International Journal of Environment Volume : 04 | Issue : 02 | April-June | 2015
ISSN: 2077-4508 Pages: 165-171

Determination of Volatile Pesticides and Chemicals which Migrate into Foods from Plastic Packages

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ABSTRACT

This research studied: *Firstly*: Migration the surrounding air pollutants such as pesticides into foods during different types of packaging materials of plastics in different food samples such as, mineral water, milk, orange juice, chipsy, roomy cheese, halawa, tomato sauce, cake, pickles, corn oil and sunflower oil at different storing periods (45 days of the short- period storing foods & 12 months for the long – period storing foods). The amount of "Malathion" as a harmful volatiles inside foods was determined quantitavely using Gas Chromatography. *Secondly*: Attempt to identify qualitatively the chemical compounds such as plasticizers which migrate from packaging materials into foods using the molecular – gas chromatography – mass spectrometry, and studying the effect of these chemicals on the physical & chemical properties of these foods. *Thirdly*: Using Agasco (F.T) Infra-red and U.V. spectra to study the interaction occurred between packaging materials H.D.P.E and packed mineral water and sunflower oil after 8 months of storage at 25˚C. In this study, results confirmed that such migration should be considered in the determination of shelf life of the food product which packaged in plastic containers in Egyptian Standard.

Key words: Pollution, pollutants, Malathion, Plasticizers, Plastic packages, foods.

Introduction

Acrylonitrile, butabiene, styrene, Celluloseacetate, cellulose propionate, polycaprolactone, polyester, polyproplyne, polystyrene, and polyurethane are different materials used to manufacturing more types of plastic packaging (Yang *et al.,* 2013)

Plastic bottles have several characters concerning with advantage and disadvantage when used for food packaging as reported by Gassel *et al.,* (2013) & Erythropel *et al.,* (2014)

Phthalates are a group of diesters of phthalic acid (dialkyl or alkyl aryl esters of 1,2 benzenedicarboxylic acid) and they are primarily used as plasticizers {substances added to plastics to increase their flexibility} . As the phthalates are not chemically bonded to the polymer, these compounds can migrate from the plastic material to the environment and, consequently, they are found in food and water in the human body (Santos and dos, 2011)

Bluthgen (2000) revealed that the main groups of migratory agents into food are monomers, starting agents and additives from plastic materials which come into contact with food stuffs. One of the most important groups of compounds capable of migrating are plasticizers in polymers and synthetic rubbers which come into contact with the food during its packaging. From the wide range of plasticizers in polymers and rubbers, the phthalic acid esters of medium chain alcohols, especially the diethyl hexyl phthalate (DEHP) are most widely used . This substance is an excellent model for environmental pollution and fugacity from contact materials .

He confirmed that the migration of these molecules elements may be assisted by the acidic pH value of the food stuffs, especially high fat foods like milk, meat, fish, sausage, salatoil, and margarine.

Bach *et al.,* (2013) reported that the second type of migration was the pesticide, especially, the volatile compounds such as Malathion . Due to the use of pesticides has increased since the end of the second world war . These compounds can be classified on the basis of their chemical structure or the action which perform pesticides are mainly used in agricultures (68%), in commercial and industrial activities (17%), domestically (8%) and (7%) in governmental application . Therefore, high levels of these chemicals may be causing contamination to the environment, and residues of pesticide could penetrate to the unpackaging foods as well as packaged foods through the packaging materials (Larroque *et al.,* 1990) .

Erythropel *et al.,* (2014) studied the plasticizer, DEHP, and its breakdown products which identified as ubiquitous environmental contaminants, and daily human exposure is estimated to be in the microgram per kilogram level, and they examined the exposure through bottled water, and packaged food and showed higher levels of DEHP in the later compared to bottled water.

Batra, (2011) studied the pollution migration of butylated hydroxytoluene by the test line suggested by EU No 10\2011, Gc. MS and GC analysis from plastic packaging materials to the product under different

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conditions, the migration amount of 2,6 – butylated hydroxytoluene under 100 degrees C and 30 minutes heating reached 76.29 mg\kg .

