Article

OCCURRENCE OF DIETHYL PHTHALATE (DEP) IN FOOD USING DIFFERENT PACKAGING: DETECTION AND TRACEABILITY

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The objective of this work was the detection and traceability of the occurrence of diethyl phthalate (DEP) migrating from flexible packaging to food. Brazil is one of the largest producers of flexible plastics widely used in food. The evaluation of migrations of contaminants for food, mainly fatty foods, elucidates the possible transmission of diseases through the daily consumption of migrant contaminants from packaging. Throughout an accurate and highly specific methodology, with no use of simulants or organic solvents during sample preparation, the proposal of this study obtained a direct DEP ranking through migration traceability via headspace: HS-GC-MS. Of the 9 samples of flexible packaging and packaged foods analyzed, DEP was identified in 100%, thus confirming its occurrence in foods. Despite the storage conditions: temperature, shelf life, fat content of the products and the manufacturer's assessment of use. It is urgent to review official standards to promote intense restrictions on the occurrence of DEP in food and encourage the benefits of reflecting daily consumption habits.

Keywords: flexible plastic packaging; fatty foods; diethyl phthalate; detection; public health.

INTRODUCTION

Flexible packaging is commonly used to package food products to maintain their freshness and prevent external contamination. It is widely used right across the market since the moldable thermo type materials used takes on the contours of the products, kept in refrigeration and ambient temperatures; such as with meat products in modified atmospheres, vacuum packed; and recently in products that go straight from the freezer to the oven, like plastic bags, as secondary packaging.

Anda-Flores *et al.*[1] affirm that polyvinyl chloride (PVC)-type thermoplastic is a rigid polymer that is softened by the addition of plasticizers in the manufacture of flexible films. Phthalates are widely applied as plasticizers in several products, the most used being orthophthalates, which are esters of 1, 2-benzene dicarboxylic acid (phthalic acid), and its *para* and *meta* isomers. The annual global consumption of plasticizers is 7.5 million tons, among which orthophthalates are the most consumed, with wide usage in food packaging, bottled water, toys, blood bags, supermarket and garbage bags.[2-4] In 2016, 2.14 million tons of plasticizers were used to produce flexible films.[5] Worldwide, it is observed that the increase in demand for phthalates follows an average annual rate of 1.3%.[6,7]

Some studies have shown that under some stress conditions (high/low temperatures, low pH, exposure to sunlight, long service life, etc.), they separate from the polymer structure and migrate from the walls to the container contents.[8-10] Phthalates have been found in a wide variety of foods and even at low concentrations can be toxic. Di(2-ethylhexyl) phthalate (DEHP) was classified as carcinogenic, mutagenic and as an endocrine-disrupting agent,[11,12] due to its effects on the liver, kidneys and reproductive system.[13,14] Among the toxic effects of diethyl phthalate (DEP) are the power of toxicity in the gastrointestinal and cardiovascular systems, and along with the reproductive system.[15-17]

Specifically, DEHP is permitted as a plasticizer in food contact materials but only for foods with high water content.[18] Due to restrictions on the use of DEHP in food contact materials, new plasticizers have been developed as commercially available substitutes. One of these is di(2-ethylhexyl) terephthalate (DEHT), the *para* isomer of DEHP.[19] A limit on the composition or SML of the DEHT has not been established by any legislation, neither for the DEP, and specific migration limits have not yet been established for any of these standards. In Brazil, the National Health Surveillance Agency (ANVISA), determined in RDC No. 326 of December 2019, that DEHP concentrations are limited to 1.5 mg kg^{-1} of the specific migration limit (SML), which are in contact with food or as a process support agent in concentrations of up to 0.1% in the final product.[20] This will force the industry to seek alternative plasticizers.[21]

Official methods are indirect, require a long experimental period, large workflow, and simulates storage and stress conditions. Food simulants, frequently used, presented methodological challenges.[22] Except that real foods are complex matrices, so there is debate about the efficiency of these methods. Guerreiro *et al.*[23] innovated with a direct analytical methodology, effective for detecting plasticizers, but using solvents in the preparation of meat samples by mass spectrometry. Both the modelling recommended by official methods still estimate migration, do not use real foods, and even methodological advances have required the preparation of samples with solvents. Highlighted, it is noted which contaminants were detected, they are indicative of interest in relation to the manufacture of polymers, and strong evidence that they come from plastic packaging.

In this context, the objective of this study was to detect and trace the presence of DEP in food packaging and in their respective foods. A survey of the occurrence of DEP was carried out on different types of flexible packaging commercially available in Brazil, used in various food products and stored at different temperatures (refrigeration, room temperature, from frozen directly to the oven and after thermal processing).

