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Monitoring of heavy metals, bisphenol A and phenol migrated from food packages for delivery

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Abstract: Food delivery is gaining popularity due to changes in lifestyle and dietary patterns. However, packages used to delivery food may contain contaminants including heavy metals, or additives added during manufacturing process which may migrate into food during processing and transportation. In this study, a total of 58 food packages for delivery were collected and tested for migration of heavy metals (lead, cadmium and arsenic), bisphenol A and phenol into food simulants. The method was validated by evaluating linearity of calibration curve, limit of detection, limit of quantification, recovery and precision. Result of heavy metal migration showed that lead was the most frequently migrated metal and the highest concentration was detected in a polypropylene sample. Although there are no specific migration limits for bisphenol A and phenol in packaging materials tested in this study, migrations of bisphenol A and phenol were detected in some packages. This may due to contamination or additives added during manufacture of packages. Risk (%) was calculated to analyze the risk associated with the migration of heavy metals, bisphenol A and phenol, and was always below 1 %. These results showed that food packages for delivery are safe in terms of heavy metals, bisphenol A and phenol migration.

Key words: food packaging, heavy metals, bisphenol A, phenol, migration

1. Introduction

Food containers and packaging are widely used to keep food from external contamination and facilitate transport. Due to the recent increase in single-person and dual-income households, the demand for food delivery is increasing owing to its convenience, and accordingly, the demand and use of food delivery containers is also increasing. The containers used for food packaging are typically made of polypropylene (PP), polystyrene (PS), polyethylene (PE), and so on.[1] Traces of heavy metals such as lead, cadmium, and arsenic may also be present in these plastic containers in the form of impurities of additives such as catalysts and stabilizers.[2] Moreover, in the manufacture of such plastic containers, various substances are

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used as raw materials, such as bisphenol A and phenol, as well as additives such as antioxidants and plasticizers. These substances may be transferred to food during storage and transport, and may affect the quality of food and the health of consumers.[2,3]

Lead and cadmium are known to accumulate in the body and cause chronic poisoning, and arsenic is known to be carcinogenic.[4] In Korea, specific migration limit of lead, a common heavy metal, is restricted to 1 mg/L in food utensils, containers, and packaging.[5] Bisphenol A is used to produce polycarbonates, epoxy resins, and so on; however, it is a skin irritant[6] and has been reported to mimic estrogen, disrupting the endocrine system[7] and raising concerns about safety. Phenol is used as a raw material for phenol resins, paints, and bisphenol A,[8] and it has been reported to cause irritation to the skin, eyes, and mucous membranes on exposure.[9] In Korea, specific migration limit of sum of bisphenol A, phenol, and p-tertbutylphenol eluted from polycarbonate, polyarylsulfone, polyarylate, and epoxy resins is limited to ≤2.5 mg/L (≤0.6 mg/L for bisphenol A) in the standards and specification for food utensils, containers and packages.[5] Fan *et al.*[10] reported that bisphenol A was detected in water stored in a polyethylene terephthalate (PET) bottle, which does not use bisphenol A for manufacturing, suggesting that it may be attributed to other routes such as contamination or recycling. Hwang *et al.*[11] reported that bisphenol A may be present in materials other than polycarbonate and epoxy resin due to contamination. Jurek *et al.*[12] reported that bisphenol A can be used in inks and for other purposes, which can also lead to exposure to bisphenol A. Even when phenol is not added during the manufacture of plastics, it may be present in plastics as degradation products of phenolic antioxidants.[13] Thus, it seems necessary to investigate the amount of these substances migrated from utensils and containers/packaging that do not yet have a standard for bisphenol A phenol levels.

This study aims to assess the safety of currently used food delivery containers and packaging by identifying the level of migrated lead, cadmium, arsenic, bisphenol A, and phenol, in addition to calculating the risk by comparing the levels with the current human safety standards.

2. Experiment

2.1. Samples

The containers and packaging for food delivery (21 PS, 26 PP, and 4 PET) were purchased at a local store and from an online store. The polyvinyl chloride (PVC) used for wrapping food was purchased from an online store.

