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Simultaneous determination of bisphenol A and alkylphenol in plant oil by gel permeation chromatography and isotopic dilution liquid chromatography–tandem mass spectrometry

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ABSTRACT

A simple analytical method was developed for the simultaneous analysis of bisphenol A (BPA), nonylphenol (NP) and octylphenol (OP) in plant oil. The target compounds were extracted by cyclohexane/ethyl acetate (1:1), purified by gel permeation chromatography (GPC), and analyzed by liquid chromatography–electrospray ionization tandem mass spectrometry (LC–ESI-MS/MS) in the negative ion mode. An isolator column was attached in front of the injection valve of the LC to separate background contaminants. Recovery studies were performed at three fortification levels. Mean recoveries were from 92.9% to 119.0%, with an acceptable coefficient of variation (4.4–18.5%, $n = 6$). The limits of quantification of the method were 2, 2 and 0.5 μg/kg for BPA, NP and OP, respectively. This method can be applied for screening and confirming target compounds in plant oil.

1. Introduction

The presence of bisphenol A (BPA) and alkylphenols (APs; in particular nonylphenol (NP) and octylphenol (OP)) in foods warrant great concern because these agents have endocrine-disrupting properties [1–3]. BPA is widely used in the production of polycarbonate (PC) plastics and epoxy resins. Other applications include the manufacture of polyester resin intermediates, flame retardants, and hydrogenated bisphenol A. NP and OP are used as antioxidants in plastics and rubber products. APs are precursors of, and the main degradation products of, alkylphenol ethoxylates (APEOs), which are non-ionic surfactants with emulsifying and dispersing actions. They have been widely used as industrial surfactants, detergents, plasticizers and emulsifiers. The most important APEOs are nonylphenol ethoxylates and octylphenol ethoxylates.

Due to their widespread use, BPA, NP and OP have been found ubiquitously in air, water and foods. Rudel et al. [4] detected the levels of endocrine-disrupting chemicals in 120 homes. They found that NP was present in all samples of indoor air at 21–420 ng/cm^3, while BPA and NP in household dust samples had a prevalence of detection of 86% and 80%, respectively. BPA, NP and OP in drinking water also serve as potential sources for human exposure. Li et al. [5] carried out a survey of the levels of NP and BPA in tap water in Guangzhou, China. They found NP to be present in all samples from six drinking-water plants, with the highest concentration being 1073 ng/L. In addition, 11 out of 12 samples contained BPA, with concentrations ranging from 2.3 ng/L to 317 ng/L. In addition, a wide range of physical types of food matrix has been identified in fish [6,7], animal tissues [8], milk [9–11] and cereals [12]. Guenther et al. [13] provided a comprehensive investigation of the occurrence of NP in 60 types of foodstuff in Germany. They concluded that NP was ubiquitous in foods.

For the general population, diet seems to be by far the major source of overall exposure to BPA, NP and OP. These compounds could pass into food via environmental pollution and migration by contact with plastics, resin lacquers, paints from pipes, and surfactants during the processing and storage of food [14–16]. Owing to their relatively high hydrophobicity [17,18], these compounds can readily contaminate and become concentrated in greasy foods. From a survey in Taiwan by Lu et al. [19], the levels of NP and OP in foods of animal origin were higher overall than in other foods. For BPA, the same conclusion was drawn by a Joint Food and Agriculture Organization/World Health Organization (FAO/WHO) Expert Meeting after reviewing 30 studies representing ~1000 samples from several countries [14].

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Plant oil is an important source of fat for human beings. According to the *Report of National Diet and Nutrition Survey in China, 2002*, the mean daily intake of vegetable oil in urban residents is 40 g/day [20]. Contamination of plant oil might come from (i) raw material, (ii) cleaning agents used during processing and (iii) migration from the containers. In the Chinese market, most plant oil is packed in plastic bottles; as additives of plastics and resin, BPA, NP and OP may migrate into the oil during storage. No method has been reported for the simultaneous determination of BPA, NP and OP in oil. Developing a method to determine the levels of BPA, NP and OP in oil is required.