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EXPERIMENTAL

Samples of packaging and food

A total of nine foods packaged in flexible plastic packaging (considered moldable, vacuum packed, PVC film, plastic bags) which were purchased in the local market in the city of Goiânia, Brazil, were analyzed. The samples were kept under refrigeration (sliced cooked ham; sliced mozzarella cheese; sliced prato cheese; ground palette cut meat; matured beef hump cut meat; yogurt); were kept under room temperature (calabresa sausage; UHT milk – "soft belly" type); were kept at frozen temperature and roasted samples inside the packaging (seasoned chicken – "thigh"), as shown in Table 1. The packaging samples in use were cut with sterilized scissors, occupied 25% of a 20 mL vial and stored under refrigeration (below 10 ºC) until the time of analysis. The food samples of these packages were minced with the aid of a sterilized knife and stainless steel cutting board, and in solid form weighed (≈ 0.1 g). They were then removed from the packages with the aid of pipettes, and in liquid form, occupied 25% of the 20 mL vial (≈ 0.1 mL) and stored under refrigeration.

Table 1 presents some of the categories of plastic packaging classified according to ABIPLAST.[24] The categories are as follows:

1. Polyethylene terephthalate (PET) (number 1), used in refrigerant bottles. They are recyclable and have a high recycling rate, are thermoplastic, which is a resistant polymer in relation to physical impact and the substances reserved by it and has a low odor/gas absorption.
2. High density polyethylene (HDPE) (number 2), is highly recyclable and used in the composition of supermarket bags, detergent and shampoo bottles, automotive oil and others. It is non-toxic and has excellent physical-chemical resistance.
3. Polyvinyl chloride (PVC) (number 3), which is a plastic not entirely derived from petroleum but is part of its vinyl monomer composition. It contains chlorine, applied in water and sewage pipes, water bottles, mayonnaise pots, juice bottles, hoses and other items, but is not recycled much due to the high cost when compared to new PVC.
4. Low density polyethylene (LDPE) (number 4), the first plastic of the polyethylene type, is a polymer with the simplest molecular arrangement that exists, has a low cost, withstands temperatures from 80-95 °C and is non-toxic, waterproof, flexible and normally used for the creation of packaging, in the composition of computer parts, toys, bottles and garbage bags. It is one of the coverings used for the edges of milk boxes.
5. Polypropylene (PP) (number 5), is non-toxic, transparent and thermoplastic, which allows it to be molded when it is at high temperatures. It is applied to facilitate products that need to be visible to consumers, in more flexible plastic packaging, such as

Table 1. Food packaging samples (EE) and packaged food samples (EA)

Sample packaging	Sample food	Description of packaging in direct contact with food	Type	Description of food	Manufacturer's recommendation
EE1	EA1	Moldable term	7 OTHER	Sliced cooked ham	Keep refrigerated at 1 to 10 ºC and after opening consume within 5 days
EE2	EA2	Moldable term	7 OTHER	Sliced mozzarella cheese	Keep refrigerated at 1 to 10 ºC and after opening consume within 5 days
EE3	EA3	PVC flexible film	Not informed	Sliced prato cheese	No information
EE4	EA4	Moldable term	7 OTHER	Cooked and smoked pepperoni sausage	Keep in a dry and cool place +25 ºC, after opening keep in the refrigerator for up to 5 days. Consume only after cooking, frying or roasting completely
EE5	EA5	PVC flexible film	Not informed	Minced shoulder meat	Keep refrigerated at 1 to 10 ºC and after opening consume within 5 days
EE6	EA6	Vacuum	Not informed	Matured hump meat	Keep refrigerated at 1 to 10 ºC until expiration date. Duration of up 73 days from the date of manufacture
EE7	EA7	Sterilized plastic bag	4 LDPE	UHT milk	No information
EE8	EA8	Sterilized plastic bag	4 LDPE	Red fruit yogurt	Keep refrigerated at 1 to 10 ºC and after opening consume within 2 days
EE9	EA9	PVC flexible film	Not informed	Frozen chicken thighs with 10% brine (water, salt, garlic, onion herbs, and additives)	Take it from the freezer to the oven, remove the outer packaging, keep the product inside the internal transparent bag suitable for baking and place in a preheated oven at 190 to 210 ºC for 10 min, bake at a medium-high temperature of a maximum of 210 ºC for 1 h and 10 min. The special bag replaces the aluminum foil. During cooking, partially open the package to release steam, leaving the product more succulent and golden. Return the product to the oven to brown for another 10 min. Or until you reach the desired color

with ice cream and kitchen pots, since they can be placed in the microwave without suffering scratches and also cups, toys, chairs and car parts. It can be recycled.

6. Polystyrene (PS) (number 6), is a polymer that can be found in the form of solid plastic or as foam (styrofoam), is recyclable, versatile, cheap and has good impact resistance. Its main applications are found in such items as disposable cutlery, supermarket trays, margarine pots and plastic hangers.
7. Other (number 7), in this category we find all polymers that are not commonly identified on packaging. When the number seven is found, it means that the plastic in question cannot be characterized by any of the other six types. However, the packaging may come without numbering regarding the type of plastic from which it is made, either because of lack of information or because of fear of consumer reaction. It is fundamental to have transparency in this process so people can make conscious choices.

