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Determination of Bisphenol A and Alkylphenols in Soft Drinks by High-Performance Liquid Chromatography with Fluorescence Detection

Yanxin Li · Shijuan Zhang · Cuihua Song · Jinmao You

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Abstract A highly sensitive and selective method was developed for the purification and determination of bisphenol A and alkylphenols in soft drinks by using 2-(11H-benzo[a] carbazol-11-yl) ethyl chloroformate (BCEC-Cl) as pre-column labeling reagent followed by high-performance liquid chromatography (HPLC) with fluorescence detection. The HPLC sensitivity of bisphenol A and alkylphenols was greatly enhanced through the introduction of BCEC-Cl moiety with excellent fluorescence property into the target molecules. Meanwhile, the introduction of highly hydrophobic BCEC-Cl moiety into the analytes also greatly increased the hydrophobicity of the target compounds and distinguished them from hydrophilic matrices. Therefore, little interference was observed. Solid-phase extraction with C18 cartridges was applied to sample purification procedure with recoveries of higher than 82 %. When 20 mL of sample was used for analysis, the limits of quantifications of the analytes were between 0.06 and 0.1 $\mu g L^{-1}$. The proposed method was successfully applied to the determination of the target compounds in soft drink samples with a much higher sensitivity than traditional HPLC methods.

Y. Li · C. Song · J. You (✉)
Shandong Province Key Laboratory of Life-Organic Analysis, College of Chemistry and Chemical Engineering, Qufu Normal University, Qufu 273165, People's Republic of China
e-mail: jmyou6304@163.com

S. Zhang
Key Laboratory of Adaptation and Evolution of Plateau Biota, Northwest Plateau Institute of Biology, Chinese Academy of Science, Xining, People's Republic of China

S. Zhang
University of Chinese Academy of Science, Beijing, People's Republic of China

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Introduction

Bisphenol A (BPA) and alkylphenols have attracted extensive scientific, environmental, and political concerns in recent years because of their worldwide pollution and serious potential effects on humans and wildlife (Casanova-Nakayama et al. 2011; Galea and Barha 2011; Kang et al. 2006; Keri et al. 2007; Rubin 2011). BPA is widely used as an intermediate in the production of polycarbonate plastics and epoxy resins, which are applied to produce plastic food containers, inner surface coating of food, and beverage cans. 4-Nonylphenol (NP) and 4-octylphenol (OP) are widely used as intermediates to produce surfactants and as stabilizers of ethylcellulose resin, oil-soluble phenol resin, and esters. Due to their widespread use, BPA, NP, and OP have been found ubiquitously in air, water, soil, and food (García-Prieto et al. 2008; Gatidou et al. 2007; Jeannot et al. 2002; Jiménez-Díaz et al. 2010; Lin et al. 2009; Liu et al. 2008; Niu et al. 2011; Wei et al. 2012). Current estimates indicate that more than 8 billion lb of BPA are produced annually, and approximately 100 tons may be released into the atmosphere each year (Rubin 2011).

Food and drink samples may contain some of these compounds because of the pollution from raw food materials or food-making process. Migration of these compounds from packaging and bottling material is another important factor for the occurrence of these pollutants in foods. Many studies indicated that BPA, NP, and OP had been found in various kinds of food or drink samples in different countries (Cao et al. 2008; Fernandes et al. 2003; Guenther et al. 2002; Schecter et al. 2010; Yonekubo et al. 2008). Since

foods and drinks are major sources of human exposure to these compounds, many countries define tolerable daily intake (TDI) for these compounds. For example, the US Environmental Protection Agency (EPA) and European Food Safety Authority have a BPA reference TDI of 50 $\mu g kg^{-1}$ per day (European Food Safety Authority 2005; Schecter et al. 2010). To get a better understanding of the contamination status of these compounds in food samples, it is desirable to develop a sensitive and easily performable food analytical method. Because of the weak fluorescence or ultraviolet property of these compounds, direct LC methods were rarely applied in the analysis of them. Most of the methods applied in the determination of BPA, OP, and NP are gas chromatography–mass spectrometry (GC–MS) (Chang et al. 2005; Gatidou et al. 2007; Lin et al. 2009) or liquid chromatography–mass spectrometry (LC–MS) methods (Ferrer et al. 2011; Maragou et al. 2006; Schmitz-Afonso et al. 2003; Wang 2009). The sensitivity of the LC–MS method is often restricted as phenolic compounds exhibit low ionization efficiency in the MS ion chamber (Higashi and Shimada 2004; Li et al. 2005; Salvador et al. 2007). Furthermore, the methods of GC–MS and LC–MS are expensive and not accessible in ordinary laboratories.