Technological progress in synthetic packaging materials allows an important and selective obtained of non condensable gas and water vapour changes such as that obtained with barrier polymers . Nevertheless, most plastic packages have a strong affinity toword hydrophobic volatile compounds, such as aroma compounds (Bruck *et al.,* 2014) .

The volatile compounds and polymer film characteristics must be taken into account to explain the transferring process. However, other structural factors exist that can affect sorption and diffusion. That is, the type of polymerization, the polymer tridimensional structure, the material cohesion maintained by weak energy bonds of the polymer and transition temperature. (Gavara *et al.,* 1996 ; Rubiol, 2011) .

Tehrany and Desobry (2004) investigated the factors which affect the migration of volatiles, additives, monomers and oligomers from packaging materials into food or adsorption of volatile compounds from the food by the polymer. They found that, these factors include the chemical structure of the migrant, molecular size and structure, fat content, and degrees of crystallinity .

Batra (2011) studied also the effect of transmission of light from colored plastics bottles (black, red, blue, colored and transparent polyethylene bottles) to the food stuff (palm oil), he found that the oil packed in black color container had low peroxide value, low acid value and low iodine value followed by red, blue and transparent plastic containers at the end time of storage, due to the effect of transmission of light on the palm oil stored for 6 months.

Cauwenberghe and Janssen (2014) studied the migration of antioxidant n-octadecyl 3 - (5,3 ditetrabutyl – 4- hydroxyphenyl) – propionate from various plastics under standard tested conditions for 10 days at 40 ˚C . The results indicated that the migration decreases in the order LDPE > HDPE > PP > PS and , the migration decreases with increasing density of the polymer .

The control of pollutants from plastics has become necessary which the movement of these different pollution volatiles to the food stuff caused a harmful effects. Its toxicological/pharmacological properties must be concern and measured such as constituent of printing colors on the outside of food packaging materials and plasticizer used to render the materials of plastics which can migrate within the material and leach out of it over time and became toxic material . Bach *et al.,* (2013) defined the factors which influence potential migration of plasticizers or other components ; structure of polymeric material, method of plasticizer addition to the polymer and compatibility of plasticizer polymer . The quantities of substances that migrate from plastics into the contact foods depend largely on the type of food, the temperature during contact, pH value of food stuff and the contact time .

The aim of this investigation presents more detailed information's concerning the pollution of the food packaging materials and the surrounding volatile compounds from the polymers to these foods due to long period of storage.

Materials and Methods

Materials:

All samples were purchased from different supermarkets (10 samples from each product), transported to the laboratory and immediately analyzed. Several product – related date were recorded, including: manufacturer, batch number, expirty date and type of packaging, materials. The samples consisted of : 1) Mineral water 2) Sunflower oil 3) Full cream milk 4) orange juice 5) Tomato Sauce 6) Cake 7) Halawa 8) chipsy 9) pickles 10) corn oil 11) Roomy cheese . The standard of Malathion was purchased from Chem. Service, Ins. (West Chester, PA)

Table 1: Type of polymer package, Thickness and company name of each product

No	Product	Stored/ month	Company name	Type of polymer package	Thickness
1	Mineral water	12	Dasani	H.D.PE	18 Micron
2	Sunflower oil	12	Cairo oil and soap	H.D.PE	12 Micron
3	Full cream milk	12	Domty	Tetrapake/PE	12 Micron
4	Orange juice	6	Enjoy	Tetrapake/PE	40 Micron
5	Tomato sauce	6	Fine Foods	PP/PE	4 Micron
6	Cake	1	Monginis	PP/PE	5 Micron
7	Halawa	12	El-Rashidi	PP/Al/PE	5 Micron
8	Chipsy	6	Chipsy	PP/PE	18 Micron
9	Pickles	3	El-salam	H.D.PE	18 Micron
10	Corn oil	12	Cairo oil and soap	PA/PE	40 Micron
11	Roomy cheese	6	Betti	H.D.PE	9 Micron

Methods:

Determination of pesticide residues:

Malathion residues were extracted from different samples according to the methods of A.O.A.C (2000) and the Pesticides Analytical Manual (1991). The extracted samples cleaned up using flourisil column (60/100 mesh) and eluted by Hewlett Packard Gas chromatography models 5890 equipped with electron capture detector (ECD), flam ionization detector (FID), and integrator 3392, fitted with H.P - capillary column (Cross linked methyl silicon Gum), 30m × 0.25mm × 0.25um film thickness, The column oven temperature was 220 ˚C and held for 30 min. injection and detector temperatures were 220 ˚C and 300 ˚C, respectively.

