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Qualitative Evaluation of Bottled Water Stored in Polyethylene Terephtalate Based on Organic Chemical Compounds

Avaliação Qualitativa da Água Engarrafada e Armazenadas em Frascos de Tereftalato de Polietileno com Base Compostos Químicos Orgânicos

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Abstract

Polyethylene terephtalate (PET) is commonly used for bottling drinking water. PET must be harmless in the sense of the migration potentially unsafe materials into its content. The quality determination of migrated organic chemicals in 15 bottled water stored in PET was performed by gas chromatography-mass spectrometry technique. Most of the organic chemical compounds including phthalate, alkyl phenol, higher alkene and organic acid were detected in the samples. However, no carcinogens and hormones were recognized in the analyzed waters. The most migrated compounds identified between 13 to 100% of bottled water. The findings of present study could be alarming for the food safety legislative establishments in Iran due to the existence of some organic compounds with adverse influence on human wellbeing. Further investigation is recommended to evaluate the risk assessment of the public health arising from the presence of these toxic contaminants in the bottled water consumed by the people.
Keywords: Polyethylene terephtalate; Migration; Bottled water; Iran

Resumo

Tereftalato de Polietileno (PET) é comumente usado para engarrafamento de água potável. PET deve ser inofensivo no sentido da migração materiais potencialmente inseguros em seu conteúdo. A determinação da qualidade de produtos químicos orgânicos que migraram em 15 garrafas de água armazenada em PET foi realizada pela técnica de cromatografia gasosa acoplada a espectrometria de massa. A maior parte dos compostos químicos orgânicos, incluindo ftalato, alquil fenol, alceno de maior peso molecular e ácido orgânico foram detectados nas amostras. No entanto, não há agentes cancerígenos e hormônios foram detectados nas águas analisadas. Os compostos migraram identificados entre 13 a 100% de água engarrafada. As conclusões do presente estudo poderia ser alarmante para os estabelecimentos legislativos de segurança alimentar no Irã devido à existência de alguns compostos orgânicos com influência negativa no bem-estar humano. Outras investigações é recomendado para avaliar a avaliação de risco da saúde pública decorrente da presença desses contaminantes tóxicos na água engarrafada consumida pelas pessoas.
Palavras-chave: Tereftalato de polietileno; Migração; Garrafa d'água; Irã

1 Introduction

The production of water stored in Polyethylene terephtalate (PET) containers is steadily growing up in last decades because of the shortage in safe drinking tap water (Nawrocki *et al.,* 2002; Leivadara *et al.,* 2008). Regarding with the consumption increment of this product, paying attention to the quality of bottled water is necessary for consumer health (Dévier *et al.,* 2013). PET bottles can be responsible for the development of unwanted compounds that migrate into its content (Dąrowska *et al.,* 2003). Some degradation reactions may occur with high temperature between 200 and 300 °C in PET bottle manufacturing and sub-products such as organic chemical compounds appear in this process (Bach *et al.,* 2012). Ingestion of hundreds of migrated packing materials may occur every day based on the consumption of water stored in PET (Grob, 2002). These chemicals cause several serious health side-effects such as skin and sensory irritation, central nervous system depression, respiratory problems, leukemia and cancer, disturbance in kidney, liver and blood systems (Ceretti *et al.,* 2010; Bina *et al.,* 2012). Some studies have proved the existence of organic compounds in bottled water (Leivadara *et al.,* 2008; Al-Mudhaf *et al.,* 2009; Pinto and Reali 2009; Ikem, 2010; Jin *et al.,* 2010; Amiridou and Voutsa 2011). For this reasons, international and national authorities established regulation to monitor unwanted material migrated (somehow toxic) to water (Castle, 1994; Salehi *et al.,* 2014). In a previous study done in Isfahan region of Iran, chemical quality of 21 brands of bottled water determined, the results compared with label information on package, showed the significant difference between chemical concentration and labeling and water quality guidelines (Moazeni *et al.,* 2013). Furthermore, in a recent study nitrate and nitrite content found in bottled water in Isfahan (Moazeni *et al.,* 2014). To author's knowledge, no study has been conducted to examine the chemical organic compounds in bottled water distributed in this region. Therefore, we aimed a study to determine the qualitative evaluation of bottled water based on organic chemical compounds.