Sensitive HPLC analysis of phenolic compounds could be achieved by introducing a suitable fluorophore or chromophore into the target molecules (Xu et al. 2010; Zhang et al. 2012). The aim of this paper is to develop a highly selective and sensitive HPLC method for the determination of BPA, OP, and NP in soft drink samples. Since most interferences in soft drink samples are water soluble, it is desirable to increase the hydrophobicity of target compounds and distinguish them from hydrophilic matrices. 2-(11H-Benzo[a]carbazol-11-yl) ethyl chloroformate (BCEC-Cl) with high hydrophobic property was therefore chosen as labeling reagent to increase the hydrophobicity of the target compounds. The sensitivity of the proposed method was also greatly enhanced due to the introduction of BCEC-Cl with excellent fluorescence property into the target molecules.

Experimental

Chemicals and Reagents

Analytical standards of OP, NP, and BPA were all obtained from Dr. Ehrenstorfer (Ausburg, Germany) with purity higher than 99 %. Methanol, dichloromethane, ethyl acetate, n-hexane, and acetonitrile were of HPLC grade (Shandong Yuwang Industrial Co., Ltd., China). Water was purified on a Milli-Q system (Millipore, Bedford, MA, USA). All other reagents used were of HPLC grade or at least of analytical grade. ODS C18 cartridges (500 mg, 6 mL) were obtained from Chrome Expert (CA, USA), and Oasis HLB cartridges (60 mg, 3 mL) were obtained from Waters (Milford, MA, USA), respectively. BCEC-Cl was prepared according to the method previously described by You et al. (2007).

Individual stock solutions of 100 $mg L^{-1}$ for all compounds were prepared in HPLC-grade acetonitrile and stored at 4 °C in the dark. Standard solutions containing all compounds were mixed and diluted with acetonitrile. Working solutions for all compounds were prepared by appropriate dilution of the stock solutions on the day of analysis.

The derivatizing reagent solution ($1.0\times10^{-3} mol L^{-1}$) was prepared by dissolving 8.1 mg BCEC-Cl in 10 mL of anhydrous acetonitrile. When not in use, all reagent solutions were stored at 4 °C in a refrigerator. To avoid the contamination of NP, OP, and BPA, glass syringes and glass tubes were employed throughout the experiments. Each tube was rinsed sequentially with tap water, high-purity water, and methanol prior to sample addition.

Sample Collection and Preparation

Eight kinds of soft drink products were purchased from a local store in Qufu City. They were all stored in cans or plastic bottles. All samples were stored at room temperature before analysis. Soda drink samples were degassed by sonication before analysis. Samples were adjusted to pH3.0 with 6 M HCl solution to ensure that all the compounds existed in their molecular form. The sample volume applied for analysis was 20 mL. For spiked sample analysis, blank tea drink samples were acidified to pH3. Then, 100 μL of standard solutions containing certain amount of analytes was added in 20 mL of blank tea drink samples with a glass syringe. The spiked samples were mixed by tumbling and then stood at room temperature for at least 30 min before analysis. Samples (including spiked samples) were then poured onto the C18 solid-phase extraction cartridges previously conditioned with 10 mL of methanol and 10 mL of water. After washing the cartridges with 10 mL of deionized water–methanol (9:1), the cartridges were dried under vacuum for 5 min; then, the analytes were eluted with 5 mL of n-hexane and 5 mL of methanol at a flow rate of 2 mL min^{-1}. The eluate was evaporated to near 1 mL under a gentle stream of nitrogen at 40 °C. It was then transferred into a 2-mL vial and further evaporated to dryness for derivatization.

