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Factors Affecting Lead Leaching from Microwavable Plastic Ware Made with Lead-Containing Pigments

D. INTHORN,[1]* O. LERTSUPOCHAVANICH,[1] S. SILAPANUNTAKUL,[1] D. SUJIRARAT,[2] AND B. INTARAPRASONG[3]

[1]*Department of Environmental Health Sciences,* [2]*Department of Epidemiology, and* [3]*Department of Public Health Administration, Faculty of Public Health, Mahidol University, 420/1 Rajvithi Road, Rajthewee, Bangkok 10400, Thailand*

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ABSTRACT

Although food contact polymers do not normally contain lead, it is suspected that lead may be leached from some microwavable plastic ware items made in Thailand with lead-containing pigments. The purpose of this study was to examine relationships with regard to lead leached from microwavable plastic ware. Four factors were studied: pH, heat level, extraction time, and number of repeated extractions. A total of 243 samples of microwavable plastic ware items locally manufactured in Thailand were used. This study used three pH values (3.5, 4.5, and 6.5) and three heat levels (levels 3, 6, and 9 [170, 500, and 850 W, respectively]). Acetic acid was used both as the extracting agent and for adjusting the pH. Samples were collected at each level at 1, 3, and 5 min, and the amount of leached lead was measured with an atomic adsorption spectrometer. The results of this study show that pH, heat, and extraction time affected the amount of lead leaching from microwavable plastic ware. The amount of lead leaching increased with decreasing pH but increased with increasing heat level and extraction time. On the basis of these three factors, the results of this study indicate that the pH of the extractant ($r = -0.592$, $P < 0.01$), the heat level of extraction ($r = 0.293$, $P < 0.01$), the extraction time ($r = 0.226$, $P < 0.01$), and the number of extractions ($r = -0.153$, $P < 0.01$) are related to lead leaching from microwavable plastic ware. The relationship between the pH of the extractant, the heat level of extraction, and the extraction time significantly moderated lead leaching from microwavable plastic ware ($R^2 = 0.511$, $P < 0.001$). For all factors, the amount of lead leaching was lower than the permissible level of 1 mg/liter specified by the Minister of Public Health. In conclusion, a combination of high acid, prolonged heating, and extraction time accelerated the amount of lead leaching from microwavable plastic ware, but the incidence of lead leaching was negligible.

Lead is a very toxic substance, causing both acute and chronic symptoms in humans *(7, 10, 18)*. Acute lead poisoning causes heart, blood circulation, and kidney problems and liver failure, which may result in death *(14)*. Chronic lead poisoning is usually associated with anemia and renal tubular cell abnormalities, which may give rise to the Falconi syndrome *(6)*. One possible cause of lead poisoning is the ingestion of food containing lead leached from food containers *(2, 12, 13, 15)*. Therefore, the sale of food containers made with lead-containing pigments is illegal in many countries. In the process of the production of ceramics, fire glazing is done by melting silica with metal oxide in various ratios. The metal oxide commonly used is lead oxide, which has a suitable surface tension and a low viscosity that facilitates the firing process. Moreover, decorating dyes, especially copper oxide, can increase lead solubility *(9)*. Inthorn et al. *(11)* found that the amount of lead leaching from decorated ceramic ware was 0.401 ppm, which was significantly higher ($P < 0.001$) than the amount leaching from nondecorated ware (0.025 ppm). However, in only 2 of 162 samples tested (1.23%) did lead leach out at >2.5 ppm, which is the maximum lead leaching value allowed by the Ministry of Public Health of Thailand. In addition, Sheets et al. *(19)* also found that ceramic dinnerware filled with 4% acetic acid and heated in a microwave oven for 2 to 5 min exhibited lead leaching of up to 5 mg per dish. In Malaysia, Mohamed et al. *(17)* studied ceramic utensils and found that 54.7% of the items tested exceeded the U.S. Food and Drug Administration's maximum permitted lead release values.