Reagents and materials

Ultrapure water was obtained using a Milli-Q system (Millipore); A DEP standard 99.5% (Sigma Aldrich); Nitric acid 66% (Synth) was used in the preparation of a 10% v/v solution; SH-Stabilwax-MS capillary column, 30 m × 0.25 mm × 0.25 µm (Shimadzu); using helium gas 5.0 analytical (White Martins) as carrier gas and nitrogen gas 5.0 analytical (White Martins) in headspace.

Detection and qualification of DEP in packaging and food in HS-GC-MS

The analyses were performed on a gas chromatograph from Shimadzu Nexis GC2030 coupled to the mass spectrometer (electrons impact) Shimadzu QP2020 NX, equipped with a SH-Stabilwax-MS column (30 m × 0.25 mm × 0.25 µm). The samples had been heated previously via headspace at 80 ºC for 30 min and a volume of 2 mL was injected into the chromatograph.

Splitless mode was used. The oven temperature was initially programmed at 40 °C min^{-1}, increasing to 160 °C at a rate of 5 °C min^{-1}, held for 5 min, increasing to 200 °C at a rate of 10 °C min^{-1}, held for 5 min, then further increased to 250 °C. The analyzes were performed from 32 to 38 min due to the DEP detection peak. Helium 5.0 was used as a carrier gas, with a pressure of 4.7 psi, a flow rate of 13.4 mL min^{-1} and linear velocity of 35 cm s^{-1}. The injector, interface and ion source temperature was maintained at 250 °C. The retention time of DEP peak was 32.8 min. The mass spectrometer operated in scan mode (range of 25 to 500 Da) and SIM (single ion monitoring) mode by *m/z* 149, 177 and 122, using 70 eV of electron ionization (EI).

The DEP compound detection was performed at the peak of *m/z* 149 (formed by the loss of an ethyl group from DEP, resulting in a phthalate ion), *m/z* 177 (formed by the loss of a CH_3CO acetyl group from DEP, resulting in a monoethyl phthalate ion) and *m/z* 121 (formed by the loss of an acetic acid from the phthalate ion; *m/z* 149, resulting in a phthalide ion, which show traceability of compounds detected by HS-GC-MS by the SIM method.

All glassware used in the experiments were previously washed with distilled water, immersed in 10% nitric acid for 24 h, rinsed in distilled water and heated in a muffle furnace at 550 ºC (Magnus Ovens) for 2 h.

RESULTS AND DISCUSSION

Figure 1 shows that the blank was performed using scan mode, confirming the absence of DEP. The identification was compared with the fragmentation database of the mass spectrometer provided by the NIST Library System (NISTT17s-2017). The DEP standard was performed in scan and SIM mode (Figure 2). All the samples, the total of nine samples of each, food packaging (EE) and packaged food (EA) were performed in SIM mode.

The method for detecting and traceability the occurrence of DEP from packaging to food *via* headspace, therefore, shows the presence of DEP in packaging and food packaged in different flexible packaging and at different storage and consumption temperatures.

Determination of phthalates in packaging and samples kept under refrigeration

The results obtained from the refrigerated food packaging samples: sliced cooked ham (EE1); sliced mozzarella cheese (EE2); sliced prato cheese (EE3); minced shoulder meat (EE5); matured hump meat (EE6); red fruit yogurt (EE8) and of the packaged foods samples: EA1, EA2, EA3, EA5, EA6 and EA8, respectively) are in Figure 3.

In packaging EE1, EE2, EE3, EE5 and EE6 the DEP detection occurred with similarities between the different packages. When compared, according to Table 1, to those with described materials (other number 7), those without information on the wrapping of the material used, the variation was approximate. These results reveal that

