of the Philippines Diliman, Quezon City, 1101 Philippines

ABSTRACT

Metals like cadmium (Cd) and lead (Pb) are introduced in the environment through natural processes and anthropogenic activities and may end up being present in food, where these metals may pose health risks. A method suitable for the simultaneous determination of Cd and Pb in various matrices of foodstuffs was validated and applied to different samples including fruit juice, bottled iced tea, several types of condiments, and in edible tissues of dried fish locally produced in the Philippines. Fruit juice and bottled iced tea samples were filtered prior to quantification of metals using inductively coupled plasma mass spectrometry (ICP-MS). Condiments and dried fish samples were mineralized using microwave-assisted nitric acid digestion before subsequent metal detection with ICP-MS. The method was validated using certified reference materials DORM 3 and NIST 1643e, and evaluation of recovery of spiked samples.The method was linear in the concentration range 0.01 to 500μg L^{-1} with correlation coefficients of 0.999 for both analytes. The estimated detection limits were 0.060 μgL^{-1} and 0.186 μgL^{-1} for Cd and Pb, respectively. The determined levels of Cd in fruit juice were in the range 0.06 ± 0.01 to 0.67 ± 0.01 μgL^{-1}, and Pb was detected in only one sample at 0.37 ± 0.02μgL^{-1}. For the bottled iced tea samples, Cd was detected in only one sample (0.13 ± 0.02 μgL^{1}) while none of the samples had detectable Pb concentration. For the condiments, the determined Cd levels were in the range 0.83 ± 0.06 to

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*Corresponding Author

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306.13 ± 2.52μgL^{-1}, whereas the determined Pb levels were in the range 2.14 ± 0.38 to 67.45 ± 7.76μgL^{-1}. For the dried fish samples, the Cd levels determined were in the range 2.00 ± 0.21 to 231.67 ± 5.32 μg kg^{-1} and that for Pb were in the range 2.38 ± 0.70 to 113.29 ± 2.25 μg kg^{-1}. These determined levels in different foodstuffs highlight the need for routine monitoring of these contaminants.

Keywords: Trace metal, fruit juices, condiments, dried fish, microwave digestion, ICPMS

INTRODUCTION

Commonly consumed beverages in the Philippines include commercial fruit juices and iced tea, which are quite popular not only due to their palatable flavors but also because of their perceived nutritive value. Over the years, businesses manufacturing these beverages have continued to increase and the market has demanded for other flavors to be introduced. Fruit juices are available in various packages (tetra packs, bottled, etc.) and flavors (apple, orange, or mixtures of flavors, etc.). Aside from fruit juice, Filipinos are also consumers of condiments which are mostly fermented products such as soy sauce (from soy beans), vinegar (from sugar cane and coconut nectar), fish sauce and fish paste, and shrimp paste (from krill). These are often used as seasonings in main dishes, as ingredients in marinade, and as dipping sauces. Dietary guidelines worldwide increasingly recommend fish consumption because of their nutritional benefits such as proteins, vitamins, minerals, and omega-3 polyunsaturated fatty acids (PUFAs) which exhibit protective effect against coronary heart disease and stroke (Kris-Etherton and others 2002, Joint WHO/FAO Expert Consultation 2003, Domingo 2007). However, concerns have been raised about fish consumption due to the potential health risks associated with environmental contaminants such as presence of metals that exhibit toxicity (Domingo 2007). All of these food products may be sources of metal contaminants when these have not passed rigorous quality checks.

Cadmium (Cd) and Lead (Pb) are ubiquitous in the environment and may enter the food chain due to their various applications and the improper handling of wastes. Cd is used as pigment in paints, in batteries, as stabilizers for PVC and can also be used in alloys (Jarup and others 1998). Likewise, Pb is used in batteries, alloys, pigments, cable sheathing, ammunition and as petrol additives (Garcia-Lestün and others 2010, Jarup and others 1998,). Improper handling of wastes containing these metals and the indiscriminate use of products containing these may lead to the release of these contaminants in the environment. Consequently, they can then enter the food chain. The presence of these metals in food is a concern because of possible negative health effects. Adverse health effects of Cd in the body include kidney, skeletal and reproductive deficiencies (Jarup and others 1998). Pb may cause poor intellectual performance in children and may induce renal and cardiovascular diseases in adults (Fewtrell and others 2004, Garcia-Lestün and others 2010).