Evaluation of the spectrum of mineral water and sun flower oil samples and packaging materials:

Agasco FT/IR 460 plus Fourier Transform Infrared (IR) Spectrometer was used to evaluate the spectrum of each packaging materials and water and oil samples at zero time and at the end of storage. The carbonyl groups index was used to determine the appeared and disappeared groups. Ultra violet and visible absorption instrumentation Unicam UV/Vis. spectrometer U.V – visible (UV2 – 300) serial No.9423 (UV2 – 300) ultra violet. Spectra was used to evaluate the spectrum of mineral water and sunflower oil samples and each packaging materials at the end of storage. Specimens of 13 mm diameter were taken from tested packaging materials and then fixed in face of IR ray source . The obtained spectrum was compared with carbonyl groups index to define the appeared and disappeared groups in spectrum as reported by furniss (1978).

Gas chromatography/ Mass spectrometry system (Gc/Ms) :

The GC/MS system was used te determine the fundamental nature of the interactions. Scientists, combined the qualitative nature of gas chromatography – mass spectrometry with the sensitivity of the human nose to understand these interactions.

A qualitative analysis for identification of organic compounds using Hp 5890 series II Gas chromatography Hp Mass selective detector 5972 series . Column Hp – 101 (Methyl/Silicon Fluid) 25m × 0.2 mm × 0.2 um film thickness. Initial temperature of oven program was 100 ˚C (set point) and the final was 250 ˚C but the initial time was 1min. and the final was 5min.

Viscosity:

The viscosity of oil was measured by using the Brouk Field viscometer Model Dv-1t, according the method of Sandra *et al.,* (1983).

pH value :

The pH value was measured at 25 ˚C using a pH meter as described by A.O.A.C (2000).

Refractive index:

The refractive index of oil was determined at 25 ˚C according to the A.O.A.C method (2000) using a refractometer (NYRL – 3 Poland).

Thickness :

Thickness of package materials was measured by using caliper micrometer, thickness of a stocking several layers of the film was measured and film thickness was reported in microns according to the method described by William (1992).

Oil stability:

Rancimat (Metrohm Ltd . CH 9100 Herisau, Switzerland) was used for the determination of oxidative and thermal stabilities of oil. The 679 rancimat comprises control unit and wet section containing 6 reaction vessels. In the west section, the oil sample (5g) is exposed to a stream of atmospheric oxygen (201/hr) at 100 ± 0.2˚C. The volatile filled with distilled water (60 ml) and continuously detected with a conductivity cell according the method of Warner & Mounts (1984). Free fatty acids of the oil was determined according to the A.O.A.C method (2000) .

Results and Discussion

The commercial polymeric materials (plastics) contain various additives mainly plasticizers, anti – oxidants, Lubricants, stabilizers, and colorants etc. These additives are usually had low molecular weight compounds possessing high mobility. They often migrate from the polymer matrix by diffusion into a liquid or solid contacting medium and can have an unfavorable effect on the food (Erythropel *et al.,* 2014)

The results recorded in Table (2) showed the physical and chemical properties of sunflower oil packed in two different polymeric materials for 12 months of storage at ambient temperature (25 ˚C). We can noticed that free fatty acid, viscosity and oxidative stability were increased in smaller thickness bottles H.D.PE (12 microns) than that of larger thickness, PP/PE (18 microns), while the refractive index (RI) decreased larger in the smaller thickness (H.D.PE) The interaction of the packaging polymer of bottles and the oil Clearly high, where the PE film reacted with the oil due to long period of storage, changing its thickness and the degree of crystallinity as shown in Table (3). This is in accordance with the report of Matsui *et al.,* (1992)

The results in Table (4) confirm the preceding ones in Tables (2,3) which the release of non – intentionally added substances from PE bottles such as aldehydes, volatile organic compounds and other components of plastics, into water and tomato sauce, for example, bisphenol at high temperature degree and long period of storage must be decrease the pH of the food stuff medium as reported by Yang *et al.,* (2013), where an increasing ratio of crystalline regions to amorphous one in the polymer decreased the solubility of the aroma compounds in the polymer .

