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Extraction and Analysis of Pesticide residues from liquid milk- A Review

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Abstract

Dairy milk has a high incidence of contamination with persistent insecticides residues. Contamination of milk with a pesticide depends on its stability, its mode of application, the duration of the intake or exposure and its metabolic fate in the animal. Pesticides are extracted from solvents such as acetonitrile, petroleum ether, hexane, dichloromethane, or acetone. Cleanup of hexane extract with official method, SPE extraction, Acetonitrile petroleum ether followed by Florisil cleanup, QuEChERS (MSPD), extraction with hexane partitioned with acetonitrile and dichloromethane. The solvent is blended with the sample and homogenized. Pesticide compounds are separated either on gas chromatography (GC) or liquid chromatography (LC), and then identified and quantified using Electron-capture detection (ECD), flame photometric detection (FPD), nitrogen–phosphorus detection (NPD), fluorescence detection, and diode-array detection (DAD) depending on the molecules to be analysed. But role of GC and LC coupled with mass spectrometry (MS) and tandem mass spectrometry (MS/MS) in pesticide residue analysis is clear in both monitoring and research applications.

Key words: Milk, Pesticide, Extraction, Separation, Chromatography.

Introduction

Milk is an essential nutritional food for infants and the young [1,2]. Highly sensitive techniques need to screen pesticides in food items such as milk. Commonly found pesticide like, organophosphorus pesticides (OPs), are widely used to protect fields, fruit crops, parasite in domestic animals [3,4]. Intensive and indiscriminate use of OPs cause acute toxicity to humans and the environment [5]. The Ops affects nervous system , overstimulation of the nerves , weakness or paralysis of the muscles [6]. Therefore a demand for the development of reliable, sensitive, simple and low cost methods. Several reports for the presence of OPs in milk worldwide using various techniques [7-11].

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Several methods have been proposed for the analysis of pesticides in milk, such as extraction with non-polar solvents [12], solid-matrix dispersion [13,14] normal-phase liquid chromatography with column switching [15], liquid–liquid microextraction [16] and solid-phase microextraction (SPME) [17] and hotwater [18]. Solid-phase extraction (SPE) has been proposed for extracting triazine herbicides [19-20], phenylurea herbicides [21] and organophosphorus pesticides [22] from milk samples. In these methods, analytes detection relies on liquid chromatography (LC) with UV detection [19,21] or gas chromatography (GC) with mass spectrometry (MS) [20] and nitrogen–phosphorus detection (NPD) [22] in milk and milk derivatives.

Recent analytical developments to minimise physical and chemical manipulations like the solvent volumes, evaporation steps, the use of toxic solvent, and to automate the extraction and clean-up procedures [23]. Pesticide compounds are separated by gas chromatography (GC) or liquid chromatography (LC), and identified and quantified by different kinds of

detection methods. Electron-capture detection (ECD), flame photometric detection (FPD), nitrogen–phosphorus detection (NPD), fluorescence detection, and diode-array detection (DAD) were mostly commonly used for pesticide identification and quantification. Role of GC and LC coupled with mass spectrometry (MS) and tandem mass spectrometry (MS/MS) in pesticide residue analysis in both monitoring and research applications. Scientific documentation on analytical methods applied to pesticide determination in animal products is less abundant. This paper will aim to summarize the documentation published on the analysis of pesticide residues in milk over the past.