2.2. Standards and reagents

The inductively coupled plasma (ICP) multi-element standard solution was purchased from Merck (Darmstadt, Germany), and standards for bisphenol A and phenol were purchased from Sigma-Aldrich (St. Louis, MO, USA) and Accustandard (New Haven, CT, USA), respectively. Nitric acid was purchased from Matsunoen Chemicals (Osaka, Japan); acetic acid and other solvents were purchased from Merck (Darmstadt, Germany), and distilled water was purified using the ELGA Purelab system (Marlow, UK).

2.3. Preparation of the test solution

The food simulants for food delivery container/packaging were prepared according to the method for preparation of migration test solution for each material specified in standards and specifications for utensils, containers and packages (Ministry of Food and Drug Safety, 2020). Considering the nature of the delivered food, water, 4 % acetic acid, and n-heptane were used as food simulants while excluding 20 % and 50 % ethanol. To analyze heavy metals, only a 4 % acetic acid was used according to the solubility of lead, cadmium, and arsenic, and the test method in the standards and specifications for utensils, containers and packages.[14] In the analysis of bisphenol A and phenol, water, 4 % acetic acid, and n-heptane were used as food simulants. For water and 4 % acetic acid, analysis was conducted without additional pretreatment, while in case of n-heptane for the analysis of bisphenol A, the sample was dried with nitrogen gas and re-dissolved in water. To

analyze phenol, n-heptane was re-extracted with water before the analysis.

2.4. Determination of heavy metals, bisphenol A and phenol

The analysis of lead, cadmium, and arsenic was performed using ICP mass spectrometry (ICP-MS, NexION 300D, PerkinElmer, USA) with conditions listed in *Table* 1. The standard solution was prepared by diluting the ICP multi-element standard solution (100 mg/L) with 4 % acetic acid to the final concentrations of 0.5, 1, 2, 5, and 10 μg/L.

High-performance liquid chromatography (HPLC, Acquity UPLC H-class, Waters, USA) was used to analyze bisphenol A and phenol levels under the conditions listed in *Table* 2. For bisphenol A, 100 mg of standard was precisely weighed and then added to 100 mL methanol to prepare the standard solution (1 mg/mL), which was diluted with water to concentrations of 2, 5, 10, 20, 50, and 100 μg/L. For phenol, the standard solution (1000 μg/mL) was diluted with water to prepare concentrations of 2, 5, 10, 20, 50, and 100 μg/L.

Table 1. Analytical conditions of ICP-MS for Pb, Cd and As analysis

Instrument parameter	Condition
R.F. Power	1600 W
Argon gas flow rate	
Auxiliary	1.2 L/min as Argon
Nebulizer	0.95 L/min as Argon
Plasma	20 L/min as Argon
Pulse stage voltage	900 V
Mass(amu)	
Pb	207.977
Cd	110.904
As	74.9216

Table 2. Analytical conditions of HPLC for Bisphenol A and phenol

Parameter	Condition
Column	Kinetex C18 (4.6 mm I.D. × 250 mm, 5 μm)
Detector	Fluorescence detector (Ex. 275 nm, Em. 300 nm)
Oven temperature	40 °C
Mobile phase	A: deionized water, B: acetonitrile A:B (70:30) to A:B (30:70) Run time : 15 min
Injection volume	20 μL
Flow rate	1 mL/min

2.5. Safety assessment

Using the results for heavy metals, bisphenol A, and phenol levels migrated from food delivery containers and packaging, the estimated daily intake level of these substances was calculated and compared to the human safety criteria for safety assessment.

3. Results and Discussion

3.1. Method validation

A new calibration curve was prepared for each analysis. The value of coefficient of determination

Table 3. Recovery, precision, limit of detection and limit of quantification of Pb, Cd and As

Element	Spiked level (μg/L)	Recovery (%) (n=3)	Precision (RSD%)		LOD (μg/L)	LOQ (μg/L)
			Intra-day (n=3)	Inter-day (n=9)		
Pb	0.5	97.07 ± 1.80	1.06	5.88	0.011	0.034
	2	99.97 ± 2.98	1.64	2.30		
	10	100.67 ± 0.92	1.32	0.84		
Cd	0.5	91.50 ± 2.72	2.21	2.93	0.007	0.021
	2	96.23 ± 0.69	0.43	1.32		
	10	100.78 ± 1.11	0.79	1.17		
As	0.5	95.27 ± 2.09	5.39	2.54	0.011	0.034
	2	98.64 ± 0.10	4.43	2.36		
	10	102.61 ± 1.55	3.01	2.30		