The adverse health effects of these metals prompted the Joint FAO/WHO food standard programme Codex Committee on Contaminants in Foods (CCCF) to release, in March 2011, a new list of Maximum Levels (MLs) for contaminants and toxins. For Cd, the Committee set a Provisional Tolerable Monthly Intake (PTMI) of 25 µg kg^{-1} body weight. In the case of Pb, the Committee estimated that the previously established PTWI of 25 µg kg^{-1} body weight was no longer sufficient to prevent the effects of Pb. Consequently, a maximum level of 0.50 mg kg^{-1} of Pb in fruit juice was established. The Joint FAO/WHO Expert Committee on Food Additives suggested maximum levels for Cd of 2 mg kg^{-1} in marine bivalve mollusks and Provisional Tolerable Weekly Intake (PTWI) of 0.007 mg kg^{-1} body weight, and maximum levels for Pb of 0.3 mg kg^{-1} in fish and PTWI of 0.025 mg kg^{-1} body weight (JECFA 1993).

Likewise, the task group on contaminants in fish and other seafood of the Marine Strategy Framework Directive (MSFD) of the European Union has set maximum values of these metals in muscle tissues of fish. According to the report, Cd should not exceed 0.050 mg kg^{-1} based on the wet weight while Pb should not exceed 0.30 mg kg^{-1} wet weight (Swartenbroux and others 2010).

To ensure the quality of products, especially food items, there has been a steady development in analytical techniques and instrument capabilities for monitoring metal concentration. The importance of analyzing trace contaminants has called for methodologies which are robust, able to detect trace concentrations while increasing the sample throughput and sensitivity in the analysis of varied sample matrices. Conventional techniques employed by most laboratories include the use of open-vessel acid digestion, dry ashing or a combination of both as pre-treatment steps to elemental analysis using flame atomic absorption spectroscopy (F-AAS). More advanced laboratories utilize graphite furnace AAS (GF-AAS) or plasma-based techniques such as inductively coupled plasma mass spectrometry (ICPMS) or optical emission spectroscopy (ICPOES). In this study, Cd and Pb were determined in fruit juice, bottled iced tea, condiment samples and dried fish samples that are locally available in the Philippines after performing a suitable pre-treatment step, either microwave digestion or simple filtration and dilution, using ICPMS.

METHODOLOGY

Chemicals and Reagents

All chemicals and reagents used in this study were of analytical reagent grade unless otherwise specified. Nitric acid was obtained from Merck (Darmstadt, Germany). Single-element standards of Cd, Pb, indium (In) and rhenium (Re) with concentrations equivalent to 10,000 ± 30 μg mL^{-1} were purchased from CPI International (Santa Rosa, CA, USA). The reference material NIST 1643e (trace elements in water) used in this study was obtained from the National Institute of Standards and Technology (Maryland, USA). Another reference material used was DORM 3 (fish protein certified reference material for trace metals), which was obtained from the National Research Council Canada – Institute for National Measurement Standards (Ontario, Canada). All dilutions and solution preparations were done using ultrapure water prepared using a Barnstead system (18.2 MΩ cm resistivity, Thermo Fisher Scientific, Selangor Darul Ehsan, Malaysia).

Instrumentation

A Multiwave 3000 microwave digestion system (Anton Paar, Graz, Austria) fitted with a 16-position rotor for high digestion performance was used for the complete mineralization of the samples. An Agilent 7500cx ICPMS (Agilent Technologies, Germany) equipped with a MicroMist glass concentric nebulizer and an integrated autosampler (I-AS with type A vials, 89 x 6 mL capacity) was used as the element-selective detector. The monitored masses were *m/z* 111 for Cd and *m/z* 208 for Pb. The internal standards used were monitored at *m/z* equal to 115 for In and 185 for Re. The optimum conditions used for the analyses were as follows: RF power = 1550 W, carrier gas flow rate = 0.85 - 0.87 L min^{-1}, make-up gas flow rate = 0.21 – 0.24 L min^{-1}, nebulizer pump = 0.1 rps, sampling depth = 7.1 - 7.6 mm and the spray chamber temperature = 2.0 °C.

