Migration of Contaminants from Packaging Materials into Dairy Products*

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Abstract: Typical dairy products (DP) include milk, cheese, butter and yoghurt. Since they are consumed widely, people are concerned about their safety. However some contaminants in the packaging materials (PM) might migrate into them. Many researches have been made on the migration into simulants of DP. But little work has been done on DP themselves due to their complex matrices. Herein the migration of contaminants from PM into DP was reviewed from year 2006 to 2010. The contaminants from plastics include styrene monomer, bisphenol A (BPA), plasticizers, melamine (MEL), photoinitiators, diphenylbutadiene (DPBD), butylated hydroxytoluene (BHT) and metal elements. The contaminants from paper and cardboard include fluorochemicals, polychlorinated dibenzodioxins and −furans (PCDDs/PCDFs). PM comprise plastics, can, primary and recycled paper and board. In some cases just the amounts of the migrants in DP were determined. While in others mathematical models based on Fick's second law were built to predict the migration values, which fitted well with the experimental values. Diffusion coefficients at certain temperatures were predicted as well. Detection methods mainly included chromatography, chromatography/mass spectrometry and chromatography tandem mass spectrometry. Commonly adopted sample pretreatment methods were solvent extraction followed by solid-phase extraction (SPE).

Keywords: Migration; Contaminants; Packaging Materials; Dairy Products

1. Introduction

In recent years the outbreaks of several food safety issues have brought contaminants migrating from PM to people's attention. Some of them even at low level can be detrimental. Because migration study on DP themselves require tedious sample pretreatment and detection method with high selectivity and sensitivity, researchers have turned to the simulants of DP, including acetic acid (3% w/v), olive oil, ethanol, ethanol/water for liquid DP and Tenax TA for solid DP [1, 2]. However according to K. Grob et al., migration into the food should definitely prevail over migration into the simulants [3]. Herein migration of contaminants from PM into DP was reviewed from year 2006 to 2010. Most studied DP included milk powder, liquid infant formula, bovine milk and cheese. The determination of migrants in DP was extensively studied. The developed methods have been validated with acceptable limits of detection (LODs), limit of quantifications (LOQs), repeatabilities and recoveries. Specific migration limits (SMLs) were set by the EC Directive for many additives used in plastics materials and articles intended for food contact. The concentrations of migrated contaminants in DP were all below SMLs. The relevant literature was summarized in table 1, where it could be seen that for most mass spectrometry methods, internal standards (IS) were used to compensate for sample preparation losses, matrix effects and instrumental instability. Usually the IS was added to the sample before further treatment. As stated by European Union Directive 2002/72/EC (EC, 2002), verification of compliance of plastic packaging materials with the existing regulations can be done by comparing values of SMLs with values predicted by ‘‘generally recognized migration models" [4]. These mathematical models can minimize the need for time-consuming and expensive experiments. It has already become a practical tool for both producers of plastic materials and food inspectors. Some mathematical models were built successfully to describe the migration to DP [4-6].

2. Plastics monomer as contaminant

Styrene is the precursor to polystyrene and several copolymers. It is weakly toxic. The maximum concentration level permitted by European legislation is 60 mg/kg. L. M. Chiesa et al. determined its content in cheese using headspace solid phase micro-extraction gas chromatography coupled to mass spectrometry (SPME-GC-MS) and found that although the content was less than the legal limit there was enrichment due to migration [7]. SPME was used because it was volatile. P. LÓPEZ et al. determined styrene-containing volatile organic compounds (VOCs) from whole milk with 23% fat content, skimmed milk with 0.3% fat content, Tenax and Porapak Q. during heating using purge and trap GC-MS in selective ion monitoring (SIM) mode [8]. The results showed that Tenax was better used as simulant for

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milk powder with low or intermediate fat content than for whole milk powder. They concluded that temperature and fat content were the most important factors in mass transfer processes and then time. M. Khaksar et al. studied migration of styrene from polystyrene cups to hot drinks including milk using liquid chromatography coupled to UV detector (LC-UV). They reached the same conclusion about migration like above [9].

