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Determination of Bisphenol A in Commercial Plastic Products by Using High Performance Liquid Chromatography with UV/Vis Detector

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تحديد ثنائي الفينول أ في المنتجات البلاستيكية التجارية باستخدام كروماتوغرافيا السائل عالي الأداء مع كاشف الأشعة فوق البنفسجية/المرئية

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Abstract:
Phenolic compounds are the most common organic compounds pollutants, harmful to human health and exhibit adverse effects on the environment. Bisphenol A (BPA) is the one of phenolic compounds extensively used as additive material in common products, such as water bottles, food container, baby feeding bottles, food and beverage cans. Exposure to BPA released from such materials increases the hazard effect due to the endocrine disruptor, which can cause various health diseases, including cancer, disorders of the nervous system, and heart disease. In the present study, BPA concentration has been determined in four different samples of plastic products (PC drinking cup, PVC mineral water bottle, baby feeding bottle, and food container). For each commercial plastic product, four samples were bought from local markets. 1g of each plastic product piece was exposed to ethanol in a conical flask sealed with parafilm at a constant temperature of 70 °C for 10 h. HPLC technique was successfully applied for the determination of bisphenol A in different types of plastic products, with acceptable recovery results ranging from 95.33% to 97.25%. The calibration curve was constructed with the mean of the peak area against the known concentration in the range of 1-13µM of BPA. A linear relationship equation was observed, and a correlation coefficient were $y=1.2992x+1.7246$, $R2=0.992$, respectively. Bisphenol A concentrations were 4.92 ± 0.110 µM in a PVC mineral water bottle, 2.49 ± 0.105 µM in a PC plastic drinking cup, 2.66 ± 0.121 µM in a food container, and 1.26 ± 0.049 µM in a baby feeding bottle. These findings suggest that the HPLC technique used in the present work has promising potential for the detection of bisphenol A in plastic samples for industrial quality control and environmental monitoring of pollution.

Keywords: : Bisphenol A, HPLC, plastic products, human health.

الملخص:
تُعدّ المركبات الفينولية من أكثر الملوثات العضوية شيوعًا، وهي ضارة بصحة الإنسان ولها آثار سلبية على البيئة. يُعدّ ثنائي الفينول (BPA) أحد المركبات الفينولية التي تُستخدم على نطاق واسع كمادة مضافة في منتجات شائعة، مثل قنينات المياه، وعلب الطعام، وزجاجات رضاعة حليب الأطفال، وعلب الطعام والمشروبات. يزيد التعرّض لـ BPA المنبعث من هذه المواد من خطر الإصابة بأمراض مختلفة، نظرًا لتأثيره المُخلّ بالهرمونات، والذي قد يُسبّب أمراضًا صحية متنوعة، بما في ذلك السرطان، واضطرابات الجهاز العصبي، وأمراض القلب. في هذه الدراسة، تمّ تحديد تركيز BPA في أربع عينات مختلفة من المنتجات البلاستيكية (كوب شرب من البولي كربونات، و قنينات المياه المعدنية من البولي فينيل كلوريد، ورضاعة حليب الأطفال ، وعلب الطعام). تمّ شراء أربع عينات من كل منتج بلاستيكي تجاري من الأسواق المحلية. عُرّض غرام واحد من كل قطعة من المنتج البلاستيكي للإيثانول في دورق مخروطي مُحكم الإغلاق بغشاء بارافين عند درجة حرارة ثابتة 70 درجة مئوية لمدة 10 ساعات. تم تطبيق تقنية كروماتوغرافيا السائل عالي الأداء (HPLC) بنجاح لتحديد تركيز ثنائي الفينول أ في أنواع مختلفة من المنتجات البلاستيكية، مع نتائج استرداد مقبولة تتراوح بين 95.33% و97.25%. تم إنشاء منحنى المعايرة باستخدام متوسط مساحة الذروة مقابل التركيز المعروف لثنائي الفينول أ في نطاق 1-13 ميكرومول.

