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Evaluation of Hexane Content in Edible Vegetable Oils Consumed in Iran

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Abstract

Solvent residue such as hexane in foodstuff, especially edible oil could be considered as the undesirable substances when exceeds maximum residue limit (MRL). The aim of this study is to determine the hexane content in various brands of edible oils. Totally forty samples (23 brands) of different types of vegetable oils including frying oils (n=14), blended oils (n=13), sunflower oils (n=6), corn oils (n=5) and canola oils (n=2) from Iran´s market were analyzed for hexane content using solid phase microextraction gas chromatography equipped with a flame-ionization detector (SPME)-GC-(FID). The hexane residue was detected in thirty-six out of forty examined samples, ranged from lower than LOD to 42.6 µg/kg. However, in all of them hexane content were below the MRL of 1 mg/kg which set by the European Union.

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Introduction

Edible oils and fats can be produced either by solvent or mechanical extraction of oilseeds. Hexane is one of the most commonly used solvents in the edible oil industry (1). Commercial hexane consists of some isomers of six-carbon paraffins, normally *n*-hexane and it has toxic effects (2). Long or acute exposure to haxane might cause several human health implications such as polyneuropathy, muscular weakness, headache, dizziness, giddiness, slight nausea (3, 4). It has been reported by O'Quinn *et al.* (1997) that hexane may cause some degeneration of the digestive system of animals (5). Hexane residue has been found toxic to humans and animals at relatively low concentrations as mentioned by Sparks *et al*. (2006) (6). Since refining, bleaching and deodorization are carried out as complementary processes before using oil as food products, it is likely that solvent residue approximately eliminated (7). However, a trace amount of hexane may be found in the final product. Therefore, maximum residual limit (MRL) has been established for hexane residue by various organizations. The European Union (EU) Community has set 1 mg/kg of hexane residue in vegetable oils (8). Several studies estimated hexane residue in vegetable oil (9), beeswax (10), annatto extracts (11) using headspace gas chromatography (GC).

Michulec *et al.* (2004) evaluated the presence of hexane, benzene and toluene in the vegetable oils, cardamom and vitamin A using HS- GC-FID (12). Ligor and Buszewski (2008) developed SHS-GC-FID and SPME -GC-FID methods to investigate the presence of solvents such as acetone, n-hexane, benzene, and toluene in various edible oils and they found that these methods could be successfully applied in the usual analysis of residue solvents in vegetable oils (13).

Based on our knowledge, there is not a comprehensive study in order to evaluate the concentration of hexane residues in consumed edible oils in Iran. Therefore, the aim of this study was to assess the hexane residues in 40 collected edible oil samples from of Iran market.

Materials and Method

Chemical Materials

Milli-Q apparatus was utilized to supply deionized water (Billerica, MA, USA).The SPME apparatus with Carboxen (CAR)/PDMS–thickness 75 lm) coating fibers was supplied from Supelco(Bellefonte, PA, USA). The precondition of SPME fibers was done at 200°C for 10 min.

Analytical grade of hexane and chemical materials were supplied from Merck (Darmstadt, Germany).

Samples Collection and Preparation

In total, 40 oil samples (consisting of sunflower (n=6), corn (n=5), canola (n=2), frying (n=14) and blended oils (n=13)) of 24 brands were collected from Iranian market (Tehran, summer 2014). All the samples were stored at the dark room (25°C), in their original packages (Polyethylene terephthalate (PET)). The analysis all were performed in triplicate.

Hexane Measurement

The solid phase microextraction Agilent Technologies 7890A GC system (SPME)-GC-(FID) (Palo Alto, CA, USA) which was coupled to a flame-ionization detector (FID)were used to determine residual hexane according to the Ligor and Buszewski (2008), Briefly, 50 mL of oil samples were placed in the HS vials (120 mL). Isolation step was carried out at 40 ± 1°C for 30 min without stirring. After that desorption was done for 5 min by the fiber in the injection port in where the temperature was adjusted 200°C (injector position – splitless). Then the oven temperature program was started (position–split). Separation was performed using The Stabilwax (Restek) column (30 m× 0.25 mm × 0.25 µm). The oven temperature program consisted of 3 min isothermal period at 40°C, then raised at 4°C/min increase to 100°C (held for 0 min), followed by increasing the temperature by 15°C/min to 190°C/min (held 4 min). Helium (99.999% purity) was utilized as a carrier gas with a pressure of 100 kPa. The temperature of split – splitless injector and the FID detector were adjusted 200°C (13).