The calibration standards were prepared in the range 0.01 μg L^{-1} to 500 μg L^{-1} which had correlation coefficients of 0.999 or better for both analytes. The method detection limits obtained were 0.060 μg L^{-1} for Cd and 0.186 μg L^{-1} for Pb. These were estimated by getting the standard deviation of the results from the analysis of seven replicate solutions containing 1 μg L^{-1} of Cd and Pb, then subsequently multiplying the SD by the Student's t value (for n = 7, t value is equal to 3.143; Ripp 1996). A drift standard, composed of 1.0 μg L^{-1} of both Cd and Pb, was measured repeatedly throughout the analysis sequence to determine the precision of the measurements within a day. The determined relative standard deviations of the repeated measurements of the drift standard were less than 1% and 2% for Cd and

Pb, respectively. These values indicate a stable instrument performance and good precision of measurements.

Sampling

All samples (fruit juice and condiments) included in the study were locally produced by different manufacturers and represent common products in the market. The samples were bought from a local supermarket which carries varied products. A total of 17 fruit juices and 5 bottled iced tea samples were included in the analysis. Various flavors of fruit juices were included: apple, grape, mango, orange, strawberry, calamansi, and guyabano. For the bottled iced tea samples, different brands that carry lemon flavor were included. Eighteen condiment samples were analyzed in the study, which included cane and spiced coconut vinegars, fish and shrimp pastes, fish sauce, soy sauce, and liquid seasoning.

The dried fish samples were collected from various wet and dry markets in Quezon City. They were identified to the nearest family by a research scientist at the Institute of Biology, College of Science, University of the Philippines Diliman. The identification of the exact species of the dried fish samples was hampered because of the drying procedure that the samples have been subjected to, which led to the fish samples losing most of the scales and fins necessary for the identification procedure.

Sample Preparation

The fruit juice and iced tea samples were analyzed for the metal content after a simple filtration and dilution process. Samples were filtered using nylon syringe filters (pore size: 0.45 µm, diameter: 25 mm; obtained from Membrane Solutions LLC, Shanghai, China) immediately after opening. Three replicates were prepared for every sample. Samples were diluted 10-fold, acidified to 1% HNO_3, and then analyzed using ICP-MS. Accuracy of the method was evaluated by analyzing NIST 1643e prepared in the same way and by spiking some samples with known amounts of Cd and Pb standards, which were then subjected to the same procedure.

The condiment samples were mineralized by microwave-assisted acid digestion after addition of 2.0 mL of HNO_3 and 6.0 mL of water for solid samples (250 mg weighed to the nearest 0.01 mg) or 2.0 mL of HNO_3 and 5.0 mL water for liquid samples (volume of sample = 1.0 mL). The digestion step was carried out by following this optimized program (modified from Magbitang and Rodriguez 2012): the system was ramped to 600W for 5 min, kept at this condition for 30 min, decreased to 500 W and held at this condition for another 10 min, before allowing the system to cool. After digestion, the volume was adjusted to 10 mL. For the dried fish analysis, the edible portions of the dried fish samples were carefully scraped using acid-washed plastic knives. Triplicate aliquots (250 mg weighed to the nearest 0.1 mg) of each sample were weighed and subjected to the optimized mineralization procedure prior to ICPMS analysis. Triplicate aliquots of reference materials DORM-3 (250 mg) and NIST 1643e (1.0 mL) were digested along with the condiment samples and dried fish samples.

RESULTS AND DISCUSSION

The results for the validation of the pre-treatment methods and ICPMS parameters using certified reference materials are summarized in Table 1. NIST reference material 1643e was used for both the validation of the filtration step used for juice and tea samples, and the microwave-assisted digestion step used for the condiment samples. The reference material DORM 3 was also used for the validation of the microwave-assisted digestion step to simulate the complex matrix of some of the condiment samples that were derived from fish. This was also the main reference material for the dried fish samples.