Sample preparation, extraction and cleanup

Milk is an emulsion of the oil-in-water type. Fat has a tendency to come to the surface, so attention has to be paid to get homogenized and representative samples. The sample should be handled carefully so that possible contamination is prevented and the loss of volatile pesticides is avoided. The analyte concentrations are generally very low and the sample matrix is complex, the interference of matrix should be considered when performing a measurement. So, the analysis of the sample requires studious sample preparation steps, extraction methods, and cleanup steps to minimize the interfering of the matrix. Most pesticide residue detection methods for food samples comprise two key preparation steps prior to identification/ quantification: extraction of target analytes from the bulk of the matrix, and partitioning of the residues in an immiscible solvent and/or clean-up of analytes from matrix co-extractives, especially fat which interferes with assays [24-26]. Pesticides are extracted from the sample employing solvents such as acetonitrile, petroleum ether, hexane, dichloromethane, or acetone. The solvent is blended with the sample and homogenized. In the case of milk, pesticides are either present in the fat phase in free form or bound to the lipoprotein or protein. So, acetonitrile is used to deproteinize the sample to release any pesticide bound to the protein and, at the same time, to precipitate the fat and protein and bring pesticides to a dissolved form. Then, the pesticides are partitioned to the petroleum ether phase. The extraction timing depends on the type of pesticide, matrix, and physicochemical properties of the solvent. The most common problem faced is the incomplete recovery and formation of emulsion during the partitioning process. This can be avoided by using a suitable solvent with sodium chloride added or by one or more solvent combinations. Recent analytical developments minimise the number of physical and chemical manipulations, the solvent volumes, the number of solvent evaporation steps, the use of toxic solvent, and have aimed to automate the extraction and clean-up procedures as far as possible [23].

Solid-phase microextraction (SPME) are example of promising new analytical techniques [27, 28]. SPME [29] has proven to be a powerful and useful technique to meet these needs, and it has emerged as a versatile alternative method of analyte extraction and preconcentration, which requires little or no organic solvents—thus does not generate poisonous residues—is easily automated, and can also improve the limit of detection. SPME encompasses sampling, extraction, preconcentration and introduction of the sample into the analytical system in a single uninterrupted process, thus avoiding contamination of the matrix. SPME is a miniaturized technique, in which the extraction and concentration processes of the analytes are carried out in dimensions that are different from solid phase extraction (SPE). In the first stage of SPME, a fiber of fused silica covered with a film of selective polymeric liquid, a solid phase, or both, is put in contact with the sample, which then results in partitioning or adsorption of the analyte between the matrix and the stationary phase. Soon afterwards, the fiber is transferred to an analytical instrument where the analytes are desorbed, separated and quantified [30-32]. SPME extraction can be performed either in the headspace (HS) or through direct insertion of fiber into the sample. HS-SPME is an attractive alternative for the extraction of organophosphorus compounds in complex matrices, such as milk, as it is simple, fast and possesses low manipulation of the sample and high sensitivity [33]. However, there are several parameters that should be optimized to obtain a greater efficiency in the extraction of organophosphorus compounds, including fiber type, the time and temperature of extraction, sample volume added to the extraction flask and time and temperature of desorption, among others [34].

Analysis of organochlorine pesticides and PCBs Fat samples were extracted and purified, following the method described by Sandmeyer (1992) with some modifications: 250 ml of milk were centrifuged during 15 min, at 4 ^{0}C at 17,300g; milk fat was removed and mixed with 25 g of anhydrous sodium sulfate and 100 ml of petroleum ether. Liquid was filtered through anhydrous sodium sulfate and evaporated under vacuum. The purified fat residue was transferred to a glass vial and kept at 20 ^{0}C prior to purification of compounds. Compounds were purified by the method of Martinez, Angulo, Pozo, and Jodral (1997). Briefly, 1 g of fat sample was mixed with 3 ml of n-hexane. The sample was applied to a chromatographic column containing 15 g of florisil and anhydrous sodium

sulfate, and eluted with 100 ml of n-hexane to extract organochlorine pesticides and PCB congeners. The eluate was filtered through anhydrous sodium sulfate, evaporated to dryness in a rotary evaporator, dissolved in 1 ml of n-hexane, and used for organochlorine pesticide and PCB congener determinations by gas chromatography with an Agilent 6890A model gas chromatograph equipped with a 63Ni microelectron capture detector (ECD).