Table 4. Recovery, precision, limit of detection and limit of quantification of bisphenol A

Simulant	Spiked level (ug/L)	Recovery (%)	Precision (RSD%)		LOD (μg/L)	LOQ (μg/L)
			Intra-day (n=3)	Inter-day (n=9)		
Water	2	125.63 ± 16.19	13.29	11.65	0.17	0.51
	20	104.41 ± 0.62	1.3	2.97		
	100	98.38 ± 0.39	0.64	4.12		
4% Acetic acid	2	106.91 ± 8.99	3.91	14.80		
	20	101.47 ± 3.65	4.15	3.04		
	100	98.69 ± 0.50	1.11	3.37		
n-Heptane	2	110.88 ± 1.67	4.53	9.09		
	20	81.44 ± 1.09	1.36	5.99		
	100	85.93 ± 0.65	0.46	4.72		

Table 5. Recovery, precision, limit of detection and limit of quantification of phenol

Simulant	Spiked level (ug/L)	Recovery (%)	Precision (RSD%)		LOD (μg/L)	LOQ (μg/L)
			Intra-day (n=3)	Inter-day (n=9)		
Water	2	88.78 ± 2.43	5.75	10.97	0.12	0.38
	20	102.29 ± 0.33	0.58	1.00		
	100	103.20 ± 0.36	0.66	0.82		
4% Acetic acid	2	91.76 ± 0.77	7.00	14.07		
	20	106.01 ± 0.41	1.25	0.79		
	100	103.77 ± 0.05	1.39	1.29		
n-Heptane	2	87.73 ± 2.08	5.44	6.57		
	20	89.11 ± 0.33	1.09	0.66		
	100	88.46 ± 0.44	2.36	0.88		

(R^2) was at least 0.999, showing an acceptable linearity for the concentration range of 0.5-10 μg/L for heavy metals and 2-100 μg/L for bisphenol A and phenol. The limit of detection (LOD), limit of quantification (LOQ), and precision for heavy metals, bisphenol A, and phenol are listed in *Tables* 3-5, respectively. The LOD and LOQ were obtained by multiplying 3.3 and 10, respectively, to the result of dividing the standard deviation of the calibration curve by the slope.[15,16] To calculate the recovery rate, the samples to which the standard solution was added in three different concentrations was analyzed and the added concentration was compared with measured concentration. The recovery rates for lead, cadmium, and arsenic were all ≥91 % while that of bisphenol A and phenol was ≥81 %, showing good recovery. To check precision, intra-day and inter-day reproducibility were calculated using the relative standard deviation (RSD%) of the results obtained from three measurements performed in one day and three measurements performed over 3 days, respectively. It was found to be ≤15 %.

3.2. Migration level of heavy metals

The results of migration of heavy metals from 58 food delivery containers and packaging are shown in *Table* 6. Among heavy metals, lead showed the highest detection frequency and mean concentration; this finding is in agreement with those of studies that have analyzed food utensils made of polycarbonate and polylactide.[2,17] There were no samples exceeding the specific migration limit of 1 mg/L for lead, and the highest concentration detected was 0.61 μg/L for polypropylene, which was very low compared to the specific migration limit. Cadmium was detected in all the materials except PET, and arsenic was detected

Table 6. Concentrations of Pb, Cd and As migrated from food packaging materials into 4% acetic acid

Packaging material	Element	Detection rate[a]	Range (μg/L)	Average (μg/L)
PS	Pb	10/21	ND[b]-0.28	0.07
	Cd	2/21	ND-0.02	0.002
	As	1/21	ND-0.09	0.004
PP	Pb	11/26	ND-0.61	0.08
	Cd	1/26	ND-0.02	0.0008
	As	0/26	ND	ND
PET	Pb	2/4	ND-0.18	0.08
	Cd	0/4	ND	ND
	As	0/4	ND	ND
PVC	Pb	4/7	ND-0.32	0.09
	Cd	1/7	ND-0.05	0.008
	As	0/7	ND	ND

[a]Detection rate : Number of detected samples/total number of samples
[b]ND : not detected or below LOQ

Table 7. Concentrations of bisphenol A migrated from food packaging materials into food simulants.

