

LOCAL STUDY ON PERFLUORINATED MATERIALS AND THEIR PRESENCE IN SOME FOODS IN BAGHDAD GOVERNORATE

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ABSTRACT

The objective of this study is to identify and quantify perfluorinated substances in different ready-to-eat foods found in the local markets of Baghdad Governorate. The emphasis is placed on these substances due to their extensive use in paper and cardboard packaging materials and the consequential health effects. The analysis utilized liquid chromatography coupled with mass spectrometric detection equipped with electrospray ionization. The values recorded for perfluorinated materials in samples of crispy chicken and french fries were (2.41 and 4.94) micrograms, respectively. As for samples of kebab, pizza, and cake, they were (5.14, 5.80 and 7.32) micrograms, respectively, and samples taken from stored ice cream (15-30–45) days recorded (0.23, 0.42 and 2.26) micrograms, respectively. It was found that the percentage of fat and the amount of perfluorinated substances in foods are directly related, and this is clearly evident from the fact that it was not detected in the French Bread samples, and may be due to the low percentage of fat which amounted to 0.86%, compared to the samples that showed positive results.

Key words: Chemical composition; Liquid chromatography followed by mass spectrometry.



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INTRODUCTION

Perfluorinated substances are a group of human-made fluorinated organic chemicals that do not occur naturally in the environment. They show at least one terminal carbon atom with fluorine atoms in place of all the hydrogen. They are either fluorinated aliphatic substances or fluorinated cyclic substances. Many substances are used. Perfluorinated materials as surfactants or in the production of fluorinated polymers. Fluorosurfactants constitute a diverse group characterized by a fluorinated carbon chain and a highly hydrophilic functional group or moiety, their molecular weight is less than 1000 daltons. Many industrially used fluorosurfactants are mixtures of compounds of different chain lengths and of undetermined purity (Zhu *et al.*, 2023). Perfluorinated materials have been used to enhance the oil and water resistance

properties of paper and cardboard (Teflon, 2021). some perfluorinated materials are used in the paper and cardboard used in packaging, and these applications particularly target fatty foods, especially those in which the food is placed while it is hot or stored for a long time, for example, fast food such as French fries, hamburgers, and popcorn bags, baking paper, baking cups, sandwich wrapping paper, butter, and dry food paper. These resistance properties are particularly important in the food packaging sector, as oils, grease, and water may be transferred from the food during baking, transportation, storage, or for use with fast food, such as when these materials are in contact with the food product (Susmann *et al.*, 2019). Given the increasing threat and highly variable presence of perfluorinated substances in food and drinking water, there is a need for

more detailed research on their concentration levels in the human diet. EFSA has set a permissible weekly consumption limit of 4.4 nanograms per kilogram of body weight per week (Commission Regulation (EU) ,2022). So, it should increase consumer awareness about food safety (Al soufi,2022), high-quality products, in-depth studies have been conducted on the packaging of foodstuffs (Al Mkhari *et al.*, 2025.), as it is possible for some of their chemical components to migrate when they come into contact with certain types of foods because there are no completely inert materials (Gupta *et al.*, 2024).The use of chemical compounds may lead to environmental risks (US Environmental Protection Agency , 2019), so it is preferable to use environmentally friendly ingredients when manufacturing (Grafia *et al.*, 2025). Most consumers do not distinguish between food-borne disease (Al khafaji, 2023), so their concern is focused on the quality of food and its good packaging (Al soufi, 2021). To maintain food quality (Alamri *et al.*, 2021), quality standards must be applied and limits must be set for materials entering the market (Husain and alkhafaji, 2022). Find Perfluorodecanoic acid 3.05 g in heptane mimetic and perfluorononanoic acid of 2.12 g ethanol were the results for the highest percentage in a study by Choi *et al.* (2018) set comprising 312 items such as pans, baking utensils, rice cookers, roaster, and roast sheets. The findings suggest of perfluorinated compounds is more pronounced in alcoholic or fatty foods. In Germany, samples of frozen French fries contained less than 1µg/kg of PFOA and PFOS in frozen French fries (Stahl *et al.*, 2011). The investigation revealed that the migration of perfluorinated compounds in freeze-dried milk was greater in whole milk as opposed to low-fat milk. Both varieties of milk underwent lyophilization (Elizalde *et al.*, 2018). However there have been only a few