The samples of liquid milk were analyzed for residues of organochlorines, organophosphates, and synthetic pyrethroids using a standardized multiresidue methodology. Purification of silica gel and anhydrous sodium sulphate About 500 g silica gel (60–120 mesh) was taken in a glass column, washed first with dichloromethane and second with acetone and air dried on Whatman No.1 filter paper. The silica gel was activated at 135^0C for 3 h. In a similar manner, anhydrous sodium sulfate was also washed and dried. A 5-g milk sample was thoroughly mixed with 20 g prewashed and freshly activated silica gel and 20 g anhydrous sodium sulfate (washed and dried) to form a free-flowing powder in a pestle mortar. The powdered sample was packed quantitatively with dichloromethane into an extraction glass column containing about 40mL dichloromethane over a plug of prewashed cotton. Care was taken to prevent air bubbles from being trapped inside the column. The column was stoppered and left for 90 min. Dichloromethane was eluted drop-wise from the column. When dichloromethane was about to reach the level of the adsorbent, the column was re-eluted with a 150-mL mixture of dichloromethane:acetone (1:2, v/v) to remove turbidity, anhydrous sodium sulphate was added to the elute. The volume was concentrated to about 2–3mL under vacuum at 35^0C. Hexane (10–15mL) was added and again the mixture was concentrated to about 5 mL. This was repeated to completely remove dichloromethane and the final volume was made with hexane [35-36].

Another extraction technique was that (5 g) samples of milk were transferred on top of a dry Chem Elut cartridge. After the liquid has drained into the cartridge wait for 15 min in order to obtain an even distribution on the filling material. A 32mm×0.70mm I.D. Luer Lock needle was attached to the lower tip as a flow restrictor and the column was eluted with three 5-mL portions of dichloromethane. A 50-mL round bottom flask, evaporated under vacuum to a small volume at a bath temperature of 40 ◦C and the last solvent traces were then removed by manually rotating the collecting flask. The residue was redissolved with 1.0 mL of mobile phase and analyzed by HPLC/GC. Evaporation of the extracts and reconstitution in low volumes of mobile phase was necessary in order to reach an adequate preconcentration of pesticides that allowed to obtain low limits of detection (LOD). Although water is the main component of whole bovine milk, high protein, fat and carbohydrate contents render it a complex matrix that produces high interference with the analytes to be determined and affects the method performance. Ready-to-use cartridges, filled with a diatomaceous earth material, have been already used in place of the usual liquid–liquid partitioning with solvents, to extract pesticides from food [13]. In the developed extraction step we used Chem Elut cartridges, from Varian, to extract and clean-up in a single step with dichloromethane the neonicotinoid insecticides from milk samples, obtaining very clean eluants. Whole bovine milk sample is added to the top of the dry Chem Elut cartridge. The high porosity, the high dispersing capacities and the high capacity for aqueous adsorption of the inert support ensures immiscibility of organic solvent and aqueous phase, avoiding emulsion formation and facilitating efficient interaction between the sample and the organic solvent. This procedure requires no sample preparation, no drying step with nitrogen flow and provides adequate clean-up of the lipid matrix.

Separation and detection

Gas chromatography coupled to mass spectrometry (GC/MS) is a powerful tool to separate, identify and quantify volatile organic compounds in the most types of complex matrices. The extraction/purification procedures, pesticide compounds are separated either on gas chromatography (GC) or liquid chromatography (LC), and then identified and quantified using different kinds of detection methods depending on the molecules to be analysed. Electron-capture detection (ECD), flame photometric detection (FPD), nitrogen–phosphorus detection (NPD), fluorescence detection, and diode-array detection (DAD) were mostly used for pesticide identification and quantification. Analytes detection relies on liquid chromatography (LC) with UV detection or gas chromatography (GC) with mass spectrometry (MS) and nitrogen–phosphorus detection (NPD) in milk and milk derivatives [19-22]. Neonicotinoid insecticides are a relatively new group of active ingredients with novel modes of action . For their distributionon large areas of agricultural land they could give rise to seriousrisks for the health and safety of the consumer. These insecticides are mainly determined by HPLC and several residue analyses for neonicotinoids with HPLC–MS and with HPLC–DAD [37] have been developed. Table-1 belows shows extraction of pesticide and analysis by specific

instrument along with recovery limit and LOD value. New technologies such as SPE, SPME, and MSPD are suitable to perform extraction and cleanup in a single step.