Derivatization Procedure

Derivatization of the analytes with BCEC-Cl proceeded in a basic water–acetonitrile solution. One hundred microliters of $NaHCO_3$ buffer (pH10), 100 μL acetonitrile, and 50 μL BCEC-Cl acetonitrile solution were added into a 2-mL vial

containing either standard or sample solution. The vial was sealed and vortexed for 1 min and then allowed to react at 50 °C for 5 min in a water bath. Then, the mixture was cooled to room temperature, and 20 μL 50 % acetic acid solution was added to adjust the pH to lower than 7.0. Finally, the derivatized sample solution was diluted to 1 mL with water–acetonitrile (3:7, *v*/*v*) and injected directly for HPLC analysis. The derivatization scheme is shown in Fig. 1.

HPLC Analysis

The HPLC analysis was performed using an Agilent 1100 Series HPLC system, equipped with an on-line degasser, a quaternary pump, an autosampler, and a thermostated column compartment. A fluorescence detector (model G1321A, Agilent, USA) was adjusted at wavelengths of 279 and 380 nm for excitation and emission, respectively. HPLC separation was achieved on a Hypersil BDS C8 column (200×4.6 mm, 5 μm i.d., Dalian Elite Analytical Instruments Co., Ltd., China) in combination with gradient elution. Eluent A was 5 % acetonitrile in water and B was acetonitrile. The flow rate was constant at 1.0 mL min^{-1}, and the column temperature was kept at 30 °C. The elution conditions were as follows: 70–100 % B from 0 to 8 min and then held for 4 min. The column was equilibrated with the initial mobile phase for 5 min before the next injection. The injection volume was 10 μL.

Quantification

Quantitative analysis was carried out by a series of injections of target compounds in the concentration range of 1.0–200 μg L^{-1} for BPA and 2.0–200 μg L^{-1} for OP and NP. A calibration curve was constructed for each compound by plotting peak area versus concentration. All target compounds from extracted soft drink samples were measured using the external standard method.

Result and Discussion

Sample Extraction and Purification

The concentrations of the target compounds in soft drinks are usually lower than the detection limits of most methods. Therefore, enrichment procedure is indispensable. The most often used HLB and C18 cartridges were studied in this method. HLB cartridges provided satisfactory recovery for BPA, but the recoveries for OP and NP were lower than 60 %. The low recoveries of OP and NP on HLB cartridges were also reported by other authors (Beck et al. 2005; Carabias-Martinez et al. 2004; Jeannot et al. 2002). C18 cartridges provided much better recoveries for OP and NP than HLB cartridges. However, it should be pointed out that solely elution by polar methanol or ethyl acetate solution could not obtain good recoveries for OP and NP. Less polar solutions such as n-hexane or dichloromethane solution should be applied to the sufficient elution of them, while for BPA, methanol or ethyl acetate alone was enough for the elution. Finally, 5 mL n-hexane and 5 mL methanol were used sequentially to elute the target compounds from C18 cartridges. The results were satisfying with recoveries of higher than 82 % for all the three analytes.

Optimization of Derivatization Parameters

Effect of pH on Derivatization

Buffer solutions and their pH values play an important role in pre-column derivatization. The derivatization of the analytes with BCEC-Cl was carried out in sodium bicarbonate buffer solution. The effects of pH on the derivatization were then investigated with sodium bicarbonate buffers (0.1 mol L^{-1}) in the pH range of 8.0–11. Maximum derivatization yields were obtained in the pH range of 9.5–10.5. Therefore, 0.1 M sodium bicarbonate buffer with pH of 10 was applied for all subsequent derivatizations.

Effect of BCEC-Cl Concentration on Derivatization

The concentration of the derivatizing reagent is a vital factor in pre-column derivatization. It should be sufficient enough to ensure complete reaction of the analytes. However, massive excess of derivatizing reagent is not a good choice. It is not only a waste of reagent but may also lead to the overload of the detector. The effects of BCEC-Cl concentrations in the range of 1.0×10^{-4}–2.0×10^{-3} mol L^{-1} were investigated in this paper. The results indicated that complete derivatization could be achieved when the BCEC-Cl concentration was 1.0×10^{-3} mol L^{-1}. Increasing the excess of BCEC-Cl beyond this level had no improvement on the yields of the derivatives.