2 Materials And Methods

2.1 Sampling

The study was conducted in Isfahan, central part of Iran. This city is the most noticeable national cultural and tourist centers. A total of 15 water stored in PET bottles (two samples for each) with different brands of water packaging plants were purchased on random basis from the supermarkets and stores. The samples were opened in the Environment Research Center laboratory in Isfahan University of Medical Sciences and analyzed on the day of collection.

2.2 Chemicals and Reagents

Methanol and MTBE with GC-grade were purchased from Merck Company (Germany). A C_{18} cartridge (500 mg) (Chromabond, Germany) with silica sorbent was applied for solid phase extraction.

2.3 Sample Preparation

Solid phase extraction was applied to prepare the mineral water samples. The cartridge was conditioned by 10 ml Millipore water and 5 ml methanol (99% Merck) to eliminate possible contamination and enhance the binding between organic compound and sorbent. Water sample (100 ml) was passed through cartridge under pressure 20 inches/Hg. Silica sorbent was washed with methanol under pressure 10 inches/Hg. Finally methanol was collected in specific tubes and dried by nitrogen gas in ambient temperature. Extraction was dissolved in 100 µl MTBE solvent and injected to GC/MS for analysis. All experiments were repeated two times. Blank solution was prepared in similar way without bottled water ensuring the compounds in the chromatogram of samples not contaminated with releasing of the cartridge, other materials and equipment used for extraction. Classification of identified compounds was based on the structure similarity including PEs, APs, organic acids and higher alkenes. The group "others" in our study refers to diverse compounds that was not set in definite group.

2.4 Apparatus

The Agilent Technology system consisting of a 5975C inert MSD, triple axis, detector equipped 7890A gas chromatograph and a split/ splitless injector was used for the confirmation of organic matter in samples. A fused silica column, HP-5 ms (5% phenyl- 95% di methyl polysiloxane; 30 m × 0.25 mm I.D, 0.25 µm film thickness) was employed with helium (purity 99.995%) as the carrier gas at a flow rate of 1 ml/min. The column temperature was

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scheduled with 100°C for 1 minute, increasing rate of 10°C/ min to 210°C as well as 5°C/ min to 250°C with ultimate temperature of 280°C at 30°C/ min. Overall run time of extraction was 25 min. The MSD was operated with electron impact ionization in selected full scan mode. The injector port was maintained at 280°C with injection 3 ml of extracted sample in splitless mode within 2 min. The effluent of GC column was passed via a transfer line held at 290°C and fed into a 70eV electron impact ionization source at 280°C (Moghadam *et al.,* 2012). The data were acquired and processed by data analysis software. The glassware containers were washed and soaked with acid detergent for 24 hours and rinsed several times with distilled water to remove absorbance.

3 Results and Discussion

Results obtained in the present study revealed the existence of unwanted compounds from PET to water. In Table 1 the name of detected chemical compounds in water tested samples are listed by their chemical classification. Chemical compounds belonging to the classes of PEs material, APs, higher alkanes and organic acids were permanently identified in the examined water samples; however, a diverse group of chemicals were detected individually. The most common organic compounds detected between 13 to 100% of the samples. Some other compounds were rarely presented in few samples. PEs are esters of phthalic acid and mostly used as plasticizers in PET. Since PEs molecules are weakly bounded to polymers, they are susceptible to be liberated into water and beverages contacted with the packaging polymer (Amiridou & Voutsa, 2011). The probable consequence of phthalates within endocrine-mediated unpleasant effects in wildlife and humans is reported over the past few decades. There is a variation among the results of PET migration studies(Amiridou & Voutsa, 2011). It was found BBP, BPA, DBP, DEHP, DEP, DMP and DNOP in bottled waters stored in polycarbonate container in Greece. Iranian data are limited with the report migration of BPA in baby bottles in Isfahan urban society (Moghadam *et al.,* 2012; Moghadam *et al.,* 2013). Traces of pharmaceutical substances, alkylphenols and some phthalates including DEHP were detected in two French brands of bottled natural mineral waters. Devier *et al*., indicated the complexity of reliable measure to qualify the contamination of mineral water samples (Dévier *et al.,* 2013).