trials, and there is little real data on the levels of perfluorinated substances in foods consumed. Due to their stability, potential for bioaccumulation, toxicity to living organisms, and ubiquitous presence in the environment, they represent environmental pollutants of concern that require more detailed regulatory action (Bil *et al.*, 2021). The current study was aimed to detect the presence of perfluorinated substances in ready-to-eat foods in the local markets of Baghdad Governorate due to their health risks to consumers due to the increased rates of eating outside the home and the increase in demand for fast food at home. Therefore, it was necessary to study their presence and identify the types of foods in which they are present.

MATERIALS AND METHODS

Sample collection:

Samples have been collected in 2022 the relevant ready-to-eat foods in fast food restaurants and markets in Baghdad Governorate. The food samples were analyzed according to their readiness to eat at 30 minutes for crispy chicken breast, french fries and French bread, as for the grilled meat kebab at 10 minutes, the meat pizza at 20 minutes and the cake at 45 minutes, while the hard ice cream samples as shown in (Table 3) were examined for 45 days, each of which was bagged separately to avoid transfer of fluorine between samples, may not necessarily reflect the general distribution of total F concentrations of foods in the market.

Chemical analysis of food:

1. Protein, moisture, fat, ash, and fiber in foods were estimated by the method mentioned in A.O.A.C. (2005).
2. Carbohydrates were calculated using the difference between components method, as mentioned by Pearson (1976).
3. The pH was estimated using a German pH meter.

Extraction of perfluorinated compounds from food:

A mixture of 2 grams of the sample was combined with a 10 ml solution of sodium hydroxide (20 mM methanol) for the purpose of alkaline digestion to extract food. Using the modified method of Llorca *et al* .(2012), amagnetic stirrer was used for 4 hours at 125 rpm to digest the samples at room temperature, and the filtrate was stored in the refrigerator until analysis in the LC/MS device.

Detection of perfluorinated compounds from food by LC-MS

To, identify perfluorescent substances method (Zabaleta *et al.*, 2017), liquid chromatography, and mass spectrometry with an electrospray ionization source from the Japanese company Shimadzu were used. A C18 column (2.1x 50 mm, 3.5 micrometers) was also used. A low-pressure gradient flow rate of 1 ml/min was set, and 20 microliters of the sample were put into the analytical column. The target analytes were then separated at 30 °C.

RESULTS AND DISCUSSION

Chemical composition of food and perfluorinated compounds in it

(Table 1) shows the results of the chemical analysis based on the wet weight of all food samples. The crispy chicken values for the percentage of carbohydrates, crude fiber, protein, fat, ash, and moisture were (9.31, 8.46, 21.87, 14.84, 1.80, and 43.72) % respectively. The pH value was recorded at 6.1, as for the French bread sample, The composition percentages for carbohydrates, crude fiber, protein, fat, ash, and moisture were as follows: (51.76, 3.52, 8.75, 0.86, 1.00,

34.11)%, with a corresponding pH value of 5. For fried potatoes, the composition was (13.41, 5.58, 10.93, 21.00, 1.59, and 47.49)% for carbohydrates, crude fiber, protein, fat, ash, and moisture, respectively, and the pH value was recorded at 5.5 for the sample under experiment, while for the pizza sample, it was found that the percentage of carbohydrates, crude fiber, protein, fat, ash, and moisture were (31.95, 8.15, 9.84, 22.42, 2.61, and 25.03) % respectively, and the pH value was 6.4. The results of the cake sample showed that the proportions of carbohydrates, crude fiber, protein, fat, ash, and moisture were (25.76, 9.77, 5.46, 19.22, 1.22, and 38.57) % respectively, and the pH value was 6.6.