Table 1: Residual Analysis of Pesticides in Milk and Milk Products (Selected Methods)

Analyte	Extraction	Separation	Analytical System	Recovery(%)	LOD(μg/kg)	References
Organochlorine pesticides	Extraction with petroleum ether	Capillary DB-5 column	GC-ECD	91.0–99.1	0.01–0.03	[38]
Organochlorine pesticides	Cleanup of hexane extract with official method	DC-200Packed on chromosorb	GC-ECD	84.5–98.2	1	[39]
Herbicides and fungicides	SPE extraction	C-18	LC-MS	82–120	0.008–1.4	[40]
Organochlorine pesticides	Acetonitrile petroleum ether followed by Florisil cleanup	HP-1 Capillary column 30 m length	GC-ECD	90–94	1	[41]
Cypermethrin, fipronil, chlorfenvinphos	QuEChERS (MSPD)	DB-5 Capillary column	GC-MS	70–120	20	[42]
Organochlorine pesticides in butter	Extraction with hexane partitioned with acetonitrile and dichloromethane	Chromosorb OV-17	GC-ECD	85–92	100	[43]
48 compounds in milk	Acetonitrile acetone	C-18, 2.1 × 100 mm and ZB-50	UPLC/ Q-TOF and GC-MS/MS	70–85 70–100	—	[44]

The quantification of the target analytes is usually performed with gas chromatography or liquid chromatography separation, followed by detection using various detectors. Gas chromatography appears to be the most useful technique for the quantitative determination of pesticide residues in milk and milk products. Possible detectors in combination with GC

are electron capture detector (ECD), thermionic detectors such as nitrogen and phosphorus detector (NPD), flame photometric detector (FPD) or flame ionization detector, and mass selective detector (MSD). High-performance liquid chromatography is the second most frequently used technique to determine very polar and low volatile pesticides. The separation mostly occurs on reverse-phase packed columns. Different types of detectors for HPLC to determine pesticide residues are UV absorption, fluorescence, conductivity, electrochemical and mass spectrometer detectors. Liquid chromatography, along with a mass selective detector (MSD), is a very powerful technique for the quantification and confirmation of pesticide compounds . An HP-5 fused silica (cross-linked 5% phenyl methyl siloxane gum) column (30 m length, 0.25 mm internal diameter, 0.25 lm film thickness) was used. The carrier gas was nitrogen (1.5 ml/min). Oven temperature was set at 50 ^{0}C, increased at 40 ^{0}C /min up to 170 ^{0}C, increased at 2 ^{0}C / min up to 180 ^{0}C and held 1 min, increased at 5 ^{0}C /min up to 200 ^{0}C and held 1 min, increased at 1 ^{0}C /min up to 210 ^{0}C and finally increased at 25 ^{0}C /min up to 250 ^{0}C and held for 3 min. Injector and detector temperatures were 225 ^{0}C and 300 ^{0}C, respectively. All samples were analyzed in duplicate and results represent the arithmetic means. To determine the quality of the method, a recovery study was performed on ten replicates of milk fat samples overspiked with organochlorine pesticides and PCBs. Mean recoveries ranged from 72.0 to 121% for organochlorine pesticides and 97.4–105 % for PCBs, and the coefficient of variation was below 10%, indicating an excellent repeatability for the method. Limits of detection and quantification were determined using the average blank values method.