Some derivatives of APs have already been identified in the samples analyzed of our study (Table 1). These compounds generate by oxidation of an additive called TNPP or degradation of APEOs as washing agents in bottle manufacturing. The other sources of APs could be the antioxidants used in laboratory equipment and materials such as vessels, tubes, detergents, the water itself or contamination through washing stages of bottles (Bach *et al.,* 2012). For example, NP is usually spread in the environment because of its usage in industry as raw material or additive in epoxy resins to improve polymerization, drying and plasticity properties. Hebert *et al*., reported the existence of disinfection by products (N-nitrosodimethylamine, halofuranones, chlorate, formaldehyde and acetaldehyde, 2,4,6-trichlorophenol and pentachlorophenol, hydrazine and two unregulated halomethanes, dichloromethane and tetrachloromethane) with greatest potential risk like critical mutagenic, genotoxic, carincogenic and cytotoxic properties in drinking water of France. The aforementioned authors emphasized the importance of improving drinking water treatment and distribution (Hebert *et al.,* 2010). Here we found higher alkenes compounds in samples. These compounds are defined with nine or further carbon atoms. Mixes of the normal alkenes could be measured in boiling point by gas chromatography (Hillen *et al.,* 1982). We identified different kinds of carboxylic acids. We haven't found information on the contamination of bottled water with those compounds in literature review. Extant of haloacetic acids in drinking water as carboxylic acids which a hydrogen atom replaced by halogen reported (Hebert *et al.,* 2010). Furthermore it is mentioned disinfection by-products occurring during the disinfection process by the reaction of disinfectants (haloacetic acids, cyanogen halides, halogenated acetonitriles, chlorinated ketones and furanones) and organic matter in water (Niri *et al.,* 2008). VOCs in group "others" are one of the main subjects in the environment (Figure 1). The wide spread of aforementioned compounds due to highly volatile nature has raised major concerns, in particular with environmental sciences. US-EPA defines VOCs as a contributor in the creation of photochemical ozone (Chary & Fernandez-Alba, 2012). VOCs can lead to disorder in liver, kidney,

Class	Compounds	Class	Compounds
PEs	BEP DBP DEHP DOP Phthalic anhydride	**AP**	- Phenol, 4,4'-(1-methylethylidene) - 2,3-Dimethoxy-1-phenyl-5,5-dimet - (4-Hydroxyphenyl)methylphen - NP - Phenol, 2,4-bis(1-phenylethyl)
Higher alkanes	- Pentacosane - Tricosane - Docosane - Dotriacontane - Heneicosane - Hexacosane - Triacontane - Heptacosane - Hexatriacontane - Eicosane - Octadecane - Tetracosane, - Nonacosane - Pentadecane - Nonadecane - Octacosane	**Organic acids**	- 1,2-Benzenedicarboxylic acid - Hexadecanoic acid, methyl ester - n-Hexadecanoic acid - Octadecanoic acid, methyl ester - 1,2-Benzenedicarboxylic acid - Hexadecanoic acid - Phthalic acid, 2-cyclohexylethyl - Tetradecanoic acid, methyl ester - Pentadecanoic acid, methyl ester - Hexadecanoic acid, 14-methyl - 2-Propenoic acid, 3-(4-methoxyph) - Heptadecanoic acid, 16-methyl- - Nonanedioic acid, dimethyl ester - Tetradecanoic acid - Pentadecanoic acid - 9-Octadecenoic acid (Z)-, methyl - Sulfurous acid, 2-propyl tridecy - Cyclopropaneoctanoic acid, 2-hex
Others	- Cyclotrisiloxane, hexamethyl - N-ethyl-1,3-dithioisoindoline - Bistrimethtlsilyl n-acetyl eicos - 2-Ethylhexyl trans-4-methoxycinn - 2-benzoyl-6,7-dimethoxy-4-methyl - Cyclohexasiloxane, dodecamethyl - Cyclopentasiloxane, decamethyl - Cyclononasiloxane, - octadecamethyl - Iron, monocarbonyl-(1,3-butadien) - 5,6,8,9,10,11,12,13-Octahydro-4H - cis-3,4,6,9-tetrahydro-10-h - 6,12-epoxy-11.beta.-eudesma-4,6- - 7,9-di-tert-butyl-1-oxaspiro[4.5] - Methyl tetradecanoate - Methyl 4-(1-ethyldecyl)benzenesu - Isomaturunin naphtho[2,3-b]fur - Tetracosamethylcyclodecasiloxa - 1,1-Bis[(3-ethyl-5-n-pentyl)-2-f - 1,3-Xylyl-15-crown-4, 2,3-.pinan - 6-Aza-5,7,12,14-tetrathiapentacene - Octadecamethylcyclononasiloxane - Octasiloxane, 1,1,3,3,5,5,7,7,9,	**Others (Continous)**	- 5,7-Diamino-1,2,3,4-tetrahydro-1 - Heptasiloxane - Silicate anion tetramer - 10-Heneicosene (c,t) - Benzothiazole, 2-(methylthio)- - t-(4,7)-E-(7,8)-8-exo-ethenyl-8- - 1-(cyclopropacyclododecan-1-ylid - trans-7,8,9,10-Tetrahydro-7-azid - Methyl4-(1-methylundecyl)benzen - Cyclohexanone, 2-[2-nitro-1-(2-n - Ethanol, 2-(2-butoxyethoxy)-, ac - 4H-Cyclopenta[def]chrysen-4-one - Phosphetane, 2,2,3,4,4-pentameth - 2,6,10,14,18,22-Tetracosahexaene - 5,6,8,9-tetramethoxy-2-methylpep - Methyl-3-(3,5-diertbutyl-4-hydr - Squalene - Heptadecanenitrile - 1,4-dihydro-9-isopropylidene-5,6