Table 2: Physical and chemical Properties of sunflower oil after storing 12 months at 25 ˚C.

Type of package	Thickness (micron)	Free fatty acid %		Refractive index (RI)		Viscosity (poise : g/cm.s)		Oxidative stability	
		Zero Time	12 Months	Zero Time	12 Months	Zero Time	12 Months	Zero Time	12 Months
PP/PE	18	0.12	10.22	1.475	1.471	44.0	640	3.78 h	247 h
H.D.PE	12	0.15	14.71	1.476	1.468	44.0	5608	4.12 h	250 h

Table 3: Thickness of H.D.PE and PP/PE packed with mineral water and sunflower oil for different storage periods at 25 ˚C

Type of package	Thickness (micron)					
	Mineral water			Sunflower oil		
	Zero time	5 months	10 months	Zero time	5 months	10 months
PP/PE	16.0	15.5	15.0	16.0	25.0	38.0
H.D.PE	9.0	8.0	7.0	9.0	12.0	18.0

Table 4: pH values of mineral water and tomato sauce in different storage periods at 25 ˚C

Packaging materials	Thickness (micron)	Storage periods (months)							
		0	4	8	12	0	4	8	12
		Mineral water				Tomato sauce			
PP/PE	18	6.63	6.21	5.93	5.72	4.24	4.17	4.11	3.98
H.D.PE	12	6.63	6.13	5.78	5.46	4.24	4.05	3.96	3.87
Strech PE	9	6.63	6.02	5.60	5.27	4.24	4.00	3.87	3.72

At the same time, there are many extrinsic factors that influence photo oxidation such as : the spectrum, intensity, degree of ligh transmittance and the oxygen permeability of the packaging materials, these factors responsible for the photosensitivity of the food stuff and change its acidity (Bach *et al.,* 2013)

Results in Table (5) illustrated the quantity (ug/g) of penetrated "Malathion" into different packaged foods by Gas chromatography analysis during validity periods of different foods.

Table 5: Effect of storage periods on penetration of volatile compounds "Malathion" (p.p.m) into different packaged foods .

Type of food	Type of packaging material	Storage periods					
		Short validity packaged foods (ug/g) (p.p.m)			Long validity packaged foods ug/g. (p.p.m)		
		3 days	10 days	1 month	3 months	6 months	9 months
Milk	Tetra pake/PE	n.d	0.001	0.004	1.312	2.813	---
Orange Juice	Tetra pake/PE	n.d	0.270	0.435	1.724	2.300	3.507
Pickles	H.D.PE	0.030	0.217	0.293	2.175	2.841	3.412
Tomato Sauce	PP/PE	0.070	0.532	1.296	3.125	3.751	4.315
Cake	PP/PE	0.021	0.370	0.912	---	---	---
Chipsy	PP/PE	n.d	n.d	0.001	1.140	1.790	---
Halawa	PP/Al/PE	n.d	0.71	1.600	2.400	7.900	11.400
Roomy Cheese	H.D.PE	n.d	n.d	0.017	3.290	6.730	---
Mineral water	H.D.PE	0.84	1.21	1.506	2.110	2.470	3.410
Corn oil	PA/PE	0.97	1.27	1.581	3.240	5.420	9.510

n.d : Not detected

We can noticed the amount of substances migrating from plastic into food stuffs such as corn oil and Halawa . In most cases, this is due to the higher solubility of the migrating organic compounds in fat compared to water and not due to increase in the diffusion coefficient due to interactions between the fat and plastic as is often assumed, as mentioned by Bruck, *et al.,* (2014).