It was also found that the values of the kebab sample percentage of carbohydrates, crude fiber, protein, fat, ash, and moisture were (0.35, 9.92, 22.96, 14.18, 0.98, and 51.61) % respectively, and the pH value was estimated at 5.9, in While the results of the ice cream sample based on weight, percentage of carbohydrates, crude fiber, protein, fat, ash, and moisture were (0.17, 0.19, 4.37, 13.95, 0.52, and 80.80) % respectively, and the pH reading was 6, it appears from the above results that the ice cream sample exhibited the highest moisture content at 80.80%, whereas the pizza sample had the lowest moisture value recorded at 25.03%. In terms of fat percentage, the pizza sample had the highest value at 22.42%, while the French bread sample displayed the lowest value at 0.86%. It is noted from the results that all values were for pH. The experimental samples fall within the range of moderately acidic foods.

Table 1. Chemical analysis of food based on wet weight.

sample	% * Carbohydrates	% * Fibers	*Protein %	* Fat %	*Ash %	%*Moisture	*pH
Crispy chicken	9.31	8.46	21.87	14.84	1.80	43.72	6.1
French bread	51.76	3.52	8.75	0.86	1.00	34.11	5
Fried Potatoes	13.41	5.58	10.93	21.00	1.59	47.49	5.5
pizza	31.95	8.15	9.84	22.42	2.61	25.03	6.4
Cake	25.76	9.77	5.46	19.22	1.22	38.57	6.6
meat kebab	0.35	9.92	22.96	14.18	0.98	51.61	5.9
Ice cream	0.17	0.19	4.37	13.95	0.52	80.80	6

*The numbers represent an average of three replicates.

(Table 2) displays the presence of the perfluorinated compound $C_{13}H_8F_3NO$, identified as 7-Hydroxy-2-trifluoromethyl phenothiazine, in the crispy chicken sample, with a recorded level of 2.41 micrograms. The chemical structure of the perfluorinated compound is illustrated in (Figure 1). Due to its composition, the compound exhibits increased solubility in edible oil 14.84% when exposed to moisture and heat during food preparation, attributed to its amine and hydroxyl content. Additionally, its solubility in water (humidity) is estimated at 43.72%, facilitated by its capability to form hydrogen bonds with oxygen and nitrogen atoms.

(Table 2) indicates the absence of perfluorinated compounds in the French bread sample, potentially influenced by the chemical composition, particularly its exceptionally low fat percentage of 0.86%. The predominant

perfluorinated compound observed in the studied samples is $C_{14}H_{16}F_5NO_3$, recognized as 2,5-dimethoxyamphetamine pentafluoropropionate, with a reading of 4.94 micrograms in the fried potato sample. Kebab samples registered a reading of 5.14 micrograms, pizza recorded 5.80 micrograms, and cake exhibited a reading of 7.32 micrograms. The elevated values in these foods are likely associated with their high fat content, ranging between 47.49% and 25.03%, as indicated in (Table 1).

(Figure 2) illustrates the structure of the perfluorinated compound $C_{14}H_{16}F_5NO_3$, featuring an amide chain linked to a phenyl ring and methoxy groups. This structural configuration facilitates the compound's solubility in oils, contributing to its migration into food during cooking.