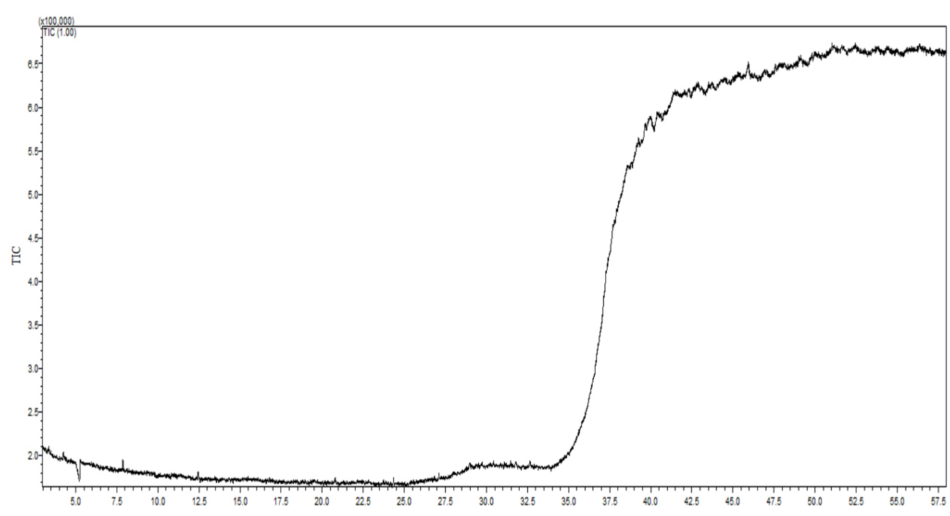

Figure 1. Chromatograms of blank chromatogram made SCAN mode

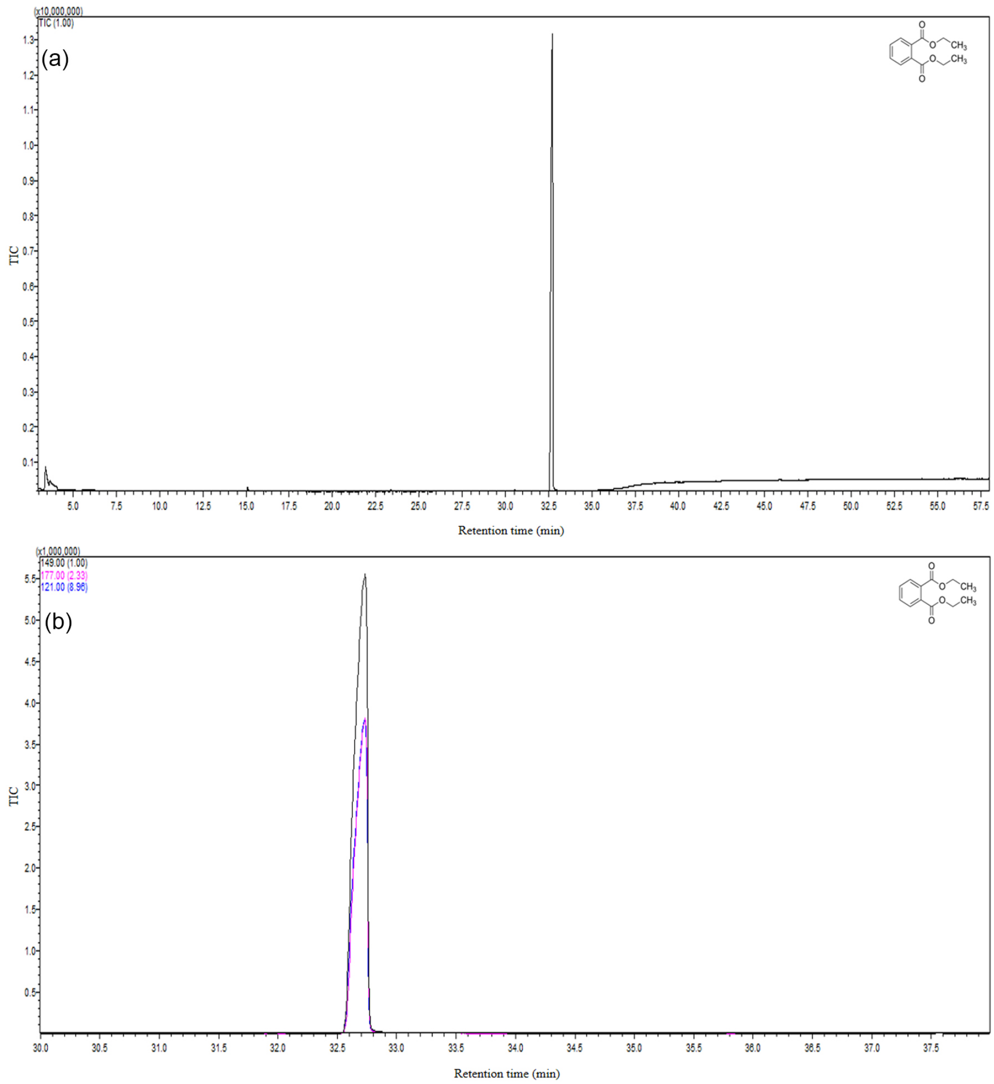

Figure 2. *(a) DEP standard chromatogram made in SCAN mode; (b) DEP standard chromatogram made in SIM mode (m/z 149, 177 and 122)*

it is still common practice for industries not to inform or generalize the plastic type of packaging applied to the food, so there is a difficulty in pointing out exactly which material such plastic packaging is made of. One of the most common types of plastics in this category is bi-oriented polypropylene (BOPP), which is present in snack packets, cookies, is recyclable when it is not in adhesive form and is not used on the edges of the wrappers.