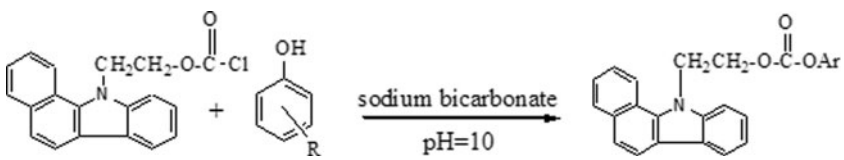

Fig. 1 The derivatization scheme of BCEC-Cl with BPA, OP, and NP

Effect of Reaction Temperature on Derivatization

Complete derivatization of BCEC-Cl with OP and NP could be achieved within 3 min at room temperature. However, the derivatization of BPA was not sufficient under this condition since it had two phenolic hydroxyl groups which could react with BCEC-Cl. The effect of reaction temperature on fluorescence intensity of BPA derivative was tested over the temperature range of 20–80 °C. The results indicated that the complete derivatization could be achieved at 50 °C for 5 min. When temperature was increased to 60 °C, no improvement or decrease in response was observed. However, when the temperature was higher than 70 °C, an obvious decrease in response was observed. This should be attributed to the fact that high temperature results in the hydrolysis of the derivatives in basic condition. Based on these results, derivatization was performed at 50 °C for 5 min with pH of 10.

HPLC Separation

Complete HPLC separation of the derivatives could be achieved on a Hypersil BDS C8 column in combination with gradient elution with water and acetonitrile as mobile phase. The three derivatives were separated within 11 min with good baseline resolution. Derivatization increased the hydrophobicity of the analytes and made them elute at increased retention times (see Fig. 2). Since most of the interferences in drink samples were hydrophilic and eluted early, the target compounds with increased hydrophobicity were therefore shifted out of the noises which were often