Different methods of pretreatment have been developed and optimized to monitor the level of target compounds in food rich in fat, such as meat [8], milk [9–11] and biota samples [6,7]. For the extraction step, steam distillation [13], Soxhlet extraction [21], accelerated solvent extraction (ASE) [8], matrix solid-phase dispersion extraction (MSPDE) [11] and ultrasonic extraction [22] are frequently applied. After that, a cleanup step is usually necessary to eliminate interferences, such as lipids. Gel permeation chromatography (GPC) [6,23] and solid-phase extraction (SPE) using Flosiril cartridges [22,24] and NH_2 cartridges [8,10,25] have been studied the most as cleanup steps. A dichloromethane (DCM) extract and two-column GPC were used to discard most of the lipids in cod by Meier et al. [6].

We developed a comprehensive analytical method based on GPC preparation directly followed by a rapid LC–electrospray ionization (ESI) tandem mass spectrometry (MS) method to determine BPA, NP and OP in samples of plant oil. An extra LC column was used before the injection valve to isolate the APs and BPA from the LC system. This method was successfully used to monitor contaminant exposure originating from different types of vegetable oil.

2. Materials and methods

2.1. Chemicals and reagents

Standard BPA (98.5%) was purchased from Dr Ehrenstorfer GmbH (Augsturg, Germany). 4-NP (mixture of compounds with branched side chains) was from Tokyo Kasei Kogyo Company Limited (Tokyo, Japan). 4-OP (99%) was from Sigma–Aldrich (St Louis, MO, USA). Internal standards BPA-d_4, 4-*n*-NP-d_4 and 4-*n*-OP-d_{17} (purity >97.8%) were purchased from CDN (Quebec, Canada). All standards were stored at −20 °C. Corn oil for research and development use was purchased from Sigma–Aldrich. Ammonium hydroxide for analysis (28–30 wt% solution of NH_3 in water) was from Acros Organics (Morris Plains, NJ, USA). Sep-Pak silica and amino-propyl SPE cartridges (6 mL, 500 mg) were from Waters (Milford, MA, USA). HPLC grade of acetone, dichloromethane and cyclohexane were supplied by Fisher (Fair Lawn, NJ, USA). Ethyl acetate was from Dima and LC–MS grade of methanol and water were obtained from Merck (Darmstadt, Germany).

Individual stock solutions (1000 mg/L) were prepared by dissolving an appropriate amount of each substance in methanol and storing at −20 °C in amber glass vessels. Working standard mixtures were prepared by combining the stock solutions and diluting in methanol. The working standard solutions were stored at 4 °C.

We avoided the use of plastics; glassware was baked for 4 h at 400 °C in a muffle furnace (L9/11/B 170; Nabertherm Industrial Furnaces Limited, Lilienthal/Bremen, Germany) before use.

2.2. Sample preparation

Aliquots (0.4 g) of oil samples were spiked with 10 ng internal standards in glass tubes and diluted to 5 mL with ethyl acetate–cyclohexane solution (1:1, v/v). Samples were shaken vigorously and vortex-mixed at 2000 rpm for 1 min. Samples (5 mL) were all injected into an Accuprep MPS-GPC System (J2 Scientific, Columbia, MO, USA). This system comprised an autosampler, a solvent delivery module, an ultraviolet (UV) detector at a fixed wavelength (254 nm), a fraction collector, and an Express column containing the polymer resin styrene–divinylbenzene Biobead SX-3 (300 mm × 10 mm). Ethyl acetate–cyclohexane (1:1, v/v) was used as the mobile phase at a flow rate of 3 mL/min. The fraction containing target compounds was collected over 14–20 min in a 100-mL glass tube. After GPC, the eluate was evaporated to dryness using a rotary evaporator (Heidolph, Schwabach, Germany) at 120 rpm and 30 °C. The residue was dissolved in 1 mL methanol, and then transferred into a vial for LC–MS/MS analyses.

2.3. LC–MS/MS analyses

LC separation was undertaken using a Waters Acquity™ UPLC™ separation module with a Waters Isolator Column (P/N: 186004476) before the injection valve and a Waters Acquity UPLC BEH C18 column (2.1 mm × 50 mm; particle size, 1.7 μm) after the injection valve. The mobile phase was A (water with 0.1% ammonium hydroxide) and B (methanol) with a flow rate of 0.3 mL/min under gradient conditions: B increased linearly from 35% to 90% in 1 min, then increased to 96% in 0.5 min and held for 2 min, and finally returned to the initial composition in 0.10 min. The columns were equilibrated for 3 min before the next injection. The injection volume was 5 μL and the column oven was set at 40 °C.