The results for NIST 1643e analyzed after simple filtration reflected good accuracy, with 103.3% recovery for Cd and 92.4% for Pb. This was comparable to the results for NIST 1643e obtained after the microwave-assisted digestion with 104.2% recovery for Cd and 97.1% for Pb. As for the more complex matrix, the values obtained were 88.0% recovery for Cd and 84.6% recovery for Pb in DORM 3 after the complete mineralization by microwave digestion. These results suggest that the microwave digestion step would be a sufficient pre-treatment step for the condiment samples and dried fish samples prior to ICPMS analysis.

Table 1. Determined values for Cd and Pb in the certified reference materials processed using simple filtration or microwave-assisted acid digestion prior to ICPMS analysis (expressed as mean concentration ± SD, n = 3)

Certified Reference Material	Element	Certified Value	Obtained Value	% Recovery
NIST 1643e[a]	^{111}Cd	6.568 ± 0.073 μg L^{-1}	6.78 ± 0.16 μg L^{-1}	103.2
	^{208}Pb	19.630 ± 0.210 μg L^{-1}	18.14 ± 1.17 μg L^{-1}	92.4
NIST 1643e[b]	^{111}Cd	6.568 ± 0.073 μg L^{-1}	6.85 ± 2.66 μg L^{-1}	104.2
	^{208}Pb	19.630 ± 0.210 μg L^{-1}	19.05 ± 2.16 μg L^{-1}	97.1
DORM 3[b]	^{111}Cd	290.000 ± 20.00 μg kg^{-1}	255.17 ± 4.13 μg kg^{-1}	88.0
	^{208}Pb	395.000 ± 50.00 μg kg^{-1}	334.27 ± 17.72 μg kg^{-1}	84.5

[a] Pre-treatment step includes simple filtration followed by dilution.

[b] Pre-treatment step includes microwave-assisted acid digestion followed by dilution.

For further validation of the filtration step used for juice and tea samples, known concentrations of the standards were spiked in some of the samples and the results are presented in Table 2. For the recovery test, juice and tea samples (one each) were spiked with 0.5, 5, and 50 µg L^{-1} concentrations of Cd and Pb standards.

Table 2. Percent recovery values for juice and tea samples spiked with known concentrations of standard Cd and Pb solutions (expressed as mean concentration ± SD, n = 3)

Sample	Concentration of Cd and Pb (µg L^{-1}) Added	Obtained value (µg L^{-1}) Cd	% Recovery	Pb	% Recovery
Juice	0.5	0.50 ± 0.02	100.3	0.36 ± 0.22	72.6
	5	5.09 ± 0.05	101.9	4.77 ± 0.15	95.4
	50	51.38 ± 0.87	102.8	48.60 ± 0.22	97.2
Tea	0.5	0.53 ± 0.02	105.3	0.29 ± 0.14	57.6
	5	5.16 ± 0.06	103.2	4.94 ± 0.36	98.9
	50	53.01 ± 0.81	106.0	48.38 ± 0.54	96.8

As shown above, good recovery values for the three different concentrations were obtained for Cd, which ranged from 100.3% to 106.0%. Recovery of Pb at the lowest concentration was observed to be low, which were at 57.6% and 72.6% for juice and tea samples, respectively. For the higher concentrations, 5 and 50 µg L^{-1}, recoveries were from 95.4% to 98.9%. These recovery values are within the acceptable values as suggested by Taverniers and others (2004) and may show the influence of the matrix of the sample on the recovery of the target analytes. The good recoveries of the spiked samples indicate that the simple filtration and dilution method used in the study is reliable in quantifying the amount of these metals in juice and bottled iced tea samples. Analysis of these samples can be done successfully without any further sample pretreatment.

The results of the analysis of Cd and Pb in actual samples of fruit juice and bottled iced tea samples, as well as their respective brand codes, are summarized in Table 3. The different brands are coded from A to L because they carry various products and flavors.

From the data obtained, it is evident that most of the juice samples analyzed have Pb content lower than the method detection limit while some have detectable Cd concentrations. Currently, there is no maximum limit set for Cd in fruit juices. The determined levels of Cd and Pb in these samples indicate that there is no significant level of the metal contaminants in these food products but the detectable levels of Cd suggest the need for routine monitoring of these contaminants in these products.

