A GC-MS METHOD FOR IDENTIFICATION OF POTENTIAL CONTAMINANTS IN FOOD PLASTIC PACKAGING AND A GC-MS METHOD FOR DETERMINATION OF CONTAMINATS IN FOODSTUFFS

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INTRODUCTION

The safety of food packaging materials is a cause of concern since they can transfer chemical compounds into the food which may affect not only the safety but also the quality of the packaged food. In this work, the potential migration of chemicals from plastic food packaging into the food was investigated. A total of fourteen samples of dairy products were selected for this study and a GC-MS method was developed to quantify selected compounds in the foodstuffs. The method was validated in terms of linearity, repeatability and recoveries.

EXPERIMENTAL

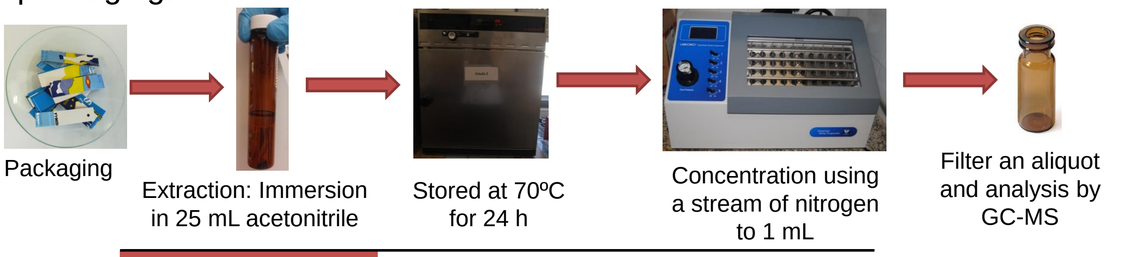
Fourteen samples of dairy products comprising milk, yogurt, flan and cheese, were purchased in a local supermarket of Santiago de Compostela. Labeling detailed information about each food was recorded, including fat/saturated fat (ranged from 1.6% to 35%). To verify the type of material, infrared spectra were acquired using an ATR (attenuated total reflectance)- FTIR spectrometer equipped with an optical crystal of diamond. The spectra identification was possible by comparing recorded spectra with several spectral libraries related with polymers and additives.

Coding	Type of sample	Fat	Part of the sample	Type of material	
				Internal side	External side
LE_01	Whole milk	3.6 %	Packaging	Polyethylene	Polyethylene
LE_02	Whole milk	3.6 %	Packaging	Polyethylene	Polyethylene
LE_03	Whole milk	3.6 %	Packaging	Polyethylene	Polyethylene
LS_01	Semi-skimmed milk	1.6 %	Packaging	Polyethylene	Polyethylene
LS_02	Semi-skimmed milk	1.9 %	Packaging	Polyethylene	Polyethylene
YN_01	Natural yogurt	2.9 %	Lid	Polyethylene terephthalate	Nitrocellulose
			Packaging	Polystyrene	Polystyrene
YS_01	Strawberry yogurt	1.9 %	Lid	Polyethylene terephthalate	Nitrocellulose
			Packaging	Polystyrene	Polystyrene
FN_01	Egg flan with caramel	1.8 %	Lid	Styrene-butadiene-isoprene rubber	Nitrocellulose
			Packaging	Polypropylene	Polypropylene
FN_02	Egg flan	3.2 %	Lid	Based in Methacylate	Based in Acrylate
			Packaging	Copolymer vinyl chloride/vinyl acetate	Epoxy resin
FN_03	Custard	3 %	Lid	Polyethylene terephthalate	Nitrocellulose
			Packaging	Polystyrene	Polystyrene
QS_01	Semi-cured cheese	35 %	Lid	Polyethylene	Polyethylene terephthalate
			Packaging	Polyethylene	Polyethylene terephthalate
			Intermediate sheet	Polystyrene	Polystyrene
QL_01	Molten cheese	13.5 %	External packaging	Polyethylene	Polyethylene terephthalate
			Internal packaging	Polyethylene	Polypropylene
QF_01	Mozzarella	18 %	Packaging	Polyethylene	Nylon (Polyamide)
QF_02	Pasteurized cheese	14 %	Lid	Polyethylene terephthalate	Based in Polyurethane
			Packaging	Polystyrene	Polystyrene

Table 1: Information about the samples of the study.

SCREENING OF THE POTENTIAL MIGRANTS IN FOOD PACKAGING MATERIALS

In a <u>first step</u>, a gas chromatography with mass spectrometry (GC-MS) method was developed to identify potential migrants in an extract of the packaging.