HPLC–DAD system and operating conditions

HPLC analyses were carried out on a HPLC system equipped with a continuous vacuum degasser, a P4000 quaternary pump and a UV6000LP detector linked to a personal computer running the ChromQuest-version 4.2 software program (ThermoQuest, Milano, Italy). The analytical column was a Synergi Hydro-RPC18 (250mm×4.6mm I.D., 4_m particle size) from Phenomenex (Torrance, CA, USA). A Security Guard column (Phenomenex) was used as pre-column. The mobile phase was a 30:70 (v/v) mixture of acetonitrile–water. The flow rate was 1.0 mL min^{-1}. The detections were performed at 271 nm for imidacloprid, at 253nm for thiamethoxam and at 244 nm for acetamiprid and thiacloprid, respectively. The injection volume was 20_L. The external standard method of calibration was used for this analysis. At least seven standard solutions (0, 0.01, 0.02, 0.05, 0.1, 0.2, 0.5 and 1.0mgL^{-1}) containing allcompoundswere analyzed by HPLC–DAD. The injection was performed three times to test the reproducibility. Calibration curves were obtained by plotting peak areas against concentrations of analytes injected [35,36].

Working parameters of GLC

ECD

The column specifications were a pyrex glass column (1m-2mm i.d.) packed with 1.5% OV-17+1.95% OV-210 on a Chromosorb W HP (80–100). The working conditions for organochlorines were as follows: column, 220^{0}C; injection port, 230^{0}C; detector, 260^{0}C; and nitrogen flow rate, 40mL/min.The working conditions for synthetic pyrethroids were as follows: column, 250^{0}C; injection port, 260^{0}C; detector, 280^{0}C; and nitrogen flow rate, 60 mL/min [35-36]

NPD

The column specifications were a pyrex glass column (1m length and 2mm i.d.) packed with 3% OV-101 on a Gas Chrom Q (100–120). The working conditions for organophosphorus compounds were as follows: column, 210 ^{0}C; injection port, 230^{0}C; detector, 250^{0}C; nitrogen flow rate, 40mL/min; hydrogen flow rate, 60mL/min and air flow rate, 100mL/min.The minimum detection limit utilizing the above method was 0.001 mg/kg for organochlorines and 0.01 mg/kg for synthetic pyrethroids and organophosphorus compounds. Liquid milk and butter samples, when spiked at 0.10 mg/kg with organochlorines, synthetic pyrethroids, and organophosphorous compounds, showed more than 90% recovery (Table 1). The residues detected in the samples were confirmed by alkali dehydrohalogenation with 2% alcoholic KOH and by use of an alternate 2% DEGS-packed GLC column [35-36].

Conclusion

In the present extraction procedure no sample preparation or pretreatment, such as precipitation of milk proteins is required. By using Chem Elut cartridges no preconditioning is required, an adequate clean-up of the lipid matrix is provided by a single extraction and no further purification is necessary. All that is a substantial advantage over the existing procedures used to purify milk samples, since the risk of losses of compounds is lowered and, mostly, the extraction procedure is time-saving. Whole milk extracts are very clean and suitable for the analysis by HPLC–DAD, with no interfering peaks at the retention time of the target compounds, indicating good selectivity of the proposed method. Pesticide compounds are separated either on gas chromatography (GC) or liquid chromatography (LC), and then identified and quantified using different kinds of

detection methods depending on the molecules to be analysed. Electron-capture detection (ECD), flame photometric detection (FPD), nitrogen–phosphorus detection (NPD), fluorescence detection, and diode-array detection (DAD) were mostly used for pesticide identification and quantification until recently. But the expanding role of GC and LC coupled with mass spectrometry (MS) and tandem mass spectrometry (MS/MS) in pesticide residue analysis is clear in both monitoring and research applications.

These observations are in contrast to earlier studies done in India, in which b-HCH residues were more frequently encountered. This significant decline in the residues of DDT and HCH seems to be the result of bans of their use in agriculture and public health programs. Although the frequency of occurrence of DDT residues was greater in butter than that of HCH, none of the butter samples exceeded the MRL of DDT. Lindane residues occurred at a lower frequency in butter than in liquid milk, and this was mainly attributed to the different physicochemical properties of lindane. Lindane residues in liquid milk are a matter of serious concern, as the estimated daily intake of lindane through the consumption of contaminated milk exceeds its ADI value for children.

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