Packaging material	Simulant	Detection rate[a]	Range (μg/L)	Average (μg/L)
PS	Water	0/21	ND[b]	ND
	4 % acetic acid	1/21	ND-1.555	0.074
	n-Heptane	1/21	ND-4.033	0.192
PP	Water	0/26	ND	ND
	4 % acetic acid	0/26	ND	ND
	n-Heptane	0/26	ND	ND
PET	Water	0/4	ND	ND
	4 % acetic acid	0/4	ND	ND
	n-Heptane	0/4	ND	ND
PVC	Water	0/7	ND	ND
	4 % acetic acid	0/7	ND	ND
	n-Heptane	0/7	ND	ND

[a]Detection rate : Number of detected samples/total number of samples
[b]ND : not detected or below LOQ

only in PS.

3.3. Migration level of bisphenol A and phenol

The results of migration of bisphenol A and phenol from 58 food delivery containers and packaging are shown in *Table* 7. Bisphenol A was detected in 2 out of 21 PS samples. One case was detected in 4 % acetic acid and the other in n-heptane, and the migration levels were 1.555 and 4.033 μg/L, respectively. The concentrations detected in this study are very low, given that there is no regulation for bisphenol A in PS, and compared to the bisphenol A specific migration limit of 600 μg/L for polycarbonate. Fasano *et al*.[18] reported that in Campagna, Italy, bisphenol A was detected in beverages in PS and PET containers when sugar drinks were analyzed for the amount of bisphenol A. Guart *et al*.[19] detected bisphenol A from the PS bottle cap septum and reported that it may have migrated from the additives used to manufacture the septum. Based on these results, it is thought that bisphenol A migrating from PS in this study may have been transferred by contamination

Table 8. Concentrations of phenol migrated from food packaging materials into food simulants

Packaging material	Simulant	Detection rate[a]	Range (μg/L)	Average (μg/L)
PS	Water	15/21	ND[b]-9.930	1.294
	4% acetic acid	15/21	ND-15.492	2.234
	n-Heptane	2/21	ND-21.860	1.199
PP	Water	10/26	ND-2.068	0.468
	4% acetic acid	8/26	ND-1.355	0.295
	n-Heptane	0/26	ND	ND
PET	Water	1/4	ND-0.489	0.122
	4% acetic acid	0/4	ND	ND
	n-Heptane	0/4	ND	ND
PVC	Water	3/7	ND-2.872	0.718
	4% acetic acid	4/7	ND-1.889	0.746
	n-Heptane	0/7	ND	ND

[a]Detection rate : Number of detected samples/total number of samples
[b]ND : not detected or below LOQ

or via the additives used in the plastic manufacturing process.

During the analysis of bisphenol A, another peak was observed; comparing the retention times of the peaks revealed that the peak was attributed to phenol; subsequently, the amount of phenol migration was analyzed (*Table* 8). When the detection frequency was compared according to material and food simulant, phenol was mainly detected in PS, PP, and PVC when the food simulant was water and 4 % acetic acid, and the mean migration level was the highest in PS. The maximum migration limit was 21.860 μg/L when the n-heptane food simulant was used in PS, which was very low compared to the specific migration limit of 2500 μg/L for polycarbonate. The detection frequency of phenol was higher than that of bisphenol A. In a previous study that analyzed food utensils made of polycarbonate, the detection frequency of phenols was also higher than that of bisphenols, and detected in food simulants except n-heptane.[20] In a study that analyzed the migration levels of bisphenols and phenols in food utensils made with different materials, bisphenol A was not detected while phenol was detected in the food simulants excluding n-heptane in materials including polylactide and thermoforming polyurethane[11]. In that study, however, phenol was not detected in PP, which may be attributed to the difference in the samples used in the experiment or the differences in the LOQs. Hwang *et al.*[11] used liquid chromatography mass spectrometry (LC-MS) for analysis and reported that the LOQ was relatively higher as there was difficulty in analyzing phenol with MS due to the small molecular weight. The LOQ of the study was 98.7 μg/L, which is higher than the level detected in this study (0.38 μg/L) or in the study by Park et al.[20] (0.6 μg/L) that analyzed phenol using liquid chromatography fluorescence detector.