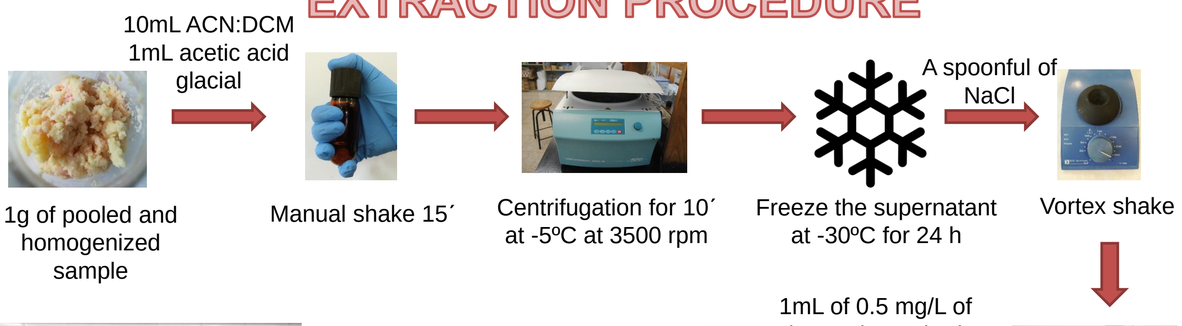
Column	ZB-5MS (30 m x 0.25 mm x 0.25 µm)
Carrier gas	Helium 1mL/min
Injection	Splitless mode
Injection volume	1µL
Tª program	Initial Tª: 40ºC/2min, 9ºC/min to 300ºC, 300ºC/3min
Data acquisition	Full scan (range m/z 35-500)
Mass detector	Electron impact
Transfer line Tª	300ºC
Detector Tª	300ºC
Spectrum library	Wiley 8th & NIST/EPA/NIH 11 Mass spectral library (version 2.0)

Table 2: Experimental conditions of GC-MS method.

QUANTIFICATION OF SELECTED MIGRANTS IN THE FOODSTUFFS

In the <u>second step</u> of the work, the packaged foods were pooled into three groups according to the population age (12-35 months, 3-9 years and 10-17 years) based on the Spanish consumption data (Enalia) and analyzed by GC-MS to quantify selected compounds.

EXTRACTION PROCEDURE



The experimental conditions of GC-MS method are the same as in the food packaging, except some differences: the injection mode split (1:5), the initial Tª in the oven is 60ºC and the data acquisition is in mode SIM.

RESULTS AND DISCUSSION

More than 90 compounds were identified in the food packaging and selected compounds were confirmed using standards. Among them, antioxidants as butylated hydroxytoluene (BHT); plasticizers as diethyl phthalate (DEP), diisobutyl phthalate (DIBP), acetyltributyl citrate (ATBC), bis(2-ethylhexyl)phthalate (DEHP), bis(2-ethylhexyl) terephthalate (DEHT), diethylhexyl adipate (DEHA); photoinitiators as benzophenone (BP); thermal decomposition product of polystyrene as 1,3-diphenylpropane (1,3-DPP); UV filters as octocrylene; and slip agents as hexadecanamide and erucamide.

Results of this study demonstrate the migration of phthalates from food packaging materials to foodstuffs, in concentrations that range from 0.02 µg/g of DEP to 0.16 µg/g of DBP (pool 10-17 years). All compounds present good linearity in the studied quantification range ($R^2 \geq 0.9900$). Appropriate recovery range at the three spiking levels (from 78.4 % to 124.4 %) and a good sensitivity was obtained with this method.

Compound	Sample Concentration (µg/g)	LOD (µg/g)	LOQ (µg/g)	Recovery (%)			Repeatability (RSD %)		
				0.25 µg/g	0.5 µg/g	1 µg/g	0.25 µg/g	0.5 µg/g	1 µg/g
DEP	0.02-0.03	0.01	0.025	112.0	113.9	102.2	16.1	8.36	2.50
BP	<LOD	0.05	0.1	107.1	103.6	102.1	14.9	7.67	2.03
1,3-DPP	<LOD	0.025	0.05	88.8	80.0	78.4	5.03	1.73	7.01
DIBP	0.05-0.10	0.005	0.005	112.1	123.1	124.4	5.40	2.80	1.61
DBP	0.09-0.16	0.005	0.005	119.8	123.9	123.0	8.52	0.98	4.26
DEHP	0.07-0.09	0.005	0.005	122.7	123.9	115.8	1.62	1.04	7.51
DEHT	<LOD	0.025	0.05	96.2	89.6	98.9	16.0	15.9	18.2

Table 3: Sample concentrations and method validation parameters.

Acknowledgement

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