The microwave oven is a convenient domestic appliance for cooking and warming food. Specific containers (certain types of plastic ware, stoneware, glassware, etc.) are required for use in a microwave oven. Microwavable plastic ware is durable with regard to temperature changes and acid corrosion, which is why most people prefer it. Although food contact polymers do not normally contain lead, it is suspected that lead may be leached from microwavable plastic ware items made in Thailand with lead-containing pigments. The Food and Agriculture Organization and the World Health Organization have established a permissible chronic lead intake level of 3.0 mg per week for adults *(14)*. In Thailand, the Minister of Public Health *(16)* has established a permissible level of 1 mg/liter (1 ppm) for lead leaching from any plastic ware. Moreover, the Food and Drug Administration has stated that the amount of lead leaching from containers depends on several factors, including the type of food, the pH of the food, and cooking time and temperature.

In Thailand, few studies concerning lead leaching from microwavable plastic ware have been conducted. This study was carried out to determine the effects of pH, extraction

* Author for correspondence.

time, and heat level on the leaching of lead from pigmented microwavable plastic ware. The results of this study may help consumers to be aware of the importance of using appropriate and safe microwavable plastic ware.

MATERIALS AND METHODS

All chemicals used in this experiment were of reagent grade. Methyl isobutyl ketone and concentrated acetic acid were obtained from BDH, ammonium pyrrolidine dithiocarbarmate was obtained from Fluka, lead nitrate solution was obtained from Sigma Chemical Co., and nitric acid was obtained from Merck. All microwavable plastic ware containers used in this study were manufactured in Thailand and were judged to have strong potential for lead leaching on the basis of their bright colors.

The experiment consisted of preparation of reagents, extraction solution, and plastic ware items, lead extraction under various conditions, preparation of the extracted samples for atomic absorption spectrophotometer measurement, and, finally, statistical analysis of the results obtained.

Preparation of reagents. Ammonium pyrrolidine dithiocarbarmate reagent was prepared by dissolving 4 g of ammonium pyrrolidine dithiocarbarmate in 100 ml of Milli Q water. The solution was further purified by shaking with 100 ml of methyl isobutyl ketone in a separator funnel for 30 s. The mixture was left standing until the solution was separated into two parts. Then the lower part of the solution was collected and the upper part was discarded *(3, 11)*.

Standard lead nitrate solution (1,000 mg/liter) was used to prepare working solutions of various concentrations to produce a standard curve for analysis with an atomic absorption spectrophotometer. Acetic acid solutions of various concentrations were prepared by diluting concentrated acetic acid with pure water (Milli Q) to produce final concentrations of 1 and 4%. Dilute acetic acid and sodium hydroxide solutions (both at 0.03 N) were used to adjust the pH.

Preparation of extraction solutions. Extraction solutions used in the leaching experiment consisted of pure water (Milli Q) adjusted to pHs of 3.5 and 4.5 with dilute acetic acid (0.03 N) and adjusted to pH 6.5 with dilute sodium hydroxide (0.03 N). They were freshly prepared before use.

Preparation of microwavable plastic ware. Microwavable plastic ware was washed thoroughly with tap water to eliminate dust and other particles, rinsed with pure water (Milli Q), and dried before use. The experiment was performed in triplicate with a total of 243 samples of microwavable plastic ware.

Lead extraction method. The extraction solution (325 ml, 50% volume) was poured into microwavable plastic ware containers before being placed in a microwave oven set at specified heat levels of 3 (50 to 53°C), 6 (67 to 69°C), and 9 (75 to 77°C) (170, 500, and 850 W, respectively). Each sample was heated for 1, 3, and 5 min. When the heating process was completed, 100 ml of the extracted liquid was pipetted into a beaker, and the solution was allowed to cool to room temperature before use. All extracted solutions were adjusted to pH 2.3 ± 0.2 with 1 N nitric acid. The amount of lead leaching was measured with an atomic absorption spectrophotometer All experiments were performed in triplicate.