In all packages and foods analyzed, DEP peaks were detected in the chromatograms of the samples, EA1 and EA2, the highest peaks. Refrigerated food packaging samples and packaged food samples were ham and mozzarella cheese, respectively. Smallest peaks at EA3 and EA5. The products analyzed were prato cheese, and each slice packed separately, with PVC plastic film surrounding the product, and ground palette cut meat and packed in a styrofoam tray and wrapped in PVC film, respectively. In the EA5 product, only the PVC film was analyzed, as it is the object of the study – flexible packaging. All refrigerated products have a considerable fat content. All are products for daily consumption due to their convenience according to the habits of the region and suffer a lot of handling. In the case of the EA2 and EA3 sample, each slice had direct contact with the packaging surface, because each slice was packed separately. In samples EA1 and EA5, there was a longer contact time with other surfaces during cutting and after processing meat products, which may indicate greater exposure to surfaces contaminated by phthalates before direct contact with PVC film packaging. Research done in Japan revealed that there was 41.0% of DEHP detected in PVC gloves used by food handlers in the packaging sector.[25] However, the polystyrene (PS) package portion (number 6), the styrofoam tray, is also a polymer used in more solid packages. According to Anda-Flores *et al.*,[1] the addition of plasticizers at low levels is unlikely to achieve the expected flexibility and adhesion in adherent films. As with PVC film, therefore, DEP is possibly present in these food samples as an impurity with other types of additives introduced during manufacturing. The detection of phthalates, DEHP or DEHT, as an impurity was discovered with a relatively high level of contamination from the use of low purity raw materials in the production of tri-octyl trimellitate plasticizer (TOTM) used in medical devices.[26,27] Trimellitic acid is used in the synthesis of TOTM and contains impurities, including *ortho*- and *para*-phthalic acid, which can be transformed into DEHP or DEHT, respectively.

Guerreiro *et al.*[23] determined the migration of phthalates to vacuum-packed refrigerated beef (rump cut) and identified the migration at up to 5 cm deep in the food, using high-resolution mass spectrometry (HRMS), without the use of food simulants. Other studies on the migration of phthalates from PVC packaging to mussels include,[28] and in ground meat.[29] In the case of conserved pickle-type plants packed in polyethylene terephthalate (PET), contamination by phthalates may also have occurred before packaging, and during cultivation, collection and transport of the samples.[30]