Table 3. Determined concentration of Cd and Pb in different juice and bottled iced tea samples (expressed as mean concentration ± SD, n = 3)

Samples		Concentration (µg L^{-1})	
Fruit Juice	**Brand**	**Cd**	**Pb**
Apple	A	< MDL	0.37 ± 0.02
	B	< MDL	< MDL
Mango	A	0.25 ± 0.05	< MDL
	B	0.23 ± 0.05	< MDL
	C	0.67 ± 0.01	< MDL
	E	< MDL	< MDL
Orange	A	< MDL	< MDL
	B	< MDL	< MDL
	C	0.12 ± 0.01	< MDL
	F	< MDL	ND
	G	0.35 ± 0.02	< MDL
Grape	A	0.10 ± 0.02	< MDL
	B	< MDL	< MDL
	C	0.06 ± 0.01	< MDL
Strawberry	C	< MDL	< MDL
Calamansi	D	< MDL	< MDL
Guyabano	E	0.12 ± 0.03	< MDL
Bottled iced tea	H	< MDL	ND
	I	0.13 ± 0.02	ND
	J	< MDL	ND
	K	< MDL	ND
	L	< MDL	ND

ND: No detectable value indicates that the signals from ICPMS measurements were not significantly higher than the blank.
<MDL: below the method detection limit.

The different condiment samples were subjected to the mineralization procedure by microwave digestion prior to elemental analysis. The determined concentration of Cd and Pb in the condiment samples, with the corresponding brand codes, are presented in Table 4. Of all the condiments included in the study, only shrimp paste is available in solid form. The determined levels of Cd in shrimp paste were 9.68 ± 2.25 and 88.69 ± 6.58 µg kg^{-1} for the two brands. For the liquid condiment samples, the determined levels of Cd ranged from 0.83 ± 0.06 to 306.13 ± 2.52 µg L^{-1}. On the other hand, the determined levels of Pb were 50.68 ± 14.9 and 63.91 ± 6.77 µg kg^{-1} in the two brands of shrimp paste. The obtained values for Pb in liquid condiment samples were in the range 2.14 ± 0.38 to 67.45 ± 7.76 µg L^{-1}. In general, shrimp and fish paste samples contain elevated levels of Cd and Pb, which may have been due to the raw material itself. Seafood and seafood products are widely known to contain elevated levels of metal contaminants.

Table 4. Determined concentration of Cd and Pb in different condiment samples (expressed as mean concentration ± SD, n = 3)

Type of Condiment	Brand	Cd	Pb
Shrimp paste*	1	88.69 ± 6.58	50.68 ± 14.9
	7	9.68 ± 2.25	63.91 ± 6.77
Fish paste	1	21.07 ± 2.43	37.59 ± 1.67
	2	116.44 ± 10.52	67.45 ± 7.76
Fish sauce	2	306.13 ± 2.52	4.24 ± 0.98
	3	1.94 ± 0.10	2.14 ± 0.38
	8	106.53 ± 1.40	18.07 ± 3.30
Soy Sauce	3	0.83 ± 0.06	<MDL
	4	6.24 ± 0.15	2.61 ± 0.74
	6	8.83 ± 0.32	6.66 ± 1.66
Spiced coconut vinegar	5[a]	2.45 ± 0.27	10.24 ± 3.33
	5[b]	3.22 ± 0.20	9.81 ± 2.46
White vinegar	4	<MDL	<MDL
	6	<MDL	<MDL
Cane vinegar	4	<MDL	<MDL
	6	<MDL	<MDL
Liquid seasoning	9	0.89 ± 0.05	3.32 ± 1.14
	10	1.94 ± 0.47	3.33 ± 0.65

Mean Concentration (μg L^{-1})

*Concentrations are expressed as μg kg^{-1}

<MDL = below detection limit

[a] spicy variant; [b] sweet variant

For the dried fish, the samples chosen for this study were dried fish types preferred by consumers and widely available in all the major wet and dry markets in the most populated city in Metro Manila. The determined Cd and Pb levels for all samples are summarized in Table 5, including information about the sources. From the data, all determined Pb levels and most of Cd levels were below the suggested limit by the EU-MSFD (Swartenbroux and others 2010). All of the determined values for Cd and Pb were below the suggested maximum limits of the JECFA for marine-derived food products (JECFA 1993). The Cd levels determined were in the range 2.00 ± 0.21 to 231.67 ± 5.32 μg kg^{-1} and that for Pb were in the range 2.38 ± 0.70 to 113.29 ± 2.25 μg kg^{-1}.