MS/MS acquisition was conducted on a Waters Xevo™ TQ MS triple–quadrupole mass spectrometer equipped with an ESI interface operating in negative ion mode with multiple-reaction monitoring (MRM). The capillary voltage was 2.8 kV. Nitrogen gas (purity, 99.9%) was used as the cone gas and desolvation gas at a flow rate of 50 L/h and 800 L/h, respectively. The electrospray source block and desolvation temperatures were held at 150 °C and 400 °C, respectively. Ion energy 1 was 0.6, and ion energy 2 was 1.0. Ultra-high-purity argon was used as the collision gas. The pressure of the collision chamber was maintained at 3.2×10^{-3} mbar. For each analyte, two transitions were selected for identification, and the corresponding cone voltage and collision energy were optimized for maximum intensity. Only one transition was monitored for the internal standards.

2.4. Method validation

Calibration curves for the three target compounds were obtained by carrying out a linear regression analysis on the ratio of standard-solution areas to internal-standard areas *versus* concentration. The concentration of BPA and NP was from 0.25 μg/L to 160 μg/L; the concentration of OP was from 0.0625 μg/L to 40 μg/L. All the concentrations were spiked with 10 μg/L internal standard. Integrated peak areas of the selected quantification MRM transitions were used to construct six-point standard calibration curves. Each point on the calibration curve was obtained as the mean of three injections.

The recovery was evaluated using 0.4 g samples spiked with 2, 5 and 10 μg/kg of BPA and NP (concentrations of OP were 0.5, 1.25 and 2.5 μg/kg) and 10 ng of internal standard in six replicates. The intra-day precision and inter-day precision was evaluated by spiking oil samples at the three concentrations in six replicates mentioned above within 1 day and over the course of five consecutive days, respectively. The LOQ for each compound was calculated by determining the signal-to-noise (S/N) ratio of 10 for the quantitative ions.

Table 1
MS/MS parameters for the analysis of target compounds.

Compound	MRM transition[a]	Cone voltage (V)	Collision energy (eV)
BPA	227.1 > 212.1	32.0	28.0
	227.1 > 133.0		16.0
NP	219.2 > 133.0	34.0	30.0
	219.2 > 147.0		28.0
OP	205.2 > 106.0	34.0	18.0
BPA-d_4	231.1 > 216.1	36.0	20.0
NP-d_4	223.2 > 110.0	38.0	20.0
OP-d_{17}	222.2 > 108.0	34.0	20.0

[a] Quantitative ion transitions are underlined.

3. Results and discussion

3.1. Optimization of LC–MS/MS

The MS/MS acquisition parameters were optimized in ESI negative mode by directly infusion of standard solution (500 μg/L) via the syringe pump at a flow rate of 20 μL/min combining the mobile phase (methanol–water, 1:1, v/v) at a flow rate of 0.2 mL/min via a T-fitting prior to liquid entering the ESI source. This provided a stable response during the optimization. Diagnostic fragment ions were selected and all mass-spectrometer parameters were optimized for maximum sensitivity. Table 1 lists the characteristic ions and collision energy for each compound. Selected product ions represented the most abundant fragments observed for each precursor at the collision energy noted.

After the establishment of MS/MS parameters, the composition of the mobile phase (i.e., methanol–water and acetonitrile–water) and the concentration of ammonium hydroxide (usually employed in reversed-phase chromatography and negative ESI mode) were compared. The results suggested that good sensitivity could be achieved using methanol and water with 0.1% ammonium hydroxide, which was consistent with our previous results [26]. For the three targets, although the responses using methanol–water as mobile phase were higher than those using methanol–water containing 0.1% ammonium hydroxide, the S/N ratio was improved at different degrees using methanol–water with 0.1% ammonium hydroxide (Fig. 1). For example, it appears that S/N was only marginally improved for NP and OP (by a factor <2 in all cases). On the other hand, observed S/N ratios for BPA quantification and qualifier ions were improved by a factor of 2.4 and 4.2, respectively, when 0.1% ammonium hydroxide was present in the mobile phase.