Table 1 Chemical organic compounds detected in bottled water samples

immune, nervous and reproductive system as well as cancers and mutation (Beare-Rogers *et al.,* 2001; Kavcar *et al.,* 2006). The main subgroups of VOCs are halogenated organics, monocyclic aromatic hydrocarbons, organic sulfides, sulfoxides, acetone, esters, BTEXs, THMs and DCM (Kavcar *et al.,* 2006; Chary & Fernandez-Alba, 2012). Volatile halogenated organics for example (THMs) forms during drinking water treatment through usage of free chlorine as a disinfectant in the presence of certainly occurring humic substances (Ruiz-Bevia *et al.,* 2009). Different studies showed miscellaneous natural or manmade sources for this compound (Leivadara *et al.,* 2008). The extant of VOCs deduced

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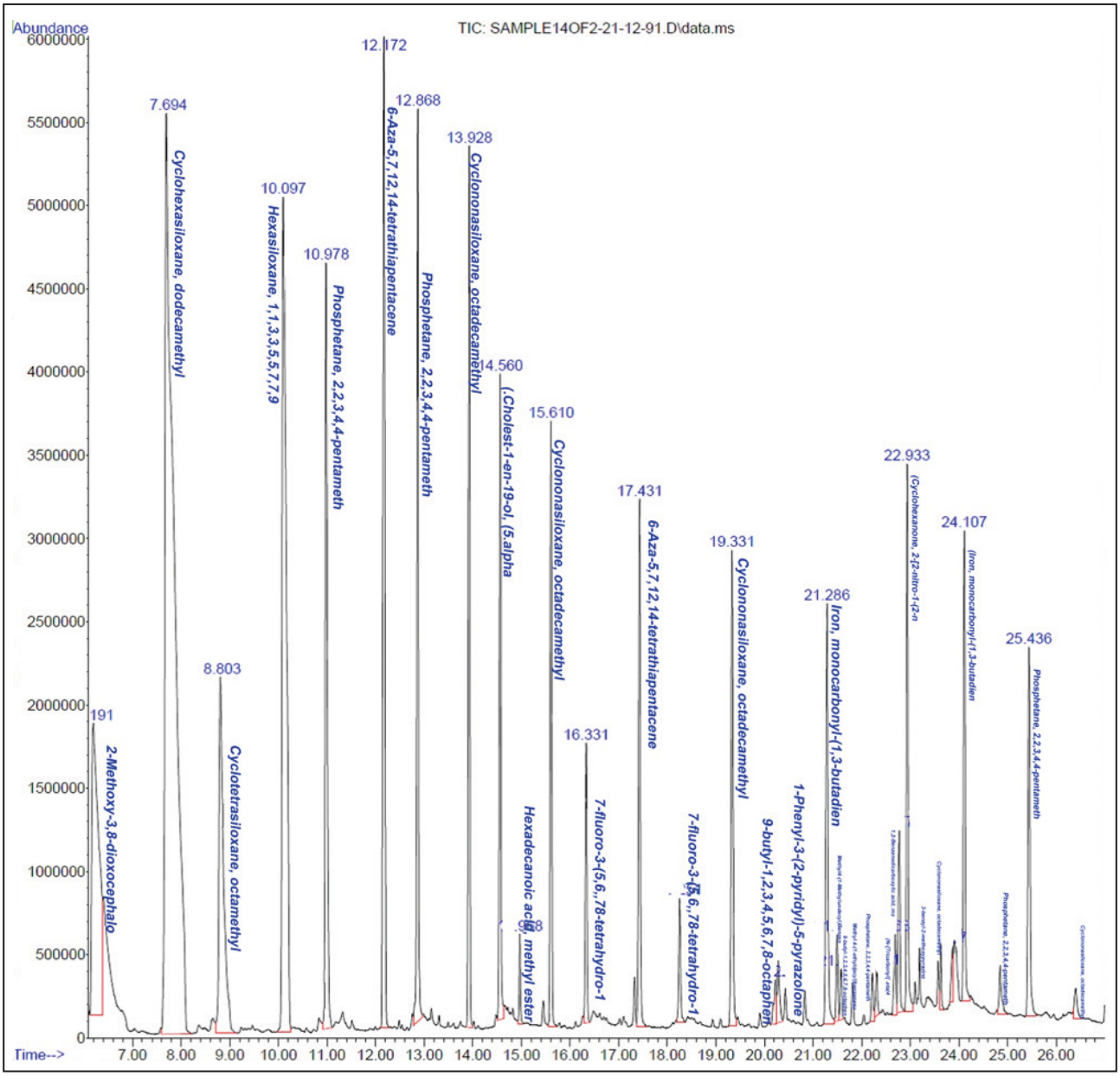