لوحظت علاقة خطية، وكان معامل الارتباط $y = 1.2992x + 1.7246$ ، و $R^2 = 0.992$. بلغت تراكيز ثنائي الفينول أ 4.92 ± 0.110 ميكرومول في قنينات المياه المعدنية من البولي فينيل كلوريد(PVC) ، و2.49 ± 0.105 ميكرومول في كوب شرب بلاستيكي من البولي كربونات(PC) ، و2.66 ± 0.121 ميكرومول في وعلب الطعام ، و1.26 ± 0.049 ميكرومول في ورضاعة حليب الأطفال. تشير هذه النتائج إلى أن تقنية HPLC المستخدمة في العمل الحالي لديها إمكانات واعدة للكشف عن مادة بيسفينول أ في عينات البلاستيك وذلك لأجل مراقبة الجودة الصناعية ورصد التلوث البيئي.

الكلمات المفتاحية: ثنائي الفينول أ، كروماتوغرافيا السائل عالي الأداء ،المنتجات البلاستيكية، صحة الإنسان .

1.Introduction

Bisphenol A are endocrine disrupting chemicals cause undesirable effects, a well-known substances that natural hormones release into the human body, even at low level lead to cancer or reduce immune system response. This chemical compound (BPA) play a vital role in plastic industries and used as raw material in several plastic products for example (infant bottles, can coatings, food packaging, water bottles, drug delivery, and dental sealants). Numerous studies have been reported that bisphenol A was determined in various types of the samples (tap water, drinking water, , foods, beverage containers, dental sealants, blood, and urine) [1,2,3].
The word's population exposure to BPA is related to the development of industrial area then discharge of BPA into different environmental matrices throughout industrial processing. Several counties (china and European union) have been banned the use of bisphenol A in infant bottles. 0.0.5 mg/kg body weight/day is the highest level and permissible daily amount for BPA, as recognized by the Environmental Protection Agency in the United State, and the European Food Safety Agency's permissible daily level is 0.04 mg/kg body weight/day [4-20].
Commonly, BPA was found in the plastic products and in the paints used to coat canned food. Such compounds can leach into drinking water and different types of food. As an example, the measured concentration of BPA was 13.5nM in infant feeding bottles [5-13-18]. Furthermore, due to the interaction of packing materials BPA migrated and was detected in the honey samples [6]. Bisphenol A was evaluated within the range of 0.52- 4.44 ng/ml in the women's breast milk samples [7-19]. Houng et al., (2012) informed that the concentration of bisphenol A was detected in various types of foods (17.0 ng/g in milk powder, 9.18 ng/g in fish, and 5.31 ng/g in vegetables). In addition, BPA was found in the water samples and various brands of plastic bottles manufacture from polycarbonate and polyvinyl chloride [9, 10].
Therefore, the present study aims to evaluate the levels of bisphenol A in four different samples of plastic products (PC drinking cup, PVC mineral water bottle, baby feeding bottle, and food container). The HPLC technique has been successfully applied for BPA detection in the real samples, with satisfactory recoveries.

Material and methods

Instruments

High-Performance Liquid Chromatography (HPLC)

Here, high performance liquid chromatography system with a UV/Vis detector model (Waters, 2690) has been used for analytes detection in the studied samples. Analytes separation were performed on an Eclipse C_{18} (250 mm x 4.6 mm, 5µm) column (Agilent USA) with the volume injection of 25 µL. The mobile phase was acetonitrile and 0.1% acetic acid (30:70, v/v), the column temperature was 40 °C, the flow rate was 1.0 mL/min, and the detection wavelength was 225 nm.

Chemical and Reagents

In this work, all chemicals and reagents were of analytical grade which were used without further purification and glass wares were washed at the beginning of each experimental. Bisphenol A, was supplied from Merck ((Darmstadt, Germany). Ethanol was obtained from RDH Germany. Acetonitrile (ACN) (HPLC grade from Burdick & Jackson) was used as mobile phase with the acetic acid solution. Deionized water was used throughout all the experiments.

Sample Processing for Analysis of Bisphenol A

Various kinds of plastic samples were bought from the local market (PC drinking cup, PVC mineral water bottle, baby feeding bottle, and food container). Each plastic sample was cut to small size and washed by distillation water. To obtain a real sample solution, the plastic samples were preserved as previous report with certain adjustments [11-12]. One gram of each plastic sample was put in a conical flask, and 50 ml of ethanol was added. Subsequently, the conical flask was sealed with parafilm. Next, sonication of the samples for one hour, immersed for 10 h into water bath at 70 °C, then allowed to cool at room temperature. The resulting liquid phase was filtered and concentrated to 5ml by using rotary evaporation. For further detection, BPA content in the final liquid phase was evaluated using a high performance liquid chromatography technique. A known concentration of BPA (standard solution) was added to the final liquid phase obtained to prepare spiked solutions and to carry out recovery studies.