3.2. Sample pretreatment

Several methods have been described regarding purification of the chemicals in lipid matrices. Among these purification protocols, SPE and GPC are the most commonly used. In the present study, we compared freezing lipid filtration-SPE and GPC.

Freezing-lipid filtration has been applied for the elimination of lipids in biological samples [27]. The extracted lipids can be removed due to the large difference in melting points of the target compounds and lipids. After freezing-lipid filtration, most of the remaining interference can be removed using a SPE cartridge. Two conventional normal-phase SPE cartridges (NH_2 and silica) were evaluated. In this procedure, 0.5 g of sample was transferred into a 50-mL beaker and spiked with internal standard (10 ng) and standards (10 ng). The mixture was sonicated with 10 mL methanol for 20 min, and then stored at −20 °C overnight. The supernatant was decanted into glass tubes, and the solvent evaporated to dryness under a stream of nitrogen at room temperature. The residue was reconstituted with 2× 0.5 mL hexane for SPE. NH_2 cartridges and silica cartridges were preconditioned sequentially with 10 mL acetone and 6 mL hexane. After load-

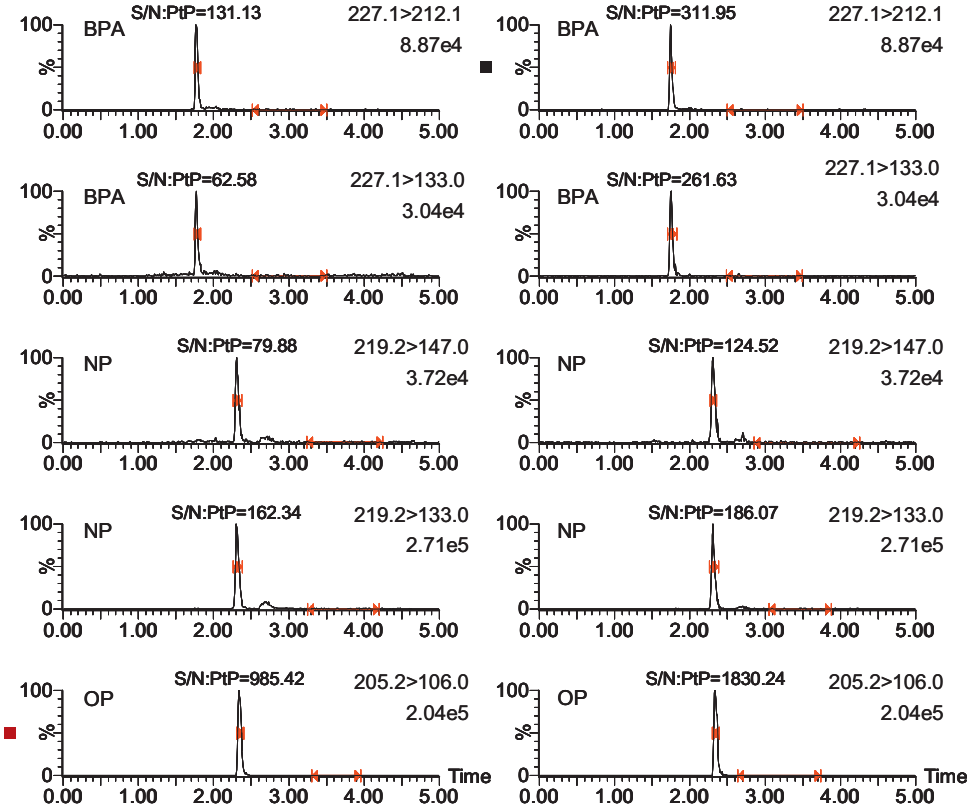

Fig. 1. Schematic diagram of the column connections.

