Original Article

การตรวจสอบปริมาณตะกั่วในลูกกวาดจากจังหวัดขอนแก่น และนครหลวงเวียงจันทน์ โดยวิธีอะตอมมิคแอบซอบชั่นสเปกโทรโฟโทเมตรี

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บทคัดย่อ

บทนำ: ได้ตรวจวิเคราะห์ปริมาณตะกั่วจากตัวอย่างทั้งหมด 40 ตัวอย่าง จากลูกกวาดจำนวนทั้งหมด 40 ชื่อการค้าที่แตกต่างกัน ตัวอย่างทั้งหมดนี้ได้เก็บมาจากตลาดในเขตจังหวัดขอนแก่น ประเทศไทย และนครหลวงเวียงจันทน์ ประเทศสาธารณรัฐประชาธิปไตยประชาชนลาว ตัวอย่างที่เก็บมาทั้งหมดนี้ตั้งอยู่บนพื้นฐานที่ลูกกวาดมีสีสดใสตามข้อกำหนดที่ 1881/2006 ของคณะมนตรีแห่งสหภาพยุโรปและประกาศกระทรวงสาธารณสุขของไทยที่ 98/2529 ได้กำหนดไว้ว่าระดับความปลอดภัยของตะกั่วต้องมีปริมาณไม่เกิน 0.2 และ 1.0 มิลลิกรัมต่อกิโลกรัม ตามลำดับ **วิธีดำเนินการวิจัย:** ตัวอย่างลูกกวาดจะถูกเตรียม โดยใช้เทคนิคการย่อยด้วยชุดให้ความร้อนและมีกรดไนตริกร่วมด้วย ตัวอย่างจะถูกชั่งมาด้วยน้ำหนักประมาณ 5 กรัม ใส่ไปในหลอดย่อยสารและเติมกรดไนตริก หลังการย่อยสารที่ใช้เวลาประมาณ 2 – 3 ชั่วโมง สารละลายใสที่ได้จะถูกแยกด้วยเครื่องปั่นเหวี่ยงหนีศูนย์กลาง ที่ความเร็ว 4,000 รอบต่อนาที เป็นเวลา 3 นาที และนำไปวัดปริมาณตะกั่วด้วยเครื่องอะตอมมิคแอบซอบชั่นสเปกโทรโฟโทมิเตอร์ ที่ความยาวคลื่น 283.31 นาโนเมตร ตัวอย่างเหล่านี้จะทำการวิเคราะห์ซ้ำจำนวน 3 ครั้ง **ผลการวิจัย:** จากการตรวจวัดปริมาณตะกั่วในตัวอย่างลูกกวาด 40 ตัวอย่าง พบปริมาณตะกั่วอยู่ในช่วงความเข้มข้น 0.00 – 0.21 และ 0.11 – 1.12 ไมโครกรัมต่อกรัม จากตัวอย่างที่เก็บมาจากจังหวัดขอนแก่นและนครหลวงเวียงจันทน์ ตามลำดับ พบค่าเฉลี่ยของปริมาณตะกั่วมีค่าเท่ากับ 0.07 ± 0.07 และ 0.39 ± 0.30 ไมโครกรัมต่อกรัม จากตัวอย่างที่เก็บมาจากจังหวัดขอนแก่นและนครหลวงเวียงจันทน์ตามลำดับ **สรุปผลการวิจัย:** ผลการศึกษาปริมาณตะกั่วจำนวน 40 ตัวอย่าง พบตัวอย่างจำนวน 11 ตัวอย่าง และ 20 ตัวอย่าง ที่ทำการเก็บตัวอย่างมาจากจังหวัดขอนแก่น และนครหลวงเวียงจันทน์ ตามลำดับ มีการปนเปื้อนด้วยปริมาณตะกั่ว

คำสำคัญ: ลูกกวาด, ตะกั่ว, อะตอมมิคแอบซอบชั่นสเปกโทรโฟโทเมตรี

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Determination of lead in candies from Khon Kaen province and Vientiane capital by atomic absorption spectrophotometry

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Abstract

Introduction: Lead content was determined in 40 different brands of candy samples available in local market of Khon Kaen province, Thailand, and Vientiane capital, Lao PDR. All samples were selected on the basis of their colorful appearances. The regulation 1881/2006 of the 2006 European Union Council and the announcement of The Ministry of Public Health of Thailand, 98/BE 2529 (1986), were recommended that the safety level for lead contamination should not be higher than 0.2 and 1.0 mg kg^{-1}, respectively. **Methods:** The candy samples were digested with heat block-assisted acid digestion. Samples were weighted to approximately 5 g into a vessel, to which nitric acid were added. After digestion 2-3 hrs, colorless solution was obtained by using centrifuge technique at 4,000 rpm for 3 minutes. The concentration of lead (Pb) in supernatant was determined by using atomic absorption spectrophotometer at the wavelength of 283.31 nm. These samples were analyzed in triplicate. **Results:** For the determination of lead from 40 candy samples, it was found that the lead levels was showed the range of 0.00 – 0.21 and 0.11 – 1.12 μg g^{-1} for candy samples which were collected from Khon Kaen province and Vientiane capital, respectively. The average of lead level was 0.07 ± 0.07 and 0.39 ± 0.30 μg g^{-1} for candy samples from Khon Kaen province and Vientiane capital, respectively. **Conclusion:** Determination of lead level in 40 candy samples was examined. It was found that 11 and 20 candy samples, collected from Khon Kaen province and Vientiane capital, respectively, contaminated with lead.