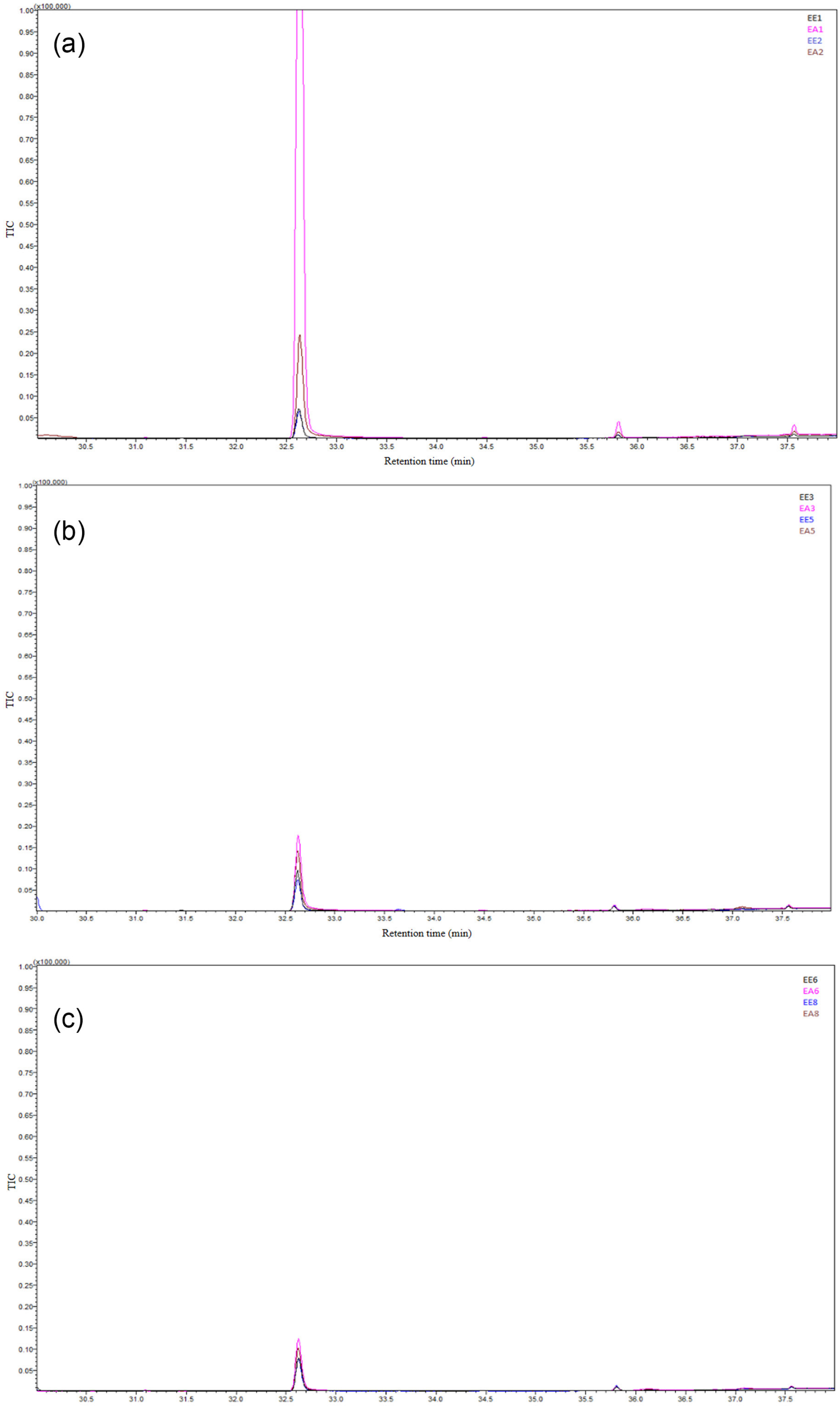

Figure 3. *Chromatograms of refrigerated food packaging samples and packaged foods samples performed in SIM mode (m/z 149, 177 and 122), respectively: (a) EE1 (ham packaging); EA1 (sliced cooked ham); EE2 (mozzarella cheese packaging); EA2 (sliced mozzarella cheese); (b) EE3 (prato cheese packaging); EA3 (sliced prato cheese); EE5 (minced shoulder meat packaging); EA5 (minced shoulder meat); (c) EE6 (matured hump meat packaging); EA6 (matured hump meat); EE8 (red fruit yogurt packaging); EA8 (red fruit yogurt)*

According to Table 1, the packaging is LDPE number 4, which stored dairy-derived food, with the pH of the food ranging from 3.6 to 6.8; stored under refrigeration. Some studies have highlighted the presence of migrating phthalates, including DEP, DEHP, and DEHT, in both low and high concentrations. Additionally, other migration investigations have detected phthalate traces. However, it's important to note that these findings primarily relate to films not in direct contact with food.[31-36]

Determination of phthalates in packaging and samples kept at room temperature

The results obtained from samples of food packaging stored at room temperature: cooked and smoked pepperoni sausage (EE4); UHT milk – "soft belly" type (EE7) and of the packaged foods samples: EA4 and EA7, respectively are in Figure 4.

Like the packaging EE1, EE2, EE3, EE5 and EE6, DEP was also detected in the EE4 packaging with similarity between the packaging, since there was little difference between the types of packages when compared. As can be seen in Table 1, those with the described materials (other number 7): the packaging information and the type of plastic material used by the manufacturer. However, it is still common for industries not to inform or generalize the type of plastic packaging applied to food, despite the requirement of health standards.

As with EE8, the EE7 packaging showed that DEP had lower peak amplitude in the chromatogram, and so there was less detection of DEP in these packaged foods. Both are LDPE number 4, which stored liquid, dairy and derived foods, specifically, EE7 and EE8 packaging of red fruit yogurt and UHT milk ("soft belly" type) (pH between 3.6-6.8). They were stored at different temperatures: under refrigeration and at room temperature, with different expiration dates, and analyzed within the expiration date. In Brazil, in other research, six PVC films for domestic use were analyzed and three samples contained DEHP in the range of 20.0 to 21.5%, the most toxic of the priority phthalates.[37]

Determination of phthalates in packaging and baked samples inside the packing

The results obtained from frozen and baked food sampled from within the packaging: frozen chicken thighs with 10% brine - water, salt, garlic, onion herbs, and additives (EE9) and of the packaged food sample: EA9, respectively are in Figure 5.

In samples EE9 and EA9, DEP peaks were detected with intensities also similar to the other packages analyzed. Despite exposure to high oven temperatures. The product analyzed was roasted chicken (thigh) from inside the packaging, as suggested by the manufacturer. Thus, it is noted that detection can be increased by heat treatment, as reported in the scientific literature.[1,23,38,39] Plastic packages that condition food at freezing temperature and then go into the oven along with food can facilitate the migration of its compounds to food.

According to research by Moreira *et al.*,[38] phthalates migrated after microwave heating to liquid foods (pH > 5) with detection limits for dibutyl phthalate (DBP) and benzyl butyl phthalate (BBP) of 0.08 and 0.31 µg L^{-1}, respectively. DBP were found at concentrations that ranged less than the limit of quantification (LOQ), which is the smallest amount of sample that can be accurately and reliably determined. Therefore, < LOQ at 7.5 µg L^{-1}. Increased migration was observed in containers used for a prolonged time, given the increase in heating time, and for fatty foods,[39] eight plasticizers were evaluated in spices and roasted chicken meat stored in plastic bags by means of GC-MS, with detection limits ranging from 0.01 to 0.18 µg kg^{-1}. It was observed that there was an increase in migration, assessed by the increase in the peak area of DEP. Di-isobutyl phthalate (DIBP) and DBP were found in the species sampled in higher concentrations and in seasoned chicken roast meat. Food simulants were applied, including water and sunflower oil, according to Brazilian and European Legislation, respectively. This explains the detection of fatty acid esters, observed in the chromatograms of the samples analyzed. The characteristics of the food can also influence contamination by these compounds, since phthalates are mainly lipophilic. In a study by Cavaliere *et al.*,[40] phthalate concentrations were determined with variations smaller than the limit of detection (LOD), defined as the smallest amount of analysis present in a sample that can be detected, but not necessarily quantified, under established experimental conditions. So, < LOD was 490 µg kg^{-1} for DBP, < LOD was 4700 µg kg^{-1} for DEHP, < LOD was 1750 µg kg^{-1} for BBP, and < LOD for DEP and dioctyl phthalate (DOP).