The interaction occurred between packaging materials H.D.PE and packed mineral water and sunflower oil after 8 months of storage at 25 ˚C are illustrated by Agasco (F.T.I.R) and U.V spectra (Table 6).

Table 6: Interaction occurred between packaging materials H.D.PEand packed mineral water and sunflower oil after 8 months of storage at 25 ˚C

Treatments	Storage period (months)	Total peaks	Waves number of new appeared peaks (cm^{-1})	Waves number of disappeared peaks (cm^{-1})
Control *	0	---	---	---
A_1	8	38	723 – 913 – 1101 – 1162 – 1273 – 1367 – 1744 – 2339 – 2660 – 3431	719 – 729 – 2852 – 2924
Control **	0	49	---	---
A_2	8	32	723 – 2031 – 2683 – 3455	846 – 873 – 913 – 967 – 984 – 1034 – 1377 – 1462 – 2683 – 2733 – 3002

*Control * : H.D.PE film before packaging with mineral water or sunflower oil. A_1: H.P.PE film after packaging for 8 months with mineral water. Control **: Sunflower oil before the packaging. A_2: Sunflower oil after the packaging for 8 months.*

The IR spectra for the H.D.PE film packed with mineral water after 8 months of storage in comparison to control before packaging showed that 10 new peaks compared to control were appeared as well as 4 peaks were vanished. While the IR spectra for the sunflower oil after 8 months of storage revealed that 5 new peaks were appeared compared to the sunflower oil before the packaging (control) as well as 11 peaks were vanished.

From the obtained results it could be noticed that the highest number of peaks which appeared was observed in case of mineral water but the highest number of peaks which disappeared was observed in case of sunflower oil. This proved that the contact between fatty food and plastic packaging may cause reciprocal transfer between the material and the surrounding medium, but the migration into water and liquid foods was unresciprocally or weak. This is in accordance with the results of Audic *et al.,* (2000), which they found also, that the migration into water and liquid foods will be higher than that into more solid food stuff, with all plastic types, and increased by contact time and temperature.

The possible migration of organic compounds from two different packaging materials into mineral water and their retention times were identified by G.C mass as shown in Tables (7,8). We can identify for 14 compounds at 12 retention times in the case of stretch PE, packging material and for about 24 compounds at 25 retention times in the case of H.D.PE packaging materials. Therefore, high levels of these chemicals may be causing a harmful effects to the customers. Scarfato *et al.,* (2000) found that these chemicals such as benzephenone, di-butyl phthalate and di-ethylhexyl adipate were incorporated by injection molding or into sheet film during processing the polymer as plasticizers.

Table 7: Qualitative migrated compounds of stretch PE. Migrated into mineral water and their retention time by G.C mass .

Retention Time	Area	Area %	Ratio %	Name of possible migrate organic compounds
4.588	35070	1.874	3.360	Silanamine
12.037	15532	0.830	1.488	3- Isopropoxy – 1,5 - hexamethy
14.154	172258	9.205	16.505	Hexadecanoic acid
15.241	80123	4.282	7.677	9,12 – Octadienoic acid,
15.298	91513	4.890	8.769	9- Octadecenoic acid, Methyl ester
15.481	39048	2.087	3.742	9- Octadecenoic acid, Methyl ester
17.209	18493	0.988	1.772	Trivinyl – S- Trazine 2
18.479	47403	2.533	4.542	Jasmolin I
18.662	1043641	55.771	100.000	Benzenamine
18.765	94049	15.714	28.175	1,2 – benzene dicarboxylic acid
19.910	18256	0.976	1.749	2- Methoxy -1,3 – bis (trimethyl silyl)
19.967	15904	0.850	1.524	Benzene

Table 8: Qualitative migrated compounds of H.D.PE migrated into mineral water and their retention time by G.C mass.