3.4. Safety assessment

The estimated daily intake (EDI) of heavy metals, bisphenol A, and phenol was calculated using the following formula.

$$\text{Estimated Daily Intake (μg/kg bw/day)} = \frac{\text{Average daily food intake} \times \text{Concentration of migrated substances}}{\text{Average weight per person}}$$

As there were no available data on the average daily food intake for delivered food, the average daily food intake of 1.5 kg per person taken from the 2018 National Health and Nutrition Survey was used, while the average weight per person was set to 60 kg based on the results of the National Health and Nutrition Survey. The concentration of migrated substances was calculated by multiplying the concentration of substances migrated (M) and the consumption

factor (CF). The concentration of substances migrated (M) was calculated using the following formula.

Concentration of substances migrated (M)
$= \sum f_T \times M_i$
$= [f_A \cdot M_A + f_B \cdot M_B + f_C \cdot M_C]$
A: Food with pH >5
B: Food with pH <5
C: Oils and fatty food

No data are available on the domestic criteria for food delivery containers for CF and food distribution factor (f_T); therefore, the values specified by US FDA were used.[21] The f_T of lead, cadmium, and arsenic was calculated as 1, since only 4 % acetic acid was used as food simulant. For M, the value for the sample with the highest migration level for each substance was used.[11] Risk was calculated by comparing the calculated estimated daily intake to the safety standard (*Table* 9). Since the provisional tolerable weekly intake (PTWI) for lead and arsenic is not currently available, the value specified by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) in 2010 was used, while the provisional tolerable monthly intake (PTMI) from JECFA was used for arsenic, considering the biological half-life.[22-24] For bisphenol A and phenol, the tolerable daily intake (TDI) was 4 μg/kg bw/day and 500 μg/kg bw/day, respectively.[25,26] The risk of each substance was found to be slightly higher for PS compared to that of other materials. However, the risk was ≤1% in all cases, suggesting that 58 food containers and packaging for food delivery were safe for the tested compounds. The calculated risk was based on the daily food intake, which means that the actual risk of tested compounds for food delivery containers could be lower when the frequency of food delivery is considered.

4. Conclusions

This study investigated the level of migration of heavy metals, bisphenol A, and phenol in 58 food

Table 9. Estimated daily intake and risk of Pb, Cd, As, bisphenol A and phenol from food packaging materials

Packaging material	Substance	M (μg/L)	EDI (μg/kg bw/day)	Risk (%)[a]
PS	Pb	0.28	9.8×10^{-4}	0.0274
	Cd	0.02	7.0×10^{-5}	0.0084
	As	0.09	3.2×10^{-4}	0.0147
	Bisphenol A	1.25	4.4×10^{-3}	0.1094
	Phenol	6.81	2.4×10^{-2}	0.0048
PP	Pb	0.61	5.6×10^{-4}	0.0158
	Cd	0.02	2.0×10^{-5}	0.0024
	As	ND[b]		
	Bisphenol A	ND		
	Phenol	1.40	1.3×10^{-3}	0.0003
PVC	Pb	0.32	4.0×10^{-4}	0.0112
	Cd	0.05	6.3×10^{-5}	0.0075
	As	ND		
	Bisphenol A	ND		
	Phenol	0.46	5.8×10^{-4}	0.0001
PET	Pb	0.18	3.2×10^{-5}	0.0009
	Cd	ND		
	As	ND		
	Bisphenol A	ND		
	Phenol	0.24	4.3×10^{-5}	8.5062×10^{-6}

[a]Risk(%) = (Estimated Daily Intake, EDI/Tolerable Daily Intake, TDI)×100
[b]ND : not detected or below LOQ

delivery containers and packaging. Analyses results showed that lead had the highest migration level and frequency among heavy metals but none of the samples exceeded the specific migration limit. Bisphenol A was only detected in PS, while phenol was detected in all materials. These materials do not yet have regulation for bisphenol A and phenol levels, and the detected amount seems to be due to contamination during manufacturing process, suggesting that continuous monitoring and management may be necessary. Analyses for safety assessment showed that all tested compounds were low-risk and safe. The findings of this study will provide insights for establishing safety management policies for food delivery containers and packaging. In the future, with advances in knowledge about consumption patterns of delivery food, more detailed risk assessment could be conducted.

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