Preparation of samples for atomic absorption spectrophotometer measurement. One milliliter of ammonium pyrrolidine dithiocarbarmate solution was added to a 250-ml separator funnel containing 100 ml of the extracted liquid. After thorough mixing, 10 ml of methyl isobutyl ketone was added, and the mixture was shaken for 30 s and allowed to separate into two phases. The upper part consisted of the organic solvent and the lower part consisted of water. The water was discarded, and the organic solvent, which was free of water, was collected and kept in 30-ml plastic sampling bottles. The bottles were wrapped with Parafilm to prevent the evaporation of methyl isobutyl ketone. The amount of lead leaching was measured with an atomic absorption spectrophotometer *(11)*.

Statistical analysis. The experimental design was limited by a variety of specific factors, namely, pH, heat, time, and number of repeated extractions. Data analysis was carried out with one-way analysis of variance, Pearson's product-moment correlation coefficient (r), and multiple-regression analysis. The Pearson product-moment correlation coefficient was interpreted as follows: a weak relationship was indicated by $r = \pm 0.01$ to ± 0.30, a moderate relationship was indicated by $r = \pm 0.31$ to ± 0.70, and a strong relationship was indicated by $r = \pm 0.71$ to ± 0.99. A perfect relationship was indicated by $r = \pm 1.00$, and no relationship was indicated by $r = 0$ *(5)*.

RESULTS

Effects of pH on the leaching of lead from microwavable plastic ware. In the first extraction, the total amounts of lead leaching at pHs of 3.5, 4.5, and 6.5 were 0.269, 0.060, and 0.056 μg/liter, respectively, at heat level 3; 0.519, 0.074, and 0.071 μg/liter, respectively, at heat level 6; and 1.070, 0.109, and 0.099 μg/liter, respectively, at heat level 9. Thus, at all heat levels, the amount of lead leaching was decreased when the pH was increased. The amounts of lead leaching at pHs of 4.5 and 6.5 were not significantly different, as shown in Table 1. The same trend was observed with subsequent extractions, but the level of lead leaching for the first extraction was higher than that for the second and third extractions.

With extractions repeated three times, it was found that the amounts of lead leaching from microwavable plastic ware at pHs of 3.5, 4.5, and 6.5 were 0.230, 0.037, and 0.033 μg/liter, respectively, at heat level 3; 0.389, 0.043, and 0.040μg/liter, respectively, at heat level 6; and 0.861, 0.057, and 0.052 μg/liter, respectively, at heat level 9. As with the first extraction, the amount of lead leaching decreased as pH increased. It should be noted that at pH 3.5 the amount of lead leaching increased when heat levels were increased, but at pHs of 4.5 and 6.5 the amount of lead leaching remained the same even when the heat levels were increased, as shown in Table 1.

Effect of heat levels on the leaching of lead from microwavable plastic ware. For the first extraction at the three pHs (3.5, 4.5, and 6.5) and the three heat levels (3, 6, and 9), the amounts of lead leaching were 0.107, 0.139, and 0.235 μg/liter, respectively, with an extraction time of 1 min; 0.123, 0.227, and 0.409 μg/liter, respectively, with an extraction time of 3 min; and 0.155, 0.298, and 0.633 μg/liter, respectively, with an extraction time of 5 min. These results indicate that the amount of lead leaching increased when heat level increased, as shown in Table 2.