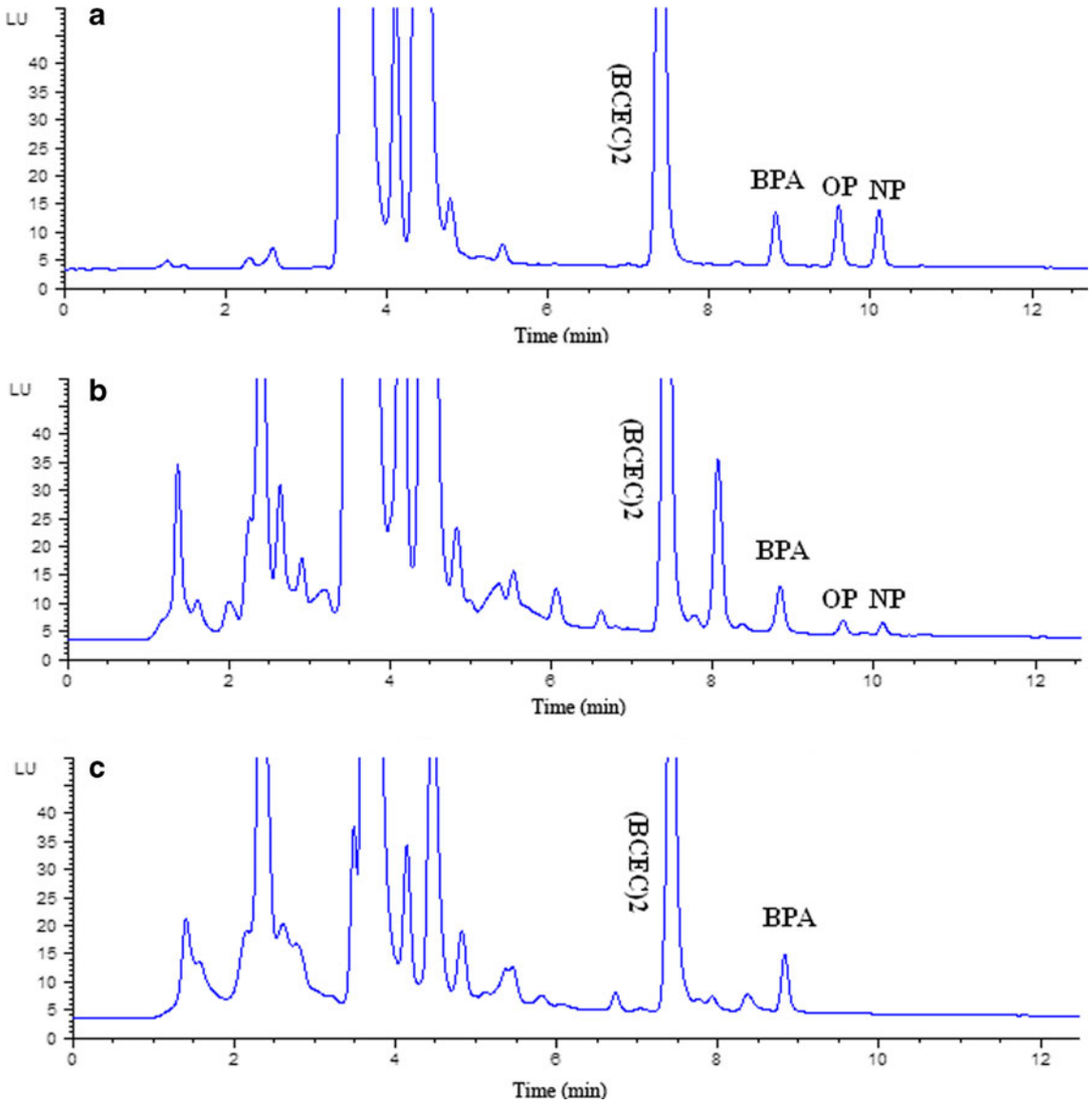

Fig. 2 Chromatograms of BPA, OP, and NP derivatives from **a** standard derivatives of BPA, OP, and NP; **b** a cola sample; and **c** a tea drink

Table 1 Recoveries and relative standard deviation of target compounds in soft drinks (n=5)

Analyte	Spiked level ($\mu g L^{-1}$)	Determined level ($\mu g L^{-1}$)	Recovery (%)	RSD (%)
BPA	0.50	0.46	90	9.6
	1.0	0.93	93	8.2
	5.0	4.7	94	7.6
OP	0.50	0.43	86	8.8
	1.0	0.92	92	7.5
	5.0	4.5	90	6.8
NP	0.50	0.41	82	8.2
	1.0	0.84	84	7.0
	5.0	4.2	84	6.4

found in HPLC analysis. As can be seen from Fig. 2, there are many interference peaks eluted before BPA, while little interference peaks are observed after the elution of BPA.

Method Validation

Linearity, Repeatability, and Reproducibility

The linear regressions of peak areas versus concentrations were fitted over the concentration range of 1.0–200.0 $\mu g L^{-1}$ for BPA and 2.0–200.0 $\mu g L^{-1}$ for OP and NP. Each standard calibration curve consisted of five points and was done on the same day of soft drink sample analysis. Good linearity was obtained for all the analytes with correlation coefficients of >0.996. For repeatability analysis, five replicates at a concentration of 1.0 $\mu g L^{-1}$ (20 $\mu g L^{-1}$ in final solution due to the method concentration coefficient of 20) were analyzed on the same day (n=5) by the same analyst. For reproducibility analysis, five replicates of 1.0 $\mu g L^{-1}$ were analyzed on three different days by different analysts. The relative standard deviations (RSDs) obtained in repeatability analysis were less than 8.0 %, while the RSDs obtained in reproducibility analysis were less than 10 %, indicating the good precision and robustness of the proposed method.