Table 1. Literature Summary

Contaminant	PM	DP	Pretreatment method	Detection method	IS	SML (mg/kg)	C_{DP} (mg/kg)
Styrene	Plastics Plastics PS cup	Cheese Milk powder Hot milk	SPME SPME Purge and trap	GC-MS GC-MS LC-UV	Nitrobenzene - -	-	0.61-0.80 Migration Migration
BPA	PC Can Can Can Can	Milk powder Liquid formula Milk Liquid formula Liquid formula	SPE SPE SPE SPE+derive SPE+derive	Micellar LC LC-ESI-MS/MS LC-ESI-MS GC-MS GC-MS	- d_6-BPA d_{16}-BPA d_{16}-BPA d_{16}-BPA	0.6	$0.19-0.24\times10^{-3}$ $0.48-11\times10^{-3}$ $<1.7, 15.2\times10^{-3}$ $2.3-12\times10^{-3}$ $2.27-10.2\times10^{-3}$
Plasticizer	PVC	Raw milk	DCM extract	GC-TOFMS	Heptacosa	3.0	<0.154 DEHP <0.017 DMP <0.099 DnBP
	Board	Milk powder	ASE	GC-MS	$[^2H_4]$-phthalates	0.5	$<64.8\times10^{-3}$ DiBP $<53.0\times10^{-3}$ DnBP
MEL	Plastics Plastics Plastics Plastics	Milk, cheese Infant formula Milk powder Milk powder	SPE ACN extract ACN extract ACN extract	DART-TOFMS LC-MS/MS LC-MS/MS tITP-CE-UV	$^{13}C_3$-MEL ($^{13}C_3$,$^{15}N_3$)-MEL ($^{13}C_3$,$^{15}N_3$)-MEL -	30	0.46-2.53 <2.96 10.5 Not detected
PIs	LDPE	Milk powder	ACN extract	LC-DAD	-	BP0.6	Migration
	Carton	Milk	SPE	GC-MS	-	BP0.6	$5.3-39\times10^{-3}$ BP $<0.8\times10^{-3}$ EHDAB
DPBD	LDPE LDPE	Milk powder Cheese	HX extract ACN+HX extract	LC-DAD LC-DAD	- -	-	Migration Migration
BHT	Plastics	Cheese	ACN+HX extract	GC-MS	MM	3.0	Spiked
FCs	Paper	Butter	HX+TFE extract	LC-ESI-MS	FC-C	-	Migration
PFCs	Carton	Milk Milk powder Yoghurt	SPE	LC-ESI-MS/MS	MPFOA+MPFOS	-	178×10^{-3} 98×10^{-3} 42×10^{-3}
PCDDs/PCDFs	Carton	Cow's milk	Pentane extract	HRGC/HRMS	^{13}C-UL-PCDD/PCDF	-	0.19×10^{-3}
DiPN	P&B	Milk powder	Ethanol extract	GC-MS	-	-	Migration

PS, polystyrene; PC, polycarbonate; DCM, dichloromethane; HX, hexane; MM, methyl myristate; TFE, trifluoroethanol

3. Plastics additives as contaminants

Bisphenol A (BPA) is used to make polycarbonate plastic and epoxy resins coatings of the can. It could migrate from the can to milk powder, concentrated liquid formula, ready-to-eat liquid formula and whole evaporated milk [10-14]. Various techniques were employed to analyze its contents. Samples were extracted with organic solvents like acetonitrile (ACN) before purification by solid phase extraction (SPE). SPE cartridges include C_{18} [10-13] and styrene-divinylbenzene EnvChrom-P [14]. For liquid chromatography coupled to mass spectrometry (LC-MS) [10], negative electrospray ionization (ESI) interface was used because BPA was more likely to lose a hydrogen ion. SIM was used for quantification. L. K. Ackerman et al. utilized SPE-LC-negative ESI-MS/MS (tandem mass spectrometry) to monitor relative retention times and ion ratios [14]. Two diagnostic product ions from each of the chosen precursor ions were needed for unambiguous confirmation. BPA was quantified using the area ratio of one BPA transition (m/z 227-133) to the isotopically labeled d_6-BPA transition (m/z 233-138). When SPE-GC-MS was used [12, 13], tedious derivation of BPA has to be made for GC analysis. Confirmation for this method was based on the retention time and the ion ratios. In addition, as the derivative of BPA, bisphenol A diglycide ether (BADGE) could disappear in milk by acting with milk proteins to form protein adducts with unknown toxicity [15].

Plasticizers like di(2-ethylhexyl) phthalate (DEHP), di-n-butyl phthalate (DnBP) and dimethyl phthalate (DMP) have been used for polyvinyl chloride (PVC) products. Using GC-TOF (time-of-flight) MS, M. Kim et al. determined the respective concentration of DEHP, DnBP and DMP in raw milk [16].