For the second extraction with the same microwavable

TABLE 1. *Amounts of lead leaching from microwavable plastic ware at various pHs and heat levels for the first, second, and third extractions*

Heat level	pH of extractant	Mean amt of lead (μg/liter) for:			
		First extraction	Second extraction	Third extraction	All extractions
3	3.5 ± 0.1	0.269	0.226	0.194	0.230
	4.5 ± 0.1	0.060	0.034	0.016	0.037
	6.5 ± 0.1	0.056	0.029	0.013	0.033
6	3.5 ± 0.1	0.519	0.366	0.281	0.389
	4.5 ± 0.1	0.074	0.037	0.018	0.043
	6.5 ± 0.1	0.071	0.033	0.016	0.040
9	3.5 ± 0.1	1.070	0.817	0.695	0.861
	4.5 ± 0.1	0.109	0.040	0.022	0.057
	6.5 ± 0.1	0.099	0.037	0.020	0.052

plastic ware at the three pHs (3.5, 4.5, and 6.5) and the three heat levels (3, 6, and 9), the amounts of lead leaching were 0.079, 0.094, and 0.106 μg/liter, respectively, with an extraction time of 1 min; 0.098, 0.135, and 0.315 μg/liter, respectively, with an extraction time of 3 min; and 0.112, 0.207, and 0.472 μg/liter, respectively, with an extraction time of 5 min. These results indicate that the amount of lead leaching increased as the heat level increased and as the extraction time increased. In addition, the level of lead leaching for the second extraction was lower than that for the first.

For the third extraction with the same microwavable plastic ware at the three pHs (3.5, 4.5, and 6.5) and the three heat levels (3, 6, and 9), the amounts of lead leaching were 0.059, 0.064, and 0.069 μg/liter, respectively, with an extraction time of 1 min; 0.080, 0.109, and 0.266 μg/liter, respectively, with an extraction time of 3 min; and 0.083, 0.143, and 0.402 μg/liter, respectively, with an extraction time of 5 min, as shown in Table 2. Therefore, if heat levels increase, the amount of lead leaching will increase, except with the 1-min extraction time. Furthermore, the amount of lead leaching for the third extraction was lower than that for the first and second extractions.

As shown in Table 2, with extractions repeated three times, at the three pHs (3.5, 4.5, and 6.5) and the three heat levels (3, 6, and 9), the amounts of lead leaching were 0.082, 0.099, and 0.137 μg/liter, respectively, with an extraction time of 1 min; 0.100, 0.157, and 0.330 μg/liter, respectively, with an extraction time of 3 min; and 0.117, 0.216, and 0.502 μg/liter, respectively, with an extraction time of 5 min. These results show that the amounts of lead leaching for microwave plastic ware in all experiments were <1 mg/liter, the maximum level allowed by the Ministry of Public Health *(16)*.

Effect of extraction time on the leaching of lead from microwavable plastic ware. The amounts of lead leaching for microwavable plastic ware for the first extraction at all heat levels and with extraction times of 1, 3, and 5 min were 0.350, 0.603, and 0.905 μg/liter, respectively, at pH 3.5; 0.069, 0.081, and 0.093 μg/liter, respectively, at pH 4.5; and 0.062, 0.075, and 0.089 μg/liter, respectively, at pH 6.5, as shown in Table 3. These results indicate that at pH 3.5 the amount of lead leaching increased two to threefold at extraction times of 3 and 5 min compared with that at an extraction time of 1 min. In contrast, no increase was seen with the longer extraction times at pHs of 4.5 and 6.5. A similar trend was seen with the second and third extractions.

For the three repeated extractions at all heat levels, it was found that only at pH 3.5 did the amount of lead leaching increase with increased extraction time. Almost no

TABLE 2. *Amounts of lead leaching from microwavable plastic ware at various heat levels and extraction times for all pH values for the first, second, and third extractions*

Extraction time (min)	pH of extractant	Mean amt of lead (μg/liter) for:			
		First extraction	Second extraction	Third extraction	All extractions
1	3.5 ± 0.1	0.107	0.079	0.059	0.082
	4.5 ± 0.1	0.139	0.094	0.064	0.099
	6.5 ± 0.1	0.235	0.106	0.069	0.137
3	3.5 ± 0.1	0.123	0.098	0.080	0.100
	4.5 ± 0.1	0.227	0.135	0.109	0.157
	6.5 ± 0.1	0.409	0.315	0.266	0.330
5	3.5 ± 0.1	0.155	0.112	0.083	0.117
	4.5 ± 0.1	0.298	0.207	0.143	0.216
	6.5 ± 0.1	0.633	0.472	0.402	0.502