The determined levels of Cd and Pb in the samples studied show that there is a need to monitor these contaminants in these types of food products. Although the concentrations in the fruit juice and tea samples are relatively low, routine monitoring is still necessary as the effects of these contaminants are dependent on the extent of the exposure. Compared with the other types of condiments, the

Table 5. Determined concentrations of Cd and Pb in dried fish samples expressed as mean ± SD (n = 3). Common names, scientific names in some and sample codes are shown (sample codes are designated with respect to source market)

Sample Code and Source	Common Name	Scientific Name	Cd Concentration µg kg^{-1}	Pb Concentration µg kg^{-1}
M1FA	Purse-eyed scad	*Selar sp.*	26.93 ± 1.31	6.35 ± 0.69
M1FB	Deep body sardinella	*Sardinella brachysoma*	25.82 ± 1.49	3.44 ± 0.78
M1FC	Beltfish	*Family Trichiuridae*	3.53 ± 0.15	35.29 ± 1.34
M1FD	Parrot fish	*Scarus sp.*	32.93 ± 0.78	8.90 ± 1.13
M1FE	Croaker	*Family Sciaenidae*	6.84 ± 0.35	47.42 ± 1.56
M2FA	Yellow tail fusilier	*Caesio caerulaurea*	57.23 ± 2.49	23.10 ± 0.93
M2FB	Deep body sardinella	*Sardinella brachysoma*	22.23 ± 0.86	15.32 ± 1.16
M2FC	Short-bodied mackerel	*Family Carangidae*	28.24 ± 1.58	66.22 ± 1.85
M2FD	Flying fish	*Cypselurus sp.*	55.27 ± 1.45	113.29 ± 2.25
M3FA	Long-jawed mackerel	*Rastrelliger sp.*	7.35 ± 1.29	2.38 ± 0.70
M3FB	Deep body sardinella	*Sardinella brachysoma*	5.68 ± 0.54	67.78 ± 4.88
M3FC	Short-bodied mackerel	*Family Carangidae*	45.01 ± 2.06	< MDL
M3FD	Tilapia	*Oreochromis sp.*	< MDL	13.94 ± 1.09
M3FE	Threadfin bream	*Nemipterus sp.*	16.34 ± 1.28	< MDL
M4FA	Cavalla fish	*Carangoides sp.*	46.45 ± 1.76	28.67 ± 0.82
M4FB	Deep body sardinella	*Sardinella brachysoma*	2.00 ± 0.21	< MDL
M4FC	Purse-eyed scad	*Selar sp.*	137.75 ± 2.75	< MDL
M4FD	Ponyfish	*Leiognathus sp.*	19.81 1.33	10.14 ± 1.15
M4FE	Barracuda	*Family Sphyraenidae*	7.70 ± 0.53	12.00 ± 1.48
M5FA	Lizard fish	*Family Synodontidae*	121.08 ± 1.89	4.14 ± 1.10
M5FB	Deep body sardinella	*Sardinella brachysoma*	2.92 ± 0.30	< MDL
M5FC	Goat fish	*Family Mullidae*	54.28 ± 1.95	27.60 ± 1.21
M5FD	Cavalla fish	*Carangoides sp.*	41.38 ± 1.45	46.59 ± 1.32
M6FA	Threadfin bream	*Nemipterus sp.*	19.38 ± 1.31	5.46 ± 0.69
M6FB	Deep body sardinella	*Sardinella brachysoma*	3.16 ± 0.37	< MDL
M6FC	Tilapia	*Oreochromis sp.*	< MDL	< MDL
M6FD	Yellow tail fusilier	*Caesio caerulaurea*	59.27 ± 1.38	64.51 ± 1.55
M6FE	Round scad	*Decapterus sp.*	22.14 ± 0.91	4.48 ± 1.22
M7FA	Round scad	*Decapterus sp.*	231.67 ± 5.32	< MDL
M7FB	Deep body sardinella	*Sardinella brachysoma*	4.32 ± 0.44	< MDL
M7FC	Long-jawed mackerel	*Rastrelliger sp.*	15.27 ± 1.18	2.42 ± 1.18
M7FD	Ponyfish	*Leiognathus sp.*	25.09 ± 0.93	5.41 ± 4.55

fish- and shrimp-derived condiments contain elevated levels of both Cd and Pb, which indicate that the contamination may come from the source material. For these types of condiments, further work should be done to ascertain where the source of contamination is from.