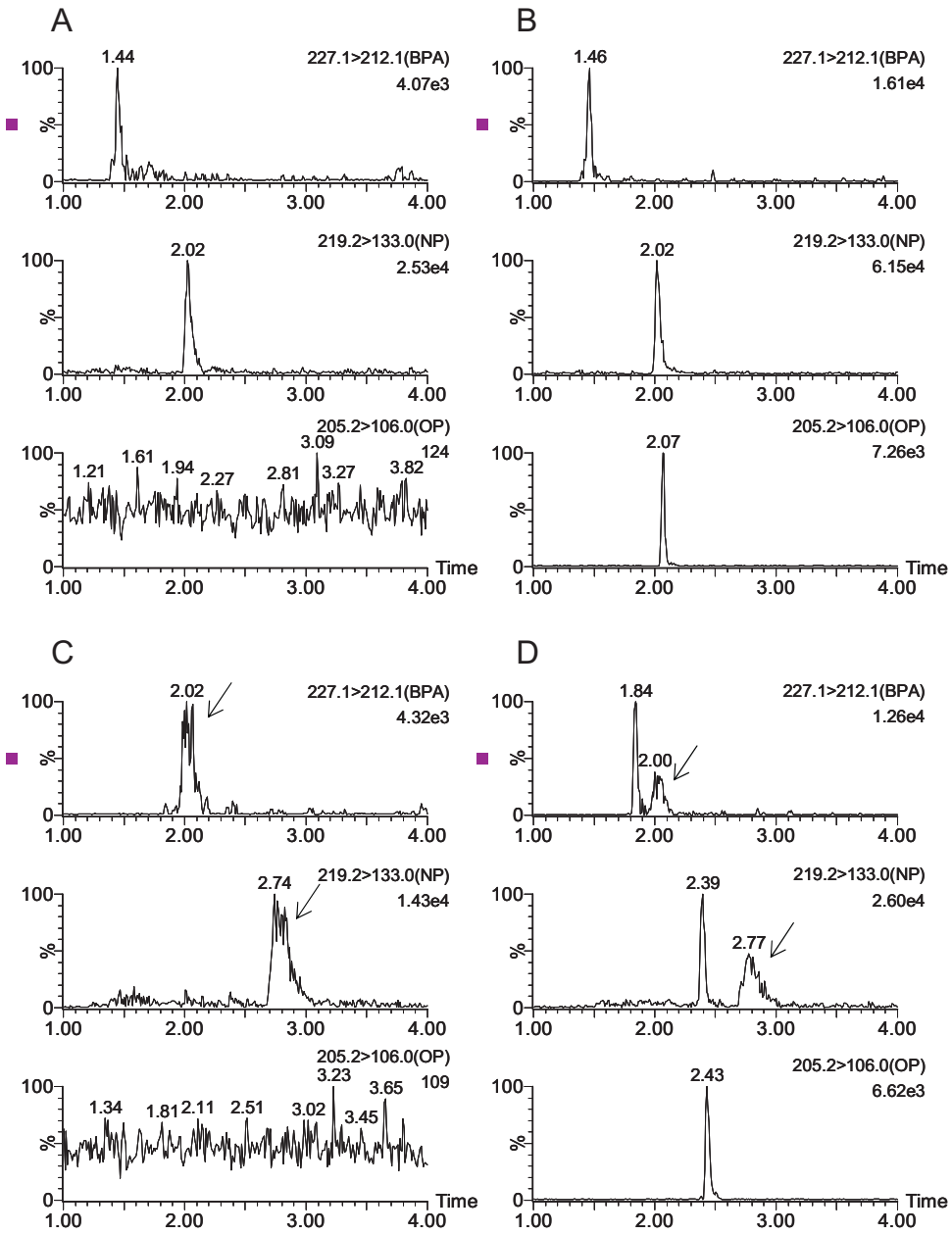

Fig. 2. LC–MS/MS chromatograms of BPA, NP and OP. (A) Zero-volume injection without an isolator column; (B) mixture standards (2, 2, and 0.5 μg/L for BPA, NP, and OP, respectively) without an isolator column; (C) zero-volume injection with an isolator column; and (D) mixture standards (2, 2, and 0.5 μg/L for BPA, NP, and OP, respectively) with an isolator column. (The arrows indicate the peaks of BPA and NP from LC system.)

ing the sample, cartridges were washed with 5 mL hexane and eluted with 5 mL acetone. Eluents were dried under a gentle stream of nitrogen and the residues reconstituted with 1.0 mL methanol for LC–MS–MS. For NH_2 cartridges and silica cartridges, the recoveries of NP and OP were <30%, and there was still an oily residue after drying, indicating that lipids were coeluted with the target compounds. We therefore changed the eluent to methanol–acetone (1:1, v/v) and dichloromethane, respectively, but the recoveries of NP and OP remained <50%. The procedure of freezing-lipid filtration after SPE was far from satisfactory because of the high lipophilicity of the analyte and lipid matrix.