Sensitivity, Accuracy, and Stability

Limits of detection (LODs) and limits of quantification (LOQs) calculated at a signal-to-noise (S/N) ratio of 3 and 10, respectively, were used to evaluate the sensitivity of the proposed method. When 20 mL of drink sample was analyzed, the LODs for BPA, OP, and NP were 0.02, 0.03, and 0.03 $\mu g L^{-1}$, respectively. The corresponding LOQs were 0.06, 0.1, and 0.1 $\mu g L^{-1}$, respectively. Accuracy (evaluated by recovery) was measured by analyzing five spiked samples at three levels (0.5, 1.0, and 5.0 $\mu g L^{-1}$). A good degree of accuracy was achieved for the analytes with recoveries ranging from 82 to 94 % (Table 1).

The stability of BCEC-Cl and analyte derivatives was also tested. Anhydrous acetonitrile solution of BCEC-Cl could be stored at 4 °C for 1 week without obvious decrease in derivatization yields for the target compounds compared to those newly prepared BCEC-Cl solution. Sample derivatives were kept in the autosampler and repeatedly analyzed at 0, 4, 8, 12, 24, and 48 h, respectively. The relative standard deviations of peak areas were <3.2 %. Therefore,

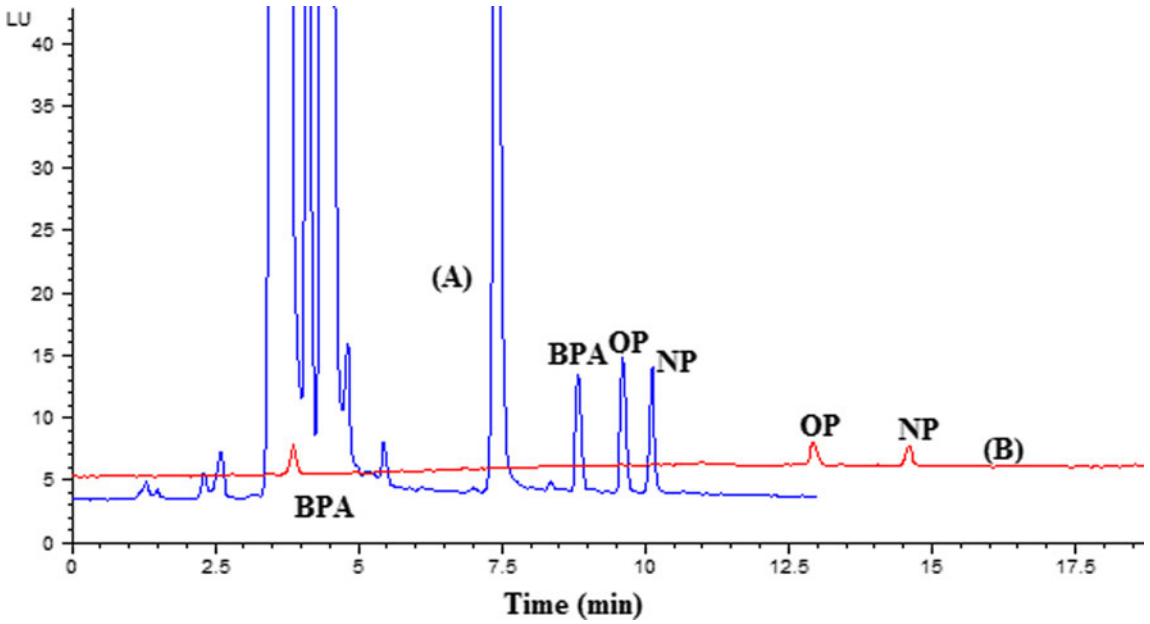

Fig. 3 Comparison of HPLC chromatograms of the analytes obtained by FL detector. *A* chromatogram of BCEC-Cl derivatives of the analytes (10, 20, and 20 $\mu g L^{-1}$ for BPA, OP, and NP, respectively); *B* chromatogram of the analytes without derivatization (1,000 $\mu g L^{-1}$)

Table 2 Comparison of the methods used before to the proposed method

Reference	Method	Sample		LOQ ($\mu g L^{-1}$)		
		Type	Amount	OP	NP	BPA
Lin et al. (2009)	GC–MS	Milk	20 g	0.03	1.0	–
Cao et al. (2008)	GC–MS	Infant formula	6 g	–	–	0.5
Schmitz-Afonso et al. (2003)	LC–MS	Egg	5 g	12	12	–
Maragou et al. (2006)	LC–MS	Milk	1 g	–	–	5
This article	HPLC	Soft drink	20 mL	0.1	0.1	0.06

– not included in the method

it can be concluded that the stability of BCEC-Cl derivatives is suitable for chromatographic analysis.