Melamine (MEL) and its analogue cyanuric acid (CA) form insoluble (MEL-CA) crystals which can precipitate in kidney tubules and cause damage of renal tissue. High-throughput, fully automated quantitative analysis of MEL and CA in dried milk, condensed milk and dried cheese was performed using direct analysis in real time (DART) ion source coupled to TOFMS [17]. After extraction and disruption of MEL-CA with methanol-5.0% formic acid, supernatant obtained after centrifugation was analyzed. Compared to LC-MS/MS, DART-TOFMS did not need chromatographic separation or incubation steps and could provide measurement in real time. But it had rather higher LOD than that of traditional LC-MS procedures. A. Desmarchelier et al. developed LC-MS/MS method to determine MEL in milk-based infant formulas and milk powder [18]. Sample preparation was very simple with no clean-up by SPE to avoid any plastic-derived contamination of the analytes during the sample preparation. Quantitative analysis was performed using tandem MS in selected reaction monitoring (SRM) mode alternating two transition reactions for each compound and their corresponding IS. Xiaoyu Wang et al. determined MEL in milk powder using transient isotachophoresis (tITP-CE-UV) [19]. After precipitation of proteins with 50% ACN, the supernatant was evaporated and reinstituted in leading electrolyte solution for analysis. By tITP, a longer plug of sample was introduced leading to a 200-fold improvement of sensitivity.

Photoinitiators (PIs) are catalysts for photopolymerization, which include Irgacure 186, benzophenone (BP), Irgacure 651, Irgacure 907, isopropyl thioxanthone (ITX) and EHA. A. Sanches-Silva et al. studied the migration from low density polyethylene (LDPE) film additivated with above PIs to milk powder [5]. After elimination of proteins with ACN, the supernatant was analyzed by HPLC-diode array detector (DAD). The migration experiments were performed to investigate the migration mechanism. Samples of milk powder were placed in contact with the plastic (one side only). Then they were wrapped in aluminum foil and placed inside a transparent plastic bag. Samples were vacuum packed to achieve intimate contact. Then they were stored under 5℃, 25℃, 40℃ for up to 30 days. The mathematical model based on Fick's second law was used to describe migration of an additive or contaminant from an amorphous polymeric packaging film to DP:

$$\frac{\partial C_p}{\partial t} = D_p \frac{\partial^2 C_p}{\partial x^2} \quad (1)$$

Where C_P (mg/kg) is the concentration of the migrant in the packaging film at time t (s) and position x (cm) and D_P is the diffusion coefficient in the film (cm^2/s). D_P is replaced by an effective (for the whole polymer-food system) diffusion coefficient D. Equation 1 can be resolved to express the amount of migrant released per area unit from the polymer into food at time t. Experimental data were fitted to the model by nonlinear regression. From the data, the model parameters D, and $K_{P/F}$ partition coefficient of the migrant between film and DP were calculated. The fit between experimental and estimated values was evaluated by means of the mean-square error (% RMSE). The low RMSE values indicate a good correlation between experimental and predicted migration values and feasibility for the model to predict migration. Factors that influence migration to DP are identities and concentrations of the migrants in the PM including molecular weight and lipophicity, the intrinsic properties of the packaging material itself, fat content of DP, the condition of contact, the surface area of the packaging material in contact with food, temperature and time of contact [2, 4]. Among plastics, the highest diffusion rates occur in polyolefins, especially LDPE films. Because this type of film is widely used and can represent the worst migration situation, it is therefore chosen for the migration tests. For a given migrant-polymer system and under controlled/fixed time/temperature conditions, migration depends mostly on fat content. Fats can penetrate into plastics to induce swelling or they can leach generally lipophilic migrants due to their ester function. Smaller molecular weights correspond to higher diffusion coefficients. Diffusion coefficients increase with temperature. A. Sanches-Silva et al. drew the conclusion that the Arrhenius relationship could be used to estimate D values at any temperature in the range between 5 and 40℃ [5]. D at 5℃ ranged between 8.4×10^{-12} for ITX and 5.1×10^{-10} cm^2/s for BP and those at 40℃ ranged between 5.9×10^{-10} (for ITX) and 6.1×10^{-9} cm^2/s (for Irgacure 184). G. Sagratini et al. tested ITX, BP, EHDAB, 184 and EDAB in milk and packaging [20]. The pretreatment included *n*-hexane extraction and SPE (Si Silica cartridge) clean-up. Compared to other methods like LC-DAD, LC-MS, LC-ESI-MS-MS, GC-MS gave the lowest LODs and LOQs, then it was used for quantification in SIM. LC-APPI-MS/MS was used to confirm these results. Atmospheric pressure photo ionization (APPI) was operated in positive mode because the migrants all contain carbonyl.