Results and Discussion

Calibration Curve

HPLC chromatograms were obtained at various concentrations of a standard solution of bisphenol A. The calibration curve was constructed with the mean of the peak area against the known concentration in the range of 1-13μM of BPA, as shown in Figure 1. A linear relationship was obtained with the equation and a correlation coefficient of $y=1.2992x+1.7246$, $R^2=0.992$, respectively.

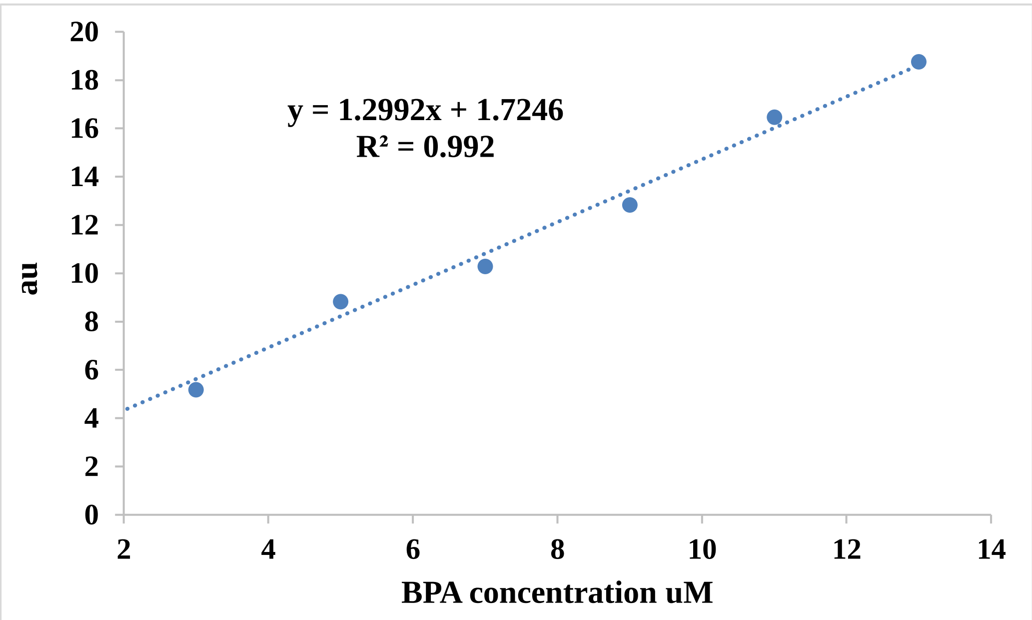

Figure 1 calibration curve obtained at various concentration of bisphenol A

Determination of bisphenol A in the plastic bottles and food container

HPLC technique was used for the detection of bisphenol A in real samples. The mean concentration of the BPA were found to be in (PVC) mineral water bottle 4.92±0.110 μM, (PC) plastic drinking cup 2.49±0.105 μM, food container 2.66±0.121 μM and baby feeding bottle 1.26±0.049 μM as shown in Table 1. The accuracy of the HPLC was evaluated by percentage recovery of bisphenol A, which was determined by adding a known concentration of the analyte to the real sample. The recovery was in the range of 95.33% to 97.25%, suggesting the HPLC method has high accuracy. The limit of detection (LOD) (defined as S/N=3, the standard deviation of the detector signal when measuring a blank), and LOD for BPA was determined to be 0.0813 μM. Moreover, the obtained results indicate that the liquid chromatography used in

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the present study has great potential for bisphenol A detection application in the practical samples.

In a previous study, Ying Qu et al., (2013) reported the BPA concentration (6.89 µM) and Ke-Jing Huang et al., (2014) found the BPA concentration (5.67 µM) in PVC mineral bottle, which is remarkably higher than our finding. In a study by Yonghong Li et al., (2016), BPA concentration (2.53 µM) was detected in PC drinking water, while Haiyu Li et al., (2016) reported that no BPA was detected in PC drinking water. Huan-shun Yin (2009) reported that BPA was found (1.59 µM) in food container samples. J. Kochana et al., 2015 reported that the highest BPA concentration (11.35µM and 3.54µM) was in the baby feeding bottle.