Moreover, the obtained values for the analytes in these condiment samples show that stricter regulations should be in place to ensure that these food products are safe for human consumption. The data presented here show that Cd and Pb are present in dried fish samples and although most of the determined levels were below the suggested limits by the EU-MFSD and the JECFA, these detected values merit routine monitoring of Cd and Pb levels in edible fish tissues especially those types that are consumed regularly.

CONCLUSION AND RECOMMENDATIONS

Different sample preparation methods suitable for the pre-treatment of fruit juice, bottled tea, condiments and dried fish samples prior to metal-selective determination by ICPMS were validated and applied to actual samples. The methods were found to be simple and suitable for different matrices. Application of the simple filtration and subsequent analysis by ICPMS to different fruit juice and bottled tea samples showed that Cd and Pb were detectable in fruit juice samples while these were not detected in the bottled tea samples. Different types of condiments and the edible tissues of dried fish samples were subjected to the mineralization by microwave-assisted acid digestion and ICPMS analysis. The results showed that Cd and Pb were present in most types of condiments, with the exception of white and cane vinegar, and are also present in edible tissues of dried fish. The results also revealed that the sample pre-treatments steps were suitable for the different samples and robust enough to allow for the simultaneous determination of Cd and Pb by ICPMS.

The presence of these metal contaminants in these food products necessitates for a wider scope of study, which should cover larger sample size and longer time frame, to assess if there is indeed a need for stricter regulations to ensure safety of the consumers. The samples included in this study are commonly consumed by the majority of the population and hence, the presence of these contaminants in the food products may mean significant exposure, which warrants the need for the wider scope of sampling area, size and duration. A study which focuses on the differences in the daily intake of varying types of consumers and the associated intake of the contaminants may also provide meaningful data for proper assessment of risk.

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Irene B. Rodriguez <ibrodriguez@upd.edu.ph> is currently a postdoctoral research fellow at the Research Center for Environmental Changes in Academia Sinica, Taiwan. Her work is centered on how trace metals interact with light to control the nitrogen cycle in marine biogeochemical systems. She is involved in research to better understand the role of trace metals in an ocean undergoing acidification, and how phytoplankton utilize these metals and accumulate these in the cells before eventually passing the metals up the trophic levels.

Riza A. Magbitang <riza.magbitang@gmail.com> is a University Research Associate I (URA I) at the Natural Sciences Research Institute, University of the Philippines Diliman. She is currently enrolled in the MS Chemistry program of the Institute of Chemistry of the same University, where she also obtained her BS Chemistry degree. Her research interests, among others, include environmental analytical chemistry, particularly optimization and validation of methods for the determination of environmental pollutants.

Melanie A. Bucsit is currently an Instructor at the Institute of Chemistry, University of the Philippines Diliman, where she also obtained her MS Chemistry degree.

Rowena Grace O. Rumbaoa <rorumbaoa@yahoo.com> is a faculty member at the Department of Food Science and Nutrition, at the College of Home Economics, University of the Philippines Diliman. She primarily handles courses in food chemistry and analysis, and food processing. She received her MS Food Science degree from the same university, where she is also currently pursuing her PhD degree. She has co-authored some works on algal polysaccharides, antioxidants, and proximate composition of foods. She has also completed researches on rootcrop antioxidants, processing and fortification of juices, modified atmosphere packaging and algal polysaccharides.

Eugene S.F. Tia and **Danica Angeline Dimaya** are graduate students at the Institute of Chemistry, College of Science, University of the Philippines Diliman.

Lowela Lou M. Cervas <lowelacervas@yahoo.com> is an Instructor at the University of the Philippines Los Baños where she handles courses on Food Analysis, Food Processing, Food Packaging and Labeling, Food Hygiene and Sanitation, and Practicum I– Pilot Scale.