Result and Discussion

Hexane Concentration

Hexane is one of the most commonly used solvents not only in the edible oil industry but also in the process of herbal medicine and bioactive components production (14, 15). So, hexane may be present in trace amounts in these kinds of products. A study has previously shown that 2,5-hexanedione which is derived from the metabolism of hexane is neurotoxic (16). Therefore, various maximum residual limit (MRL) has been determined for residual solvent in these products in order to reduce or eliminate chronic toxic effects of the solvents. So, in this study the hexane residue in 40 collected edible oil samples from of Iran market was evaluated. The limit of detection (LOD) and the limit of quantification (LOQ) were 3 µg/kg and 5µg/kg, respectively. The high correlation coefficients (0.981) indicated that there is a good linear behavior in the tested range (5-100 µg/kg). In this study, analysis of 40 oil samples described that 36 of them had hexane residue and its concentration ranged from < LOD to 42.6 µ/kg (Table 1). The results showed that 35% of the samples had hexane residue ranging from 5-42.6 µg/kg, while 10% of the samples were free of hexane and any hexane residue was detected in these 4 samples. The 55% of remaining samples had hexane residue, but it value was lower that LOQ.and LOD. As can be seen in Table 1 the highest hexane content as observed in canola oil. Based on the maximum residue limit of 1 mg/kg of hexane in the vegetable oils which is set by the European Union, none of the analyzed samples exceed the limit (8). There are only a few studies about the hexane residue in vegetable oils. Commercial orujo oil samples with various percentages of virgin olive oil (5–10%) were analyzed by Peña *et al.* (2003) for the presence of residual hexane and the results showed that almost all the samples were negative in term of hexane residue (lower than LOD) and just two samples had hexane at concentration of 2 and 3 mg/kg (3). The presence of solvent residue, including hexane, benzene, and toluene has been investigated by Michulec *et al.* (2004) in the different brands of primrose oil using HS-GC-FID. The value of 0.004, 0.009 and 0.017 mg/kg were achieved as limits of detection (LOD) for hexane, benzene, and toluene, respectively. They found that in all the primrose oils, the content of benzene was lower than LOD. Hexane residue in the most of the samples was lower than LOD, and only three samples had the value of 0.13, 0.16 and 0.70 mg/kg for hexane content (12). Similarly, the content of toluene was lower than LOD in the most oil samples. Oh *et al.* (2005) found that hexane residue varied from trace amounts to 2.8 mg/kg in 87 commercial vegetable oils (9). Additionally, Ito *et al*. (2012) evaluated 23 annatto extract products for hexane residue by applying HS-GC/FID, and nearly 91% of the samples were free of hexane and only two samples containing hexane in the value of 0.6 and 0.7 mg/kg (11). Ligor and Buszewski (2008) investigated the presence of some solvents including acetone, n-hexane, benzene, and toluene in various edible oils using SPME-GC-FID and SHS-GC-FID methods. A value of 0.003 and 0.002 mg/kg were obtained for a limit of detection by SPME-GC-FID and SHS-GC-FID methods, respectively. Their results showed that the residual amount of hexane in various vegetable oils was between 0.005 –0.460 mg/kg, which was below the permitted value of Polish norm (1000 mg/kg) (13). Jeong et al. (2017) developed HS-GC/MS method to evaluate hexane residue in health functional food products. They claimed that by applying this approach, it is likely to determine hexane residue in various kinds of these commodities. Their results showed that the average content of hexane in γ-Linolenic acid, omega-3 fatty acid, and conjugated linoleic acid was 0.39, 0.45 and 0.74 mg/kg, respectively. Furthermore, they achieved the value of 0.56 and 0.76 mg/kg as the mean content of hexane residue in lecithin and phosphatidylserine, respectively (14). Based on our study and similar research that evaluated the presence of hexane in the vegetable oils, bixin-based products, and some commercial functional food products, it seems that there is no concern about hexane residue in these commodities.

Conclusion

Hexane is the solvent that is widely utilized in the process of herbal medicine and bioactive components production and oil industry. However, it may not be completely removed and hexane residue, especially more that standard limit is undesirable. In this study the content of hexane in forty samples (23 brands) of different type of vegetable oil including frying, blended, sunflower, corn, and canola oils were investigated. Although, hexane residue was determined was detected in thirty-six out of forty examined samples, hexane content was below the MRL of 1 mg/kg which set by the EU in all of oil samples.

Conflict of Interest

Table 1. Content of hexane residue in different brands of vegetable oil

Type of edible oil	Hexane residue (µg/kg)	Type of edible oil	Hexane residue (µg/kg)	Type of edible oil	Hexane residue (µg/kg)
Frying oil (Brand)		Blended oil (Brand)		Sunflower oil (Brand)	
1 (A)	<LOD	1 (A)	<LOD	1 (A)	5±0.2
2 (B)	ND	2 (B)	<LOD	2 (B)	<LOD
3 (C)	<LOQ	3 (C)	4.8±0.15	3 (C)	ND
4 (D)	9±1.44	4 (D)	<LOQ	4 (I)	<LOD
5 (E)	5±0.45	5 (E)	10.4±0.32	5 (J)	<LOD
6 (F)	<LOQ	6 (F)	<LOD	Corn oil (Brand)	
7 (G)	<LOD	7 (G)	<LOQ	1 (A)	5.5±0.15
8 (H)	5.5±0.2	8 (H)	<LOQ	2 (B)	<LOQ
9 (I)	4.6±0.15	9 (J)	<LOD	3 (P)	LOD
10 (K)	4.8±0.45	10 (N)	<LOQ	4 (Q)	5.7±0.15
11 (L)	ND	11 (O)	ND	5 (R)	18.3±0.25
12 (M)	<LOD	12 (T)	<LOD	6 (S)	6.9±0.2
13 (U)	5.4±0.15	13 (W)	<LOD	Canola oil (Brand)	
14 (V)	<LOQ			1 (A)	42.6±0.2
				2 (B)	<LOD

The authors declare there are no conflicts of interest.

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