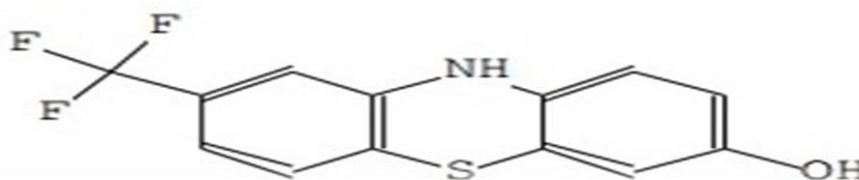


Figure 1. Perfluorinated compound $C_{13}H_8F_3NOS$

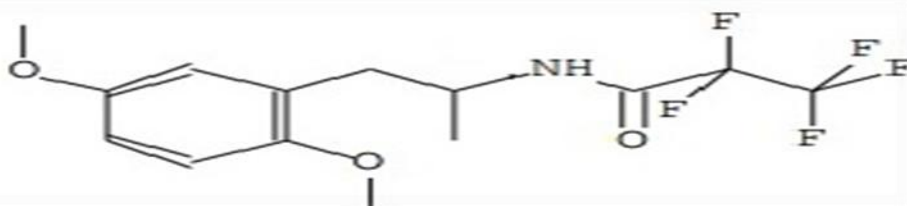


Figure 2. Perfluorinated compound $C_{14}H_{16}F_5NO_3$

Table 2. Perfluorinated compound in food

Sample	Perfluorinated compound $\mu\text{g/gm}$ [min]
Crispy chicken	$\text{C}_{13}\text{H}_8\text{F}_3\text{NO}_3$ 2.41 30
French bread	0 30
Fried Potatoes	$\text{C}_{14}\text{H}_{16}\text{F}_5\text{NO}_3$ 4.94 30
Meat Kebab	$\text{C}_{14}\text{H}_{16}\text{F}_5\text{NO}_3$ 5.14 10
pizza	$\text{C}_{14}\text{H}_{16}\text{F}_5\text{NO}_3$ 5.80 20
Cake	$\text{C}_{14}\text{H}_{16}\text{F}_5\text{NO}_3$ 7.32 45

(Table 3) presents the sole perfluorinated compound identified in the ice cream sample, with recorded readings at a storage temperature of -18°C over periods of (15, 30, 45) days, measuring (0.23, 0.42 and 2.26) micrograms, respectively. Illustrated in (Figure 3) is the structure of the perfluorinated compound identified as $\text{C}_{13}\text{H}_{11}\text{F}_5\text{O}_5$,

commonly known as Benzeneacetic acid, 3-methoxy-4-(2,2,3,3,3-pentafluoro-1-oxopropoxy). This compound features hydroxyl groups that enhance its solubility in water, attributed to its capacity to form hydrogen bonds with oxygen atoms. Furthermore, the presence of methoxy groups in the compound facilitates its solubility in oils.

Table 3. Perfluorinated compound in Ice cream

Sample	[Duration (day) Temperature -18°C]	Perfluorinated compound $\text{C}_{13}\text{H}_{11}\text{F}_5\text{O}_5$ $\mu\text{g/gm}$
Ice cream	0	0
	15	0.23
	30	0.42
	45	2.26

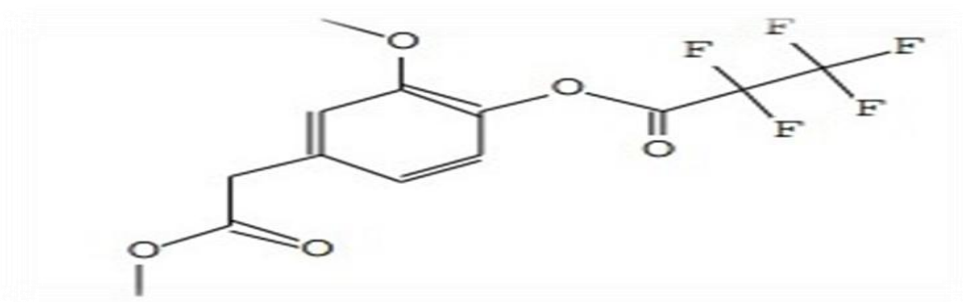


Figure 3. Perfluorinated compound $\text{C}_{13}\text{H}_{11}\text{F}_5\text{O}_5$

From the information presented, it is evident that all recorded values of compounds migrating into food exceed the limits set by the European Food Safety Authority, which established an acceptable weekly amount of ng/kg of body weight per week 4.4