Signal Enhancement

Containing a benzene ring, BPA, OP, and NP can be determined by direct HPLC method with ultraviolet (UV) or fluorescence detection (FL). However, the sensitivity was relatively low. For example, HPLC methods with direct FL (García-Prieto et al. 2008) and UV detection (Yoshida et al. 2001) had been applied to the analysis of BPA in canned vegetables and fruits with quantification limits of 9 $\mu g kg^{-1}$ and 5 $\mu g L^{-1}$, respectively. The quantification limits of the above two HPLC methods are much higher than the 0.06 $\mu g L^{-1}$ of this method. Signal enhancement effect was also depicted by comparing the chromatogram of BPA, OP, and NP derivatives obtained in this method with that obtained by direct HPLC method with FL detection. As shown in Fig. 3, BPA was eluted at increased time, and its sensitivity was enhanced by roughly two orders of magnitude compared to that without derivatization. Besides, the sensitivity of this method was also compared with some sensitive GC–MS and LC–MS methods reported before (see Table 2). Though the proposed method was performed by HPLC, the sensitivity of this method was equivalent or superior to many GC–MS and LC–MS methods reported before. An exception was observed for OP, whose LOQ obtained by GC–MS was three times lower than this method. GC–MS showed excellent property in alkylphenols analysis. We think that is the reason why GC–MS is preferred by many researchers in the analysis of phenolic compounds. The low sensitivity of LC–MS methods may be partly attributed to the low ionization efficiency of these compounds in the MS ion chamber (Higashi and Shimada 2004; Li et al. 2005; Salvador et al. 2007).

Application

The developed method was successfully applied to the determination of BPA, OP, and NP in soft drinks. Figure 2 shows the chromatograms of a cola sample and a tea drink sample obtained by this method. BPA was found in six samples with concentrations ranging from 0.10 to 0.86 $\mu g L^{-1}$; OP was only found in one sample with a concentration of 0.25 $\mu g L^{-1}$, while NP was found in two samples with concentrations lower than 0.22 $\mu g L^{-1}$. Their concentrations were summarized in Table 3. The concentrations of BPA in soft drink products are much lower than those reported in milk or meat samples (Chen et al. 2010; Ferrer et al. 2011; Lin et al. 2009; Maragou et al. 2006) and are similar to the research done by Cao et al. (2009) in the Canada soft drink market. If an adult (60 kg body weight) consumed 500 mL of soft drink per day, the dietary intake of BPA will be less than 0.0072 $\mu g kg^{-1}$ of body weight per day based on the highest BPA level in soft drinks (0.86 $\mu g L^{-1}$), much lower than the provisional TDI of 50 $\mu g kg^{-1}$ of body weight per day established by EPA.

Conclusions

A highly sensitive and selective HPLC method with fluorescence detection was developed for the determination of BPA, OP, and NP in soft drinks. The HPLC sensitivity was greatly enhanced due to the introduction of BCEC-Cl with excellent fluorescence property into the molecules of the analytes. Derivatization also increased the hydrophobicity of the analytes, and therefore, little interferences were observed in the HPLC chromatogram. The merits of high sensitivity and little

Table 3 Concentrations of BPA, OP, and NP in soft drink samples ($n=3$)

Soft drink product	BPA ($\mu g L^{-1}$)	OP ($\mu g L^{-1}$)	NP ($\mu g L^{-1}$)
Cola A	0.86	nd	nd
Cola B	0.58	0.25	0.22
Cola C	0.12	nd	nd
Energy drink A	0.16	nd	0.14
Energy drink B	nd	nd	nd
Tea drink A	0.10	nd	nd
Tea drink B	0.60	nd	nd
Tea drink C	nd	nd	nd

nd not detectable, <LOD

interference produced by derivatization with BCEC-Cl distinguished the proposed method from the numerous methods dealing with BPA, OP, and NP in drink samples.

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