GPC has been extensively used as an effective cleanup procedure for removing high-molecular-weight interferences on the basis of the difference in molecular size between interference and the analyte. A great advantage of GPC is that it is appropriate for polar and non-polar compounds. We preferred an express GPC column (300 mm × 10 mm) to save time and to conserve the solvent. Different plant oils, namely sunflower oil, linseed oil, peanut oil, soybean oil, corn oil, camellia seed oil, olive oil, rice bran oil and blend oil, were tested for GPC purification. Small amounts of free fatty acids in some oil samples with similar molecular masses as target compounds coeluted with the analytes. Nevertheless, the high response and high selectivity provided by LC–MS–MS ensured that the additional cleanup of collected GPC fractions was not necessary.

The dryness procedure was another critical step in sample preparation. Meier et al. [6] have indicated that evaporation for only 5 min after drying results in the loss of 4-*n*-OP of ~50%. Hence, in our test, rotary evaporation was immediately halted after the extracts were dried, a moderate temperature (30 °C) was used and precautions were taken to reduce evaporation.

3.3. Quality control

Quantifying trace levels of BPA and APs in samples unambiguously is a challenge because of widespread background contamination. It has been reported that ~0.02 μg/L BPA was found in Milli-Q water [28]. No matter what mobile phase was used (HPLC grade of methanol, Milli-Q water, LC–MS grade of methanol and water tested in the study), NP and BPA were detected in a zero-volume-injection, which means making a LC–MS/MS sequence run with 0 μL injection (Fig. 2A and B). Similar results were obtained using other LC–MS instruments, including Agilent 1210 series LC system equipped with an Agilent ZORBAX Extend-C18 (3.0 mm × 50 mm; 1.8 μm), followed by an Agilent 6460 series triple–quadrupole MS and an Agilent 1200 series LC system equipped with a Waters Acquity UPLC BEH C18 column (2.1 mm × 50 mm; 1.7 μm), followed by an Applied Biosystems API 3200 triple–quadrupole MS. This indicates that the background contamination prevails in all LC systems. Thus, we employed an isolator column between the in-line filter and injector to separate NP and BPA from background contamination in the sample. As shown in Fig. 2C and D, retention times of background peaks for BPA and NP (2.00 min and 2.77 min, respectively) presented somewhat shifts compared with retention times of BPA and NP from samples (1.84 min and 2.39 min, respectively). This can be ascribed to the different lengths of stationary phase interaction. At low values of %B in mobile phase, the background contaminants from LC system and target analytes from sample are concentrated at the head of the isolator column and analytical column. When %B is increased to promote elution, analytes from LC system have to interact with more stationary phase (isolator column + analytical column) than that in the sample (only analytical column). Thus, the former appears longer retention times.

The entire analytical procedure should avoid the contamination of NP and BPA as much as possible. Various quality controls were applied to ensure the accuracy of the method. To evaluate whether contamination occurs during pre-treatment, a procedural blank and a blank-matrix extract were applied in each batch of multiple test samples. There were no obvious peaks of target compounds in these blanks. Additionally, a reagent blank was injected after each five-sample injection to check for carry-over and for simple cleaning of the chromatographic system.

3.4. Method validation

In general, isotopic-dilution methods were employed to compensate for the loss of target analytes during sample preparation and for ion suppression of the mass spectrometer analysis. BPA, 4-NP and 4-OP were calibrated with BPA-d_4, 4-*n*-NP-d_4 and 4-*n*-OP-d_{17}, respectively. Acceptable linearities for all target compounds were obtained with correlation coefficients of $r > 0.99$. The LOQ was 2 μg/kg for both BPA and NP, and 0.5 μg/kg for OP.