Figure 1 Chromatogram for one of the samples showing the presence of group "others" in current study

in bottle waters of Kuwait in the concentration of much lower than established as safe by WHO guidelines and US-EPA maximum contaminants levels (Al-Mudhaf *et al.,* 2009). In Greece, VOCs as well as other carcinogen and hormone disrupter PEs were not recognized in bottled drinking water. But the disinfection by product compounds such as THMs and haloacetic acids (HAAs) were detected at low concentration (Saleh *et al.,* 2001; Leivadara *et al.,* 2008; Pavón *et al.,* 2008). The presence of THMs in bottled water indicated in Turkey, Greece, U.S.A. (Leivadara *et al.,* 2008; Ikem, 2010). Kavcar *et al.* (2006) surveyed the risk assessment of VOCs identified in drinking water in Izmir of Turkey. They concluded the lack of carcinogenic affect in population of this city (Kavcar *et al.,* 2006).

4 Conclusions

In this study, examining the organic chemical compounds in mineral water stored in PET bottles showed the existence of different categories of those including PEs, AP, higher alkenes, organic acids and others. Furthermore, the results indicated

the importance of bottled water stored in PET as a source of transmission of migrated organic chemical compounds to human. More studies are needed to define the risk assessment of the public health in Isfahan society through consumption of contaminated bottled water.

5 List of Abbreviations

APs: alkyl phenols; APEOs**:** poly ethoxylated nonylphenols; BBP: butyl benzyl phthalate; BEP: Bis(2-ethylhexyl) phthalate; BPA: Bisphenol A; BTEX:benzene, toluene, ethylbenzene, and xylene; DBP: di (n-butyl) phthalate 1,2-Benzene; DCM: dichloromethane; DEHP: di (2-ethylhexyl) phthalate; DEP: diethyl phthalate; DMP: dimethyl phthalate; DNOP: di(n-octyl)phthalate; DOP: Di-n-octyl phthalate; FDA: Food and Drug Administration; GC/MS: Gas Chromatography/ Mass Spectrometry; HAAs: haloacetic acids; MTBE: methyl tert-butyl ether; NP: 4-nonylphenol; PEs: Phthalates; THMs: trihalomethanes; TNPP: tris(nonylphenyl) phosphite; USEPA: United Stated Environmental Protection Agency; VOCs: volatile organic compounds; WHO: World Health Organization

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7 Ethical Considerations

Ethical issues (Including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, redundancy, etc.) have been completely observed by the authors.

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