TABLE 3. *Amounts of lead leaching from microwavable plastic ware at various extraction times and pHs and at all heat levels for the first, second, and third extractions*

		Mean amt of lead (μg/liter) for:			
pH of extractant	Extraction time (min)	First extraction	Second extraction	Third extraction	All extractions
3.5 ± 0.1	1	0.350	0.220	0.165	0.245
	3	0.603	0.477	0.420	0.500
	5	0.905	0.712	0.585	0.734
4.5 ± 0.1	1	0.069	0.032	0.015	0.039
	3	0.081	0.037	0.019	0.046
	5	0.093	0.042	0.022	0.052
6.5 ± 0.1	1	0.062	0.027	0.012	0.034
	3	0.075	0.034	0.016	0.042
	5	0.089	0.038	0.020	0.023

change was seen when the extractant had pHs of 4.5 and 6.5, as shown in Table 3.

Effect of repeated extractions on the leaching of lead from microwavable plastic ware. For all pHs, heat levels, and extraction times, the amounts of lead leaching for the first, second, and third extractions were 0.258, 0.180, and 0.142 μg/liter, respectively. Thus, the amount of lead was highest at the first extraction.

Statistical analysis of results on the leaching of lead from microwavable plastic ware. Table 4 presents the means, standard deviations, and Pearson product-moment correlation coefficients for lead leaching amount, pH, heat level, extraction time, and number of repeated extractions. An examination of Table 4 reveals that there is no significant correlation among the variables.

As expected, the amount of lead leaching was statistically significant for pH ($r = -0.592$, $P < 0.01$), heat level ($r = 0.293$, $P < 0.01$), and extraction time ($r = 0.226$, $P < 0.01$) but not for the number of repeated extractions ($r = -0.153$, $P > 0.01$). Therefore, it appears that the negative correlations for pH and number of repeated extractions relate to the amount of lead leaching.

The level of lead leaching for the first extraction was higher than that for the second and third extractions. Table 5 presents the results of multiple-regression analysis of the effects of pH, heat level, and extraction time on the leaching of lead from microwavable plastic ware. When multiple-regression analyses were entered into the model, the inclusion of all extraction factors made a significant difference in the regression equation. As expected, pH, heat level, and extraction time explained 51.1% ($P < 0.001$) of the variance in lead leaching amounts ($R^2 = 0.511$). Inspection of the beta weight in the regression equation shows that all of the hypotheses except hypothesis IV were supported, with each of the first three having a significant beta weight ($\beta = -0.592$, $P < 0.001$; $\beta = 0.293$, $P < 0.001$; and $\beta = 0.226$, $P < 0.001$, respectively).

DISCUSSION

The results of this study show that the amounts of lead leaching from brightly pigmented microwavable plastic ware were significantly different when pHs of the extractant were different. The amount of lead leaching increased when the pH of the extractant decreased from pH 4.5 to 3.5 but not when the pH decreased from 6.5 to 4.5. The effects of pH on lead leaching might be due to the larger amount of H^+ ions available to react with the microwavable plastic ware at low pHs, causing more lead leaching.

Our results were similar to those of Domling and Kolb *(4),* who studied the release of lead and cadmium from utensils that was caused by 4% acetic solution and acidic foods such as dairy products, wine, beer, fruit juices, and soft drinks. It was found that a cola drink and a compote