Table 1 Determination of BPA in commercial plastic products by HPLC

Samples	Measured value (µM) Mean ± SD	Added (µM)	Found (µM)	RSD %	Recovery %
PVC mineral water bottle	4.92±0.110	1.00	5.89	2.23	97.25
PC plastic drinking cup	2.49±0.105	1.00	3.45	4.24	96.31
Food container	2.66±0.121	1.00	3.61	4.54	95.33
Baby feeding bottle	1.26±0.049	1.00	2.23	3.87	97.12

The HPLC chromatograms of different studied samples (PC drinking cup, PVC mineral water bottle, baby feeding bottle, and food container) are shown in figure 2. In the present study, the best method that could detect the bisphenol A from the plastic bottles, a trace amount of BPA was determined in the real samples using the standard addition method.

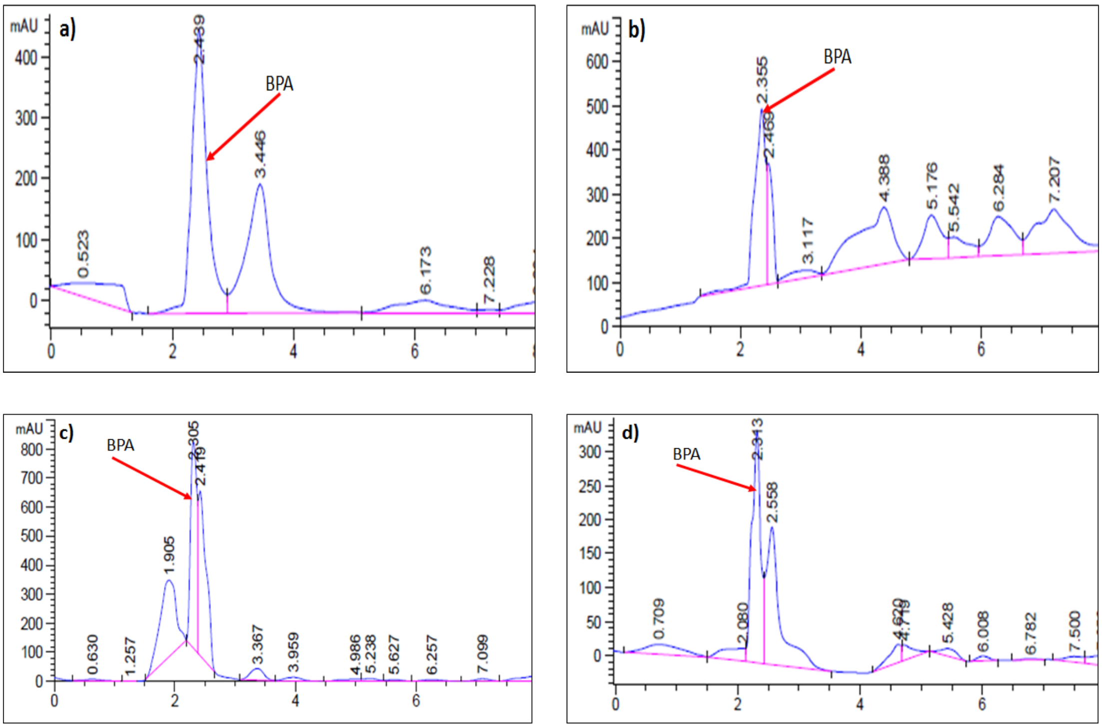

Figure 2 HPLC Chromatograms obtained by the extraction of a) PVC mineral water bottle, b) PC plastic drinking cup, c) food container and d) baby feeding bottle

Conclusion

The present study proved that the analytical method has been efficiently used for bisphenol A detection in commercial plastic products using high-performance liquid chromatography with a UV/Vis Detector. The detection limit (S/N = 3) of BPA was 0.0813 µM, and the RSD was below than 4.55%. In addition, good recoveries were obtained within the range of 95.33% to 97.25%. based on the obtained results, the HPLC method used for the determination of bisphenol A in the real samples in the present work has a simple procedure, good accuracy, and great potential for application in BPA determination in practical samples. The limitation of the present work is the extraction period for the real samples; therefore, extraction time over longer periods should be investigated. Phthalates are chemical compounds that are extensively used as plastic additives. Leaching of these compounds from the plastic into water and food containers poses toxicity risks to human health and the environment; hence, in future work, the detection of these compounds (Phthalates) should be evaluated.

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