(Commission Regulation (EU) ,2022).The presence of perfluorinated substances was influenced by several factors, notably the contact time of the food substance with paper and cardboard. It was noted that the quantity of the compound and the contact time were

directly proportional, as illustrated in the ice cream sample . All studied foods fall within the moderate acidity range (pH 5-6.9). The pH level inversely affects the quantity of perfluorinated compounds in food; lower pH values correspond to higher amounts of perfluorinated compounds. Acidic foods, therefore, facilitate the migration process more than basic foods, as confirmed by AbulFadl *et al.* (2019). The impact of humidity on the presence of perfluorinated compounds in the studied foods was also observed particularly evident in the low values of the ice cream sample, which had a high moisture percentage of 80.80%. This aligns with findings by Fengler *et al.* (2011), indicating that lower humidity leads to a greater amount of perfluorinated materials migrating to the food, adversely affecting the migration process. A direct relationship was identified between the percentage of fat and the quantity of perfluorinated substances in the food. This relationship was evident in the non-detection of perfluorinated compounds in samples of French bread , which contained a very low fat percentage of 0.86%, compared to positive samples. This finding is consistent with the mechanism proposed by Choi *et al.* (2018), suggesting that the lower the percentage of fat, the lower the amount of perfluorinated compounds.

CONCLUSION

Upon comprehensive consideration of various food factors, it was evident that their influences interplay and may have varying degrees of impact. Notably, the interaction between the moisture ratio and fat percentage exhibits a more substantial influence than the pH of the samples under investigation.

Furthermore, the observed increase in values of the perfluorinated compound $C_{13}H_{11}F_5O_5$ in ice cream samples with an extended storage period suggests potential migration from the storage container. This emphasizes the need

for further studies examining the presence of perfluorinated substances in packaging materials within local markets. Such investigations are crucial for understanding and mitigating potential risks associated with these substances in the food supply chain.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

DECLARATION OF FUND

The authors declare that they have not received a fund.

AUTHOR/S DECLARATION

- We confirm that all Figures and Tables in the manuscript are original to us. Additionally, any Figures and images that do not belong to us have been incorporated with the required permissions for re-publication, which are included with the manuscript.
- Author/s signature on Ethical Approval Statement.
- Ethical Clearance and Animal welfare
- Funds:

AUTHOR'S CONTRIBUTION STATEMENT

- Huda F. Al-Attar and A. K. Al-Darwash were responsible for designing the study. The all experiments were conducted by Huda F. Al-Attar. and Huda F. Al-Attar and A. K. Al-Darwash composed the manuscript.

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دراسة محلية عن المواد البيروفلورية وتواجدها في بعض الأغذية في محافظة بغداد

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المستخلص

هدف هذه الدراسة هو تحديد وقياس المواد الكيميائية الفلورية في مختلف أنواع الطعام الجاهز المتوفرة في أسواق محافظة بغداد. يتم التركيز على هذه المواد نظرًا لاستخدامها الواسع في مواد التعبئة والتغليف من الورق والكرتون والتأثيرات الصحية الناتجة عنها. تم استخدام التحليل باستخدام كروماتوغرافيا السوائل بالتزامن مع الكشف الكتلي المزود بتقنية الرش الكهربائي، سجلت المواد البيروفلورية قيمًا في عينات الدجاج المقرمش والبطاطا المقلية (2.41 و 4.94) ميكروغرام على التوالي، أما عينات الكباب والبيتزا والكيك (5.14، 5.80 و 7.32) ميكروغرام على التوالي، و العينات المأخوذة من الأيس كريم المخزون (15-30-45) يوماً فقد سجلت (0.23، 0.42 و 2.26) ميكروغرام على التوالي. وجد أن نسبة الدهون وكمية المواد البيروفلورية في الأغذية ترتبطان بعلاقة طردية، وهذا الأمر يظهر بشكل واضح من عدم الكشف عنها في عينات الصمون الفرنسي ويمكن ان يكون بسبب انخفاض نسبة الدهون والتي بلغت 0.86% بالمقارنة مع العينات التي أظهرت نتائج إيجابية.

الكلمات المفتاحية: التركيب الكيميائي؛ الكروماتوغرافيا السائل متبوعًا بالكشف الطيفي الكتلي.