TABLE 4. *Means, standard deviations, and correlations among variables (*n *= 243)*

			r (*P* value) for variable:				
Variable	Mean	SD	1	2	3	4	5
1. Lead leaching (μg/liter)	0.193	0.311	0.000 (1.000)	−0.592 (0.000)	0.293 (0.000)	0.226 (0.000)	−0.153 (0.017)
2. pH of extractant	4.833	1.250		0.000 (1.000)	0.000 (1.000)	0.000 (1.000)	0.000 (1.000)
3. Heat level of extractant	6.00	2.45			0.000 (1.000)	0.000 (1.000)	0.000 (1.000)
4. Extraction time (min)	3.00	1.64				0.000 (1.000)	0.000 (1.000)
5. No of extractions	2.00	0.82					

TABLE 5. *Results of multiple-regression analysis* (n = 243)

Variable	R^2	b	Beta	t	Significance of t	Significance of F
pH of extractant	0.511	−0.226	−0.592	−13.072	0.000	0.000
Heat level of extraction		0.112	0.293	6.470	0.000	
Extraction time		0.086	0.226	4.994	0.000	
No. of extractions		−0.058	−0.153	−3.384	0.001	
Constant value		0.366		5.189	0.000	

contained 461.9 and 246.1% heavy metal, respectively. In another study, Harris and Elsea *(8)* found that when a glazed drinking vessel was filled with a chilled cola of pH 2.7 for 30 min, 5 mg of lead leached out.

Our results with regard to the effects of heat show that the amount of lead leaching increased when heat levels increased. This finding might be due to the acceleration of the reaction between the extractant and the microwavable plastic ware by the increased heat. This result is similar to that of Beckman and Sark *(1),* who studied the release of lead from glazed and enameled kitchen utensils. A short-term experiment was conducted by boiling the utensils three times with acetic acid (4 g/liter), and a long-term experiment was conducted by keeping vessels filled with acetic solution for 24 h at room temperature. The results showed that the boiling method tended to give higher values than did the room-temperature method. Mohamed et al. *(17)* also studied ceramic utensils as sources of lead poisoning. Ceramic vessels were filled with 4% acetic acid solution and kept at room temperature (30 to 32°C) for 24 h. Spoons were completely immersed in the minimum amount of 4% acetic in a beaker, and after 24 h the results showed that the lead leaching out of 54.7% of the items tested exceeded the maximum values permitted by the U.S. Food and Drug Administration.

As expected, the amount of lead leaching increased when the extraction time was increased. This result was due to the fact a that longer contact time would better allow the reaction between the extractant and the microwavable plastic ware to occur.

The mean values of lead leaching with repeated extractions were not significantly different. This finding did not support the hypothesis that the amount of lead leaching from microwavable plastic ware for the first extraction would exceed those for the second and third extractions. This result might be due to differences in the production processes for microwavable plastic ware made from polypropylene, which is the lightest thermoplastic and has excellent properties of flexibility and toughness over a wide range of temperatures, resulting in slightly different amounts of lead leaching.

CONCLUSIONS

Although food contact polymers do not normally contain lead, it is suspected that lead may be leached from some brightly pigmented microwavable plastic wares that are produced in Thailand. In this study, the amount of lead leaching from microwavable plastic ware increased when the pH of the extractant decreased; in contrast, lead leaching increased when the heat level and the extraction time increased. Moreover, analysis of variance revealed that mean amounts of lead leaching were significantly different ($P < 0.001$). However, these amounts of lead leaching were lower than the maximum level of 1 mg/liter allowed by the Ministry of Public Health *(16).*

Multiple-regression analysis revealed that the three factors analyzed (pH, heat level, and extraction time) had effects on the amount of lead leaching from microwavable plastic ware. In addition, the combination of a high acidic pH, a high temperature, and a long extraction time extracted more lead than did a combination of the first two factors.

Lead was easily leached out with a high acidic pH, a high temperature, and a long contact time and especially with a combination of these factors. Microwavable plastic ware made with lead-containing pigments should not be heated with acidic food in Thai cuisine, including tom yum, kang som, and tom klong, although the use of a microwave at low temperatures and for short times may be safe. Consumers should be made aware of the need to use appropriate microwavable plastic ware under appropriate conditions for certain kinds of food.

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