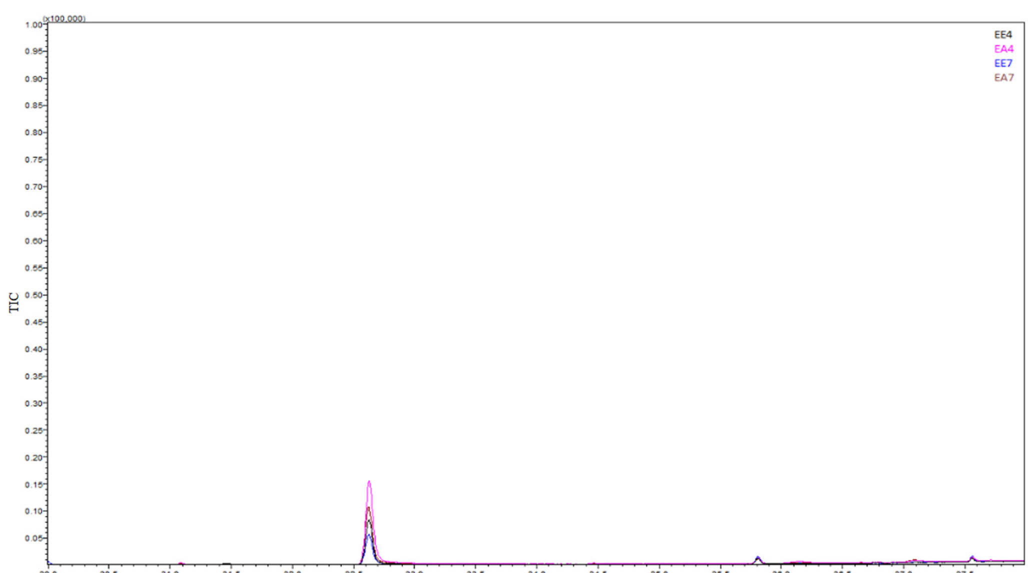

Figure 4. Chromatograms of samples of food packaging stored at room temperature and packaged food samples performed in SIM mode (m/z 149, 177 and 122), respectively: EE4 (cooked and smoked pepperoni sausage packaging); EA4 (cooked and smoked pepperoni sausage); EE7 (UHT milk – "soft belly" type packaging); EA7 (UHT milk – "soft belly" type)

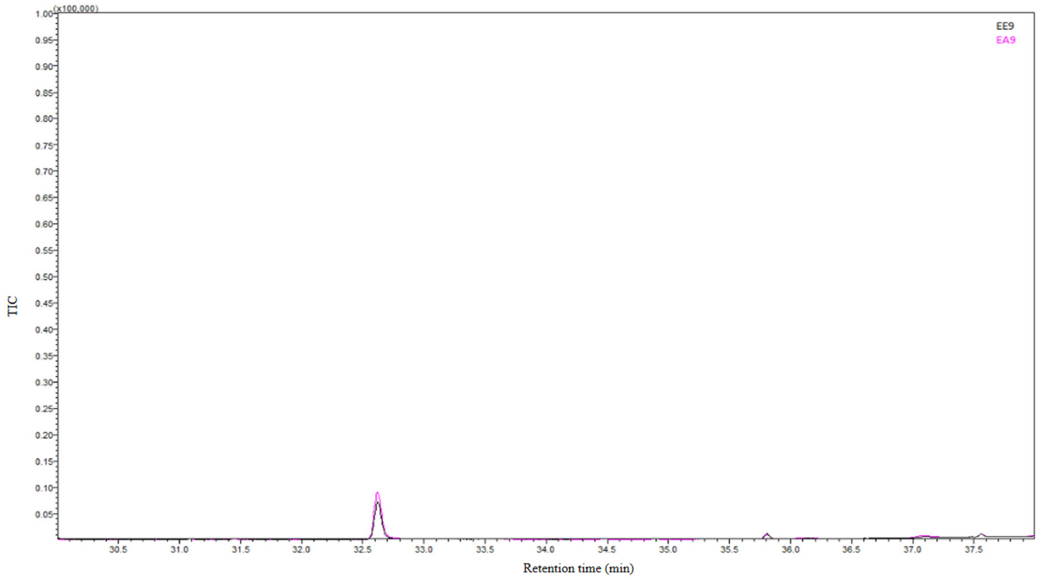

Figure 5. *Chromatograms of frozen and baked food sampled within the packaging and packaged food samples performed in SIM mode by m/z 149, 177 and 122, respectively: EE9 (frozen chicken thighs with 10% brine packaging); EA9 (frozen chicken thighs with 10% brine - water, salt, garlic, onion herbs, and additives)*