A. Sanches-Silva et al. studied the migration kinetics of optical brightener diphenylbutadiene (DPBD) from LDPE into whole milk powder with 25% fat content [6]. The result showed that D were 3.2×10^{-10} and 1.8×10^{-9} cm^2/s at 25℃ and 40℃ respectively. J. M. Cruz et al. studied the migration of DPBD, triclosan and butylated hydroxyl-toluene (BHT) from migrants-additivated LDPE into cheese with fat content ranging from 4.51% to 35.7% [4]. Samples were vacuum-packed and stored at 5

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℃ and 25℃ respectively. D of DPBD at 5℃ ranged between 3.2×10^{-11} to 1.12×10^{-9} cm^2/s. Diffusion coefficients of DPBD within cheeses D_{cheese} were calculated according to Moisan's equation. They were 2.4 and 6.4×10^{-8} cm^2/s for soft and Gouda cheeses respectively, which were much higher than those in the mass transport system (diffusion in polymer and transfer to food). Therefore they believed that the migration from LDPE to the cheese was the rate-limiting factor. A. Sanches-Silva et al. also determined the spiked BHT in soft cheese, Gouda cheese and other food as a result of migration from plastic packaging [21]. They compared the performance of LC-UV and GC-MS and found GC-MS gave better recoveries.

E. P. Soares et al. studied migration of metal elements Co, Cr and Sb from high density polyethylene (HDPE) to DP. These elements originate from catalysts and different types of plastics additives. The migration results were 0.12 in Co, 2.17 in Cr and lower than 0.09 $\mu g \cdot dm^{-2} \cdot kg^{-1}$ in Sb respectively [22].

4. Paper additives as contaminants

Fluorochemical-treated paper, grease/oil resistant, is generally used for food in the fast-food industry. T. H. Begley et al. studied the migration of fluorochemicals (FCs) or so-called polyfluoroalkyl phosphate surfactants from paper to butter (an emulsified food with 78% fat) and simulants using LC-ESI-MS [23]. Greater migration was observed into butter than simulants. Under certain circumstances, oil-containing emulsifier is recommended as the fatty-food simulant. Perfluorooctanoic acid (PFOA) is residual and toxic contaminant in the fluorochemical paper coating. Its content in paper was determined as well. J. M. Wang et al. studied the mean concentrations of total perfluorinated compounds (PFCs) in milk, milk powder and yoghurt [24]. The PFCs levels in milk packaged in three kinds of carton packaging were significantly different.

P. W. Ackermann et al. tried to find if there was still migration of toxicologically relevant polychlorinated dibenzodioxins and –furans (PCDDs/PCDFs) from the cartons into cow's milk when the cartons were no longer bleached with elemental chlorine [25]. High resolution gas chromatography coupled with high resolution mass spectrometry (HRGC/HRMS) was used and no significant migration was found.

5. Contaminants from recycled paper and board (P&B)

As the result of more and more attention paid to preservation of natural resources, the use of recycled P&B as food PM has increased. The major incorporated contaminants are diisopropylnaphthalenes (DiPNs) and phthalates. DiPNs originate from photocopy paper and phthalates are associated with inks. Studies have been made on their kinetic migration to milk powder or simulants like Porapak and Tenax [26-28]. The migration into milk powder was the lowest compared to that into the simulants and at high temperature Porapak was recommended rather than Tenax [26]. V. I. Triantafyllou et al. investigated migration of ten model contaminants at 70 and 100℃ from recycled paper fortified with above contaminants to baby cream and milk powder respectively [27]. They found very high migration for DiPN, DBP and methyl stearate while low migration for volatile compounds due to the loss in the vapor phase. Low grammage and high recycled pulp content of paper resulted in greater migration. At high temperature, fat can melt and penetrate into the paper to extract the contaminants. S. Gartner et al. studied the migration of phthalates including diisobutyl phthalate (DiBP) and DnBP from recycled paperboard into infant food like milk powder and semolina powder [28]. They found that the migration via gas phase transfer followed the kinetics of a zero-order reaction. They also concluded that paper was not an appropriate barrier to the transfer of nonpolar and volatile contaminants into dry food independent of its fat content. The application of secondary packaging like aluminum-coated foil could somewhat prevent the contamination.

6. Conclusion

The migration of contaminants from PM to DP was reviewed. The migration mechanism was discussed. Since most of DP contain high content of fat, contaminants migrate easily. In order to avoid this contamination, new PM like edible films have been developed. Since edible films have many advantages like edible, biodegradable, pollution-free, flavor-retaining and gas-barriering, they were used to package DP like cheese [29]. However due to their high cost and immature manufacturing process, their application to package DP is quite limited.

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