Retention time	Area	Area%	Ratio%	Name of possible organic compounds
3.603	1163671	0.850	3.916	Heptane,
10.714	94250	0.688	3.172	1 -phenyl-1 ,5-hexanedione
13.648	1040427	0.760	3.501	N-benzenesulfonylazetidin 3- One benzoic acid,
14.364	746147	0.545	2.511	1- Octene,
14.451	193187	0.141	0.650	2Dodence,
15.704	1361601	0.994	4.582	Acetic acid,
15.753	1072811	0.784	3.610	1,1 Dimethyl-2- (1,1-dimethylethyl) 2-Dodecene
15.817	2034927	1.486	6.848	2- Octene,
17.764	6349664	4.638	21.369	Pentadecanoic acid,
17.993	1549114	1.131	5.213	2- Octene,
19.343	583446	0.426	1.962	Cyclopropane,
20309	9832429	7.181	33.090	9,1 2- Octadecadienoic acid,
20.444	9155747	6.687	30.8 12	9- Octadecanoic acid,
20.536	1468221	1.072	4.941	Pentane,

Table 8: *Cont.*

20.886	3644996	2.662	12.267	Cyclopentañeundecanoic acid
22.270	10025964	7.323	33.741	N-benzenesulfonylazetidin 3-one_Benzaldehyde,
22.498	2647920	1.934	8.911	3-Undecene
22.943	1842914	1.346	6.202	2,4-Diamino-6hydroxy-5-thiocyanat
23 .066	9535349	6.964	32.090	3-pyridinecarboxylic acid
24.471	2749654	20.083	92.537	3-carbomethoxypiperidine
24.918	2409103	1.759	8.107	4-Chlorobuten -3une
27.121	1508000	1.101	5.075	N-benzenesulfonylazetiddin 3-ONE
27.156	1397665	1.02 1	4.704	3-pyridinecarboxylic acid
27.521	29714634	2 1.702	100.000	1,2-Benzenedicarboxylic acid
27.870	9202629	6.721	30.970	

Results given in Table (9) showed a qualitative analysis of sunflower oil packed 12 months at 25 ˚C in H.D.EP by U.V. spectra. The compounds; nitrate, nitrite and Izo were identified in H.D.PE packaged in H.D.PE packed in sunflower oil after 12 months of storage while thiocyanate, Nitroze and izo were identified in sunflower oil sample stored in H.D.PE for 12 months. Stoffers *et al.,* (2004) studied specific migration (SM) of stabilizers such as Irgafos 168 from HD.PE into sunflower oil and found that, specific migration in H.D.PE 0.150 mg/dm^2 during storage at 25 ˚C for 10 months, and migration was found to be higher into pure fat and margarine than into mayonnaise. Furthermore, the transfer of compounds from plastics was found to decrease in the order L.D.PE > H.D.PE > PP > PS > ABS (Acrylonitrile butadiene styrene).

Table 9: Qualitative analysis of sunflower oil packed in H.D.PE of U.V. spectra after 12 months of storage at 25 ˚C

Sample	Qualitative Compounds			
	Min.	Max.	Identified compounds	Symbol
H.D.PE film (control)	---	---	---	---
H.D.PE packaged in mineral water (12 months)	---	---	---	---
H.D.PE packed in sunflower oil (storing 12 months)	12 12 10 10 10 3-25	257 263 293 305 347 371	Nitrate Nitrate IZo Nitrite Nitrite Izo	$-ONO_2$ $-ONO_2$ -N = N- -ONO -ONO -N=N-
Sunflower oil (control) Sun flower oil sample stored in H.D.PE for 12 months	10 50 100 3-25	325 257 299 341	Nitrite Thiocyanate Nitroze Izo	-ONO -S-STN -N = O -N = N-

Conclusion

From the obtained results it concluded that migration of plasticizers and/or pesticide compounds from plastic bottles affected by properties of packaging materials and chemical structure of plasticizer compounds so, it could be suggested that such migration should be considered in the determination of shelf life of the product which packaged in plastic containers in Egyptian Standard. On the other hand, plastic packaging materials are not suitable for packaging fatty food, but if it necessary, must be minimized the storage period (short validity) and its temperature. It can be advised to package the edible oils in glass bottles to avoid migration of toxic compounds from the containers to the oils and to make it safe for human consumption.

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