In addition to migration being facilitated in foods that underwent heat treatment inside the package,[10] researchers indicated another factor that enhances migration.[41,42] As well as storage time, phthalate concentration levels in food samples based on short shelf life and consumption with short storage time is much less than the acceptable quality assurance period of at least one year. Cheshmazar *et al.*[10] suggested that in subsequent studies, a careful survey should be made, under long-term supervision, of the kinetics of phthalate migration models from the walls of the flexible packaging to food. Among the analytes, the concentration of DEHP and di-*n*-butyl phthalate (DnBP) after 1 year grew about 30 and 15 times the standard limit (1.5 and 0.3 mg kg^{-1} for DEHP and DnBP) respectively in European Commission.[43] Jeddi *et al.*[44] found that after 180 days of storage, the predicted concentration of DEHP already exceeded the guidelines recommended by the U.S. FDA (6 mg L^{-1});[45] and WHO (8 mg L^{-1}).[46] Alp and Yerlikaya[36] also monitored the phthalate ester in seafood samples packed in polypropylene (PP), polyvinyl chloride (PVC), tin and glass containers for 4 months to determine the time-dependent migration of phthalate esters, and recorded that the highest DEHP value of 830.30 ng kg^{-1} in preserved PP-packed "*bonito tuna*" after the fourth month of storage. Consequently, the prolonged storage time of food in PET containers and PVC films, under acidic conditions, leads to the accumulation of phthalates in food, and their concentration will exceed the allowed limit. Therefore, the quality of food can vary during the course of warranty and long-term supervision of phthalates in food packaged in containers is vital.

Discussion about public health risk

Although the scientific literature indicates the connection with numerous adverse health effects, the limits for the presence of DEP from food contact packaging have not yet been established as health standards. This is also the situation for DEHT, which is also used as a plasticizer,[19] even though they are already considered as priority polluting substances, U.S. EPA,[47] and dangerous to the environment, ECE.[48] Among the group of phthalate compounds however, the limit for the use of DEHP has already been established as a technical support agent and in materials for repeated use in contact with non-fatty food products (0.1%), EC - European Commission,[43] EFSA,[49] MERCOSUR - GMC/RES No. 39/2019,[50] ANVISA - RDC No. 326/2019.[20] Regarding regulation in the US, the use of DEHP is approved as a plasticizer in food contact materials only for foods with high water content, 21-CFR-181.27, U.S. FDA.[18]

The result of the lack of restrictive legislation regarding the wide use of plasticizers by the industry, especially by the food industry, and specifically phthalates worldwide can be seen in numerous studies on the detection and quantification of phthalates. The occurrence, migration and risk assessment associated with estrogen activity and other adverse effects on human health is connected to the daily consumption of industrialized foods in flexible packaging. The occurrence of these plasticizers to foods and their metabolites is confirmed in several studies, and indicates the transmission of diseases to humans and their negative impact on the environment. According to Khaustov *et al.*,[51] the hypothesis about the insufficient level of barrier properties in PET packaging for water storage by changing properties over time and the release of substances harmful to health was confirmed by the results of the experiments. The method of packaging radically affects the composition of the food, causes loss of properties for consumption and acquires dangerous characteristics. Thus, the lack of reliable methods to determine phthalate concentrations in food and water greatly limits the justification of its normative values in adjacent media and the construction of kinetic models of its transformation in interaction with the components of the natural environment.

CONCLUSIONS

Certain types of phthalates, such as diethyl phthalate (DEP), are commonly found in a wide variety of foods and have been linked to health problems, even at low concentrations.

The development of effective methods to detect and track the migration of phthalates into food is crucial to protecting public health. In this study, a direct detection method using HS-GC-MS was proposed, which proved to be accurate, highly specific and adequate technique to identify traces of DEP in food samples. The use of headspace ensured that DEP is detected in samples of 9 EEs and 9 EAs, without the need for pre-treatment and use of any simulants.

Thus, it was verified that the detection of phthalates occurred under different storage conditions (temperatures, recommendations for use and expiration date of the manufacturer), consumption temperature and in different food matrices. It was also observed that heat treatment, after freezing, followed by heating in the oven, can enhance the migration of phthalates to foods inserted in the same package, according to the scientific literature.

Therefore, it is urgent to revise the health standards for flexible packaging, especially those that have direct contact with the surface of food. In this context, there is a demand for proposals for greater restrictions on the use of phthalates in flexible packaging, mainly for food, limiting their occurrence through the establishment of sanitary control and specific parameters. Such measures are crucial to minimize the risks associated with chronic and long-term exposure to phthalates, which can have serious consequences for public health.

Incidentally, the restrictive parameters established do not include the wide variety of phthalates used in Brazil. This therefore highlights the urgency of comprehensive regulatory action aimed at protecting consumers from the harmful effects of the wide range of phthalates, their specificities and their combinations.

This current study focuses solely on detecting the presence or absence of DEP using the HS-GC-MS method. Future studies could be carried out to enhance the analytical methodology and accurately quantify the contaminants migrating from food packaging, applying a direct methodology, without need for solvents in sample preparation and simulants, highlighting the need for methodological advances with real samples.

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