Sample preparation was evaluated using a standard spiking test at three concentrations in corn oil applied for R&D only, with each condition carried out in six replicates. Recoveries for the entire method are reported in Table 2. The mean recoveries of each compound ranged from 92.9% to 119.0%. The reproducibility of this method was represented by the percent relative standard deviation (RSD) at each fortification level for each compound (Table 2). The precision of the method was within 20%, which is fully compatible with the requirement set by the European Union and AOAC. The inter-day precision was 7.0–18.4%, demonstrating good reproducibility of the method. Additionally, spiking test in other 8 kinds of plant oil was performed at concentration level of 5 μg/kg, 5 μg/kg and 1.25 μg/kg for BPA, NP and OP, respectively. Acceptable results were obtained (mean recoveries ranged over 91.2–119.8% and RSDs ranged over 3.4–20.0%, as shown in Table S1), indicating the method is appropriate for other plant oil.

Table 2
Recoveries and relative standard deviation of target compounds in oil ($n = 6$).

Analyte	Spiking level (μg/kg)	Recovery (%)	Relative standard deviation (%)
BPA	2	106.5	7.9
	5	101.9	10.6
	10	94.7	7.7
NP	2	98.0	10.2
	5	119.0	8.3
	10	98.9	7.4
OP	0.5	92.9	18.5
	1.25	98.1	15.3
	2.5	102.7	4.4

3.5. Method application

The proposed method was applied to the analysis of real oil samples. Twenty-one types of vegetable oil with different packaging materials, manufacturing processes and brands purchased from supermarkets in Beijing, China, were analyzed. Table 3 lists the concentration of each compound detected in these samples. BPA was not detected and OP was found in 8 samples with levels ranging from <LOQ to 10.6 μg/kg, whereas NP was detected in 17 samples at levels ranging from <LOQ to 14.8 μg/kg. Oils numbered 1–17 were packed with plastics containers and those numbered 18–21 were placed in glass bottles. From the data, we speculated that there was no migration of BPA from the packaging of oil, and that the occurrence of NP and OP could not be attributed to the migration from plastic containers. The reason why BPA was not detected may be because most of the plastic packaging of oil is made from polyethylene terephthalate (PET), which does not contain BPA. Loyo-Rosales et al. [15] studied the migration of NP from plastic containers to water and a milk surrogate. They found that neither NP nor OP could be detected in extracts from water stored in PET containers. The same conclusion was drawn by Toyo'Oka et al. [24]. NP and OP may arise from the use of pesticides and detergents, as well as the production of plastic and rubber during processing.

Several studies have been conducted on the possible association between human health and exposure to BPA, NP and OP, but

Table 3
Concentration of BPA, NP and OP in oil samples.

Sample number	BPA (μg/kg)	NP (μg/kg)	OP (μg/kg)
1	ND	<LOQ	1.0
2	ND	<LOQ	ND
3	ND	2.0	ND
4	ND	ND	ND
5	ND	3.4	ND
6	ND	2.1	ND
7	ND	5.5	ND
8	ND	8.4	5.2
9	ND	5.4	3.2
10	ND	2.0	ND
11	ND	2.2	10.6
12	ND	<LOQ	<LOQ
13	ND	3.6	ND
14	ND	<LOQ	ND
15	ND	ND	ND
16	ND	<LOQ	ND
17	ND	ND	3.8
18	ND	ND	5.8
19	ND	<LOQ	1.0
20	ND	3.8	ND
21	ND	14.8	ND

ND, not detected; LOQ, limit of quantification.

no clear conclusions have been reached. Among the toxicological investigations on NP, a lowest observed adverse effect level (LOAEL) of 15 mg/kg body weight (bw)/day was set based on the reproductive effects in an oral three-generation study [29]. The data have been widely used for the risk assessment of NP. The Danish Environmental Agency derived a tolerable daily intake (TDI) of 5 μg/kg bw/day for NP from these data [30]. Assuming that one adult consumes 40 g of plant oil per day, the maximum NP was estimated to be 9.9 ng/kg bw, which is much lower than the TDI. Therefore, the present level in oil for BPA, NP and OP is safe for human health.

4. Conclusions

A GPC cleanup followed LC–ESI-MS/MS detecting procedure was developed for the simultaneous identity and quantification of BPA, NP and OP in plant oil with excellent selectivities and sensitivities. An isolator column was successfully utilized to alleviate interference from LC–MS system. The described method was successfully applied to the detection of commercial samples.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.chroma.2011.06.005.

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