Keywords: Candies, Lead, Atomic absorption spectrophotometry

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Introduction

Lead has become highly toxic metal. There is an obvious toxicological impact of this metal on children since their intestinal absorption is significantly higher than in adults (Martínez *et al.*, 2012). Lead is a metal largely used in industry, mainly in the manufacturing of pigments, coatings, containers, ointments, electric batteries and even liquor. Lead exposure during childhood causes anemia, abdominal pain, neurological and adverse development effects, learning disabilities, kidney damage and hypertensions. The consumption of lead-contaminated foods is the main source of human exposure to this heavy metal (Iwegbue *et al.*, 2015).

In the present era of industrialization and development country, one concern should be the health of population. Children are the most vulnerable age group to any kind of contamination in food chain. Candies are the favourite food items of children and are often presented to them as represented of affection from their parents. Some of ingredients, milk solids, cocoa solid, hydrogenated vegetable oil, permitted emulsifier, buffering agent, edible starch, colorant and package may be the source of lead contamination (Dahiya *et al.*, 2015). In this sense, lead level in food products is controlled by the Regulation 1881/2006 of the 2006 European Union Council, which set maximum in food stuffs of 0.2 mg kg^{-1} of lead level (Martínez *et al.*, 2012).

In this study, the determination of lead content in candies (from Khon Kaen province, Thailand and Vientiane capital, Lao PDR) were investigated. For Thailand, the safety level of lead contamination should not be higher than 1.0 mg kg^{-1} (The Ministry of Public Health of Thailand, 1986). Several methods for the detection of heavy metal ions have been developed over the last few years, such as flame atomic absorption spectrophotometry (Altunay *et al.*, 2016), graphite furnace atomic absorption spectrophotometry (Acar *et al.*, 2000), inductively couple plasm (Kim *et al.*, 2008) and DNAzyme-based microarray (Zuo *et al.*, 2009). However, while the most sensitive methods reported have to resort to sophisticated systems and procedures, others suffer from instabilities of the reagents used, high cost, tedious and lengthy analysis time (Altunay *et al.*, 2016). In this study, flame atomic absorption spectrophotometric method was selected for analysis of lead level due to simple, economic and rapid.

Materials and Methods

Instrumentations

- Atomic absorption spectrophotometer, PinAAcle 900F, PerkinElmer®; USA.
- Electrodeless discharge lamp (EDL) for Pb, PerkinElmer®; USA.
- Sample preparation digestion block, SPB 100-12, PerkinElmer®; USA.
- Deionized distillated water unit, Millipore® Milli-Q; USA.

Chemicals

- Nitric acid (HNO_3), J.T. Baker; USA.
- Lead standard solution (Pb), Spectrosol® grade, BDH; UK.

Sample Collection

The 40 candy samples were selected on the basis of their colorful appearances. Candy samples were purchased form open market in Khon Kaen province, Thailand and collected from the local market in Vientiane capital, Lao PDR. Figure 1 was showed some of candy samples for this studied.

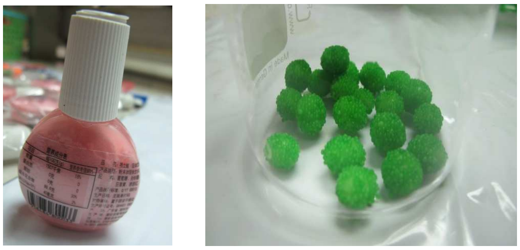
(a) (b)

Figure 1 Some of candy samples for this studied
(a) Ground candy (b) Ball candy

Sample Preparation

Each candy samples was weighted 5 g using top-loading balance and transferred into 100 ml digestion vessels. Nitric acid (40 mL, 50% v/v) were added into vessel of samples. The samples were heated by digestion block at 110 ^{o}C. After heating for 3-4 hrs, colorless solution was obtained which was leaved it to cool down at room temperature and transferred the solution into 25 ml graduated volumetric flask. Then, the total volume of solution was adjusted to 25 ml with deionized distillated water. Afterwards transferred the appropriate sample volume to the centrifuge tube. The clear solution was separated using centrifuge at 4,000 rpm for 3 minutes, then transferred supernatant to an appropriate tube and measured lead with atomic absorption spectrophotometer at the wavelength of 283.31 nm (Dahiya *et al*., 2005, Ruengsitagoon *et al.*, 2009). The optimization of analysis and experimental conditions was carried out by mean of univariate method (DeJesus *et al*., 2011, Ruengsitagoon *et al*., 2005). All candy samples were processed and digested in triplicate.

Standard of lead solution preparation

The standard of 50 µg mL^{-1} lead stock solution was prepared by pipette 5 mL of lead standard solution 1000 µg mL^{-1} into 100 mL graduated volumetric flask. The solution was adjusted volume to 100 mL with deionized distillated water. The standard curve of lead standard solution was prepared by diluting the lead standard 50 µg mL^{-1} standard solution to appropriate concentration of 0.25, 0.50, 0.75, 1.0, 2.0 and 3.0 µg mL^{-1}. The lead standard stock solution was transferred to 50 mL graduated volumetric flask in volume of 0.25, 0.50, 0.75, 1.0, 2.0 and 3.0 mL according to the working concentration, respectively. All laboratory glass wares were cleaned by soaking in a detergent solution and acidified solution, followed by washing with concentrated nitric acid and rinsed several times with deionized distilled water.

Results

Using the proposed method for determination of lead level under the optimum conditions, the linear calibration graph over the appropriate concentration range of 0.25 – 3.0 µg mL^{-} of lead standard solution was constructed. It could be expressed by the regression equation $y = 0.0193x - 0.0004$ (r^2: 0.9995; n=5) where y represents the absorption in absorbance and x is lead concentration in µg mL^{-}. Thus, the amounts of lead in sample were quantified according to the above regression line of equation. The precision of the proposed method was studied through the repeatability by measuring seven replicated of two lead standard solutions (low and high concentration of working calibration curve) at 1.0 and 3.0 µg mL^{-1}. The percentage recoveries of 1.0 and 3.0 µg mL^{-1} of lead solution were found to be 104.50 % and 97.25 % (n=7), respectively.

The 40 candy samples were investigated lead content. The lead levels was showed the range of 0.00 – 0.21 and 0.11 – 1.12 µg g^{-1} for candy samples from Khon Kaen province and Vientiane capital, respectively (Table 1).

Table 1 Lead level in candy samples

Sample Code	**Candy samples from Khon Kaen**	**Sample Code**	**Candy samples from Vientiane**
			Lead level founded (µg g^{-1})
K01	0.13	V01	0.22
K02	0.15	V02	0.20
K03	0.12	V03	0.15
K04	0.16	V04	0.11
K05	ND	V05	0.21
K06	ND	V06	0.31
K07	ND	V07	0.22
K08	ND	V08	0.24
K09	ND	V09	0.47
K10	0.10	V10	0.87
K11	0.09	V11	0.70
K12	ND	V12	1.12
K13	0.05	V13	0.36
K14	0.04	V14	0.34
K15	0.21	V15	0.24
K16	0.18	V16	1.02
K17	0.13	V17	0.55
K18	ND	V18	0.19
K19	ND	V19	0.16
K20	ND	V20	0.15

ND : None detected

It was found that 1 and 15 candy samples,collected from Khon Kaen province and Vientiane capital, respectively, were shown the lead level higher than the Regulation 1881/2006 of the 2006 European Union Council (0.2 μg g^{-1}). Interestingly, it was found that only 2 candy samples were obtained from Vientiane capital had the lead level higher than the Announcement 98/BE2529 (1986) of the Ministry of Public Health, Thailand (1.0 mg kg^{-1}). The average concentration of lead level was 0.07 ± 0.07 and 0.39 ± 0.30 μg g^{-1} for candy samples from Khon Kaen province and Vientiane capital, respectively (Table 2).

Table 2 Analysis characteristics of lead level

Analytical Characteristics	**Candy samples from Khon Kaen**	**Candy samples from Vientiane**
Sample (*n*)	20	20
*Pb level higher than 0.2 μg g^{-1} (*n*)	1 (5%)	15 (75%)
**Pb level higher than 1.0 μg g^{-1} (*n*)	none	2 (10%)
Pb in range of (μg g^{-1})	0.00 – 0.21	0.11 – 1.12
Average of Pb level (μg g^{-1})	0.07 ± 0.07	0.39 ± 0.30

*Regulation 1881/2006 of the 2006 European Union Council

**Announcement 98 /BE2559 (1986) of the Ministry of Public Health, Thailand

Discussions and Conclusion

Determination of lead content in 40 candy samples was examined. It was found that 11 and 20 candy samples, collected from Khon Kaen province and Vientiane capital, respectively, contaminated with lead. The highest of lead level was shown to be 0.21 and 1.12 μg g^{-1} for candy samples from Khon Kaen province and Vientiane capital, respectively.

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