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Published in:
International Journal of Food Contamination

Link to article, DOI:
10.1186/s40550-016-0026-6

Publication date:
2016

Document Version
Publisher's PDF, also known as Version of record

Petersen and Jensen *International Journal of Food Contamination* (2016) 3:3
DOI 10.1186/s40550-016-0026-6

International Journal of Food Contamination
a SpringerOpen Journal

SHORT REPORT Open Access

Phthalates in soft PVC products used in food production equipment and in other food contact materials on the Danish and the Nordic Market 2013-2014

Jens Højslev Petersen* and Lisbeth Krüger Jensen

Abstract

Background: Food contact materials (FCM) containing phthalates can be a source of food contamination when used in plastics for food production equipment, in utensils for food contact and in packaging. Since 2008 several of the phthalates used for FCM were regulated in the EU; some of them because they were well-known endocrine disruptors. Results of the Danish Food Authorities control in 2008 and 2009 showed 23 % non-compliant samples. Critical FCMs turned out to be those made from plasticised PVC and sold as suitable for contact with fatty foodstuffs. Targeted follow up control campaigns were therefore arranged by the Danish food authorities (latest in 2013) and by the Nordic food authorities in a common campaign in 2014.

Findings: FCM plastics were analysed for phthalate content and when needed additionally tested for migration of phthalates according to the declared area of use with respect to food type, contact temperature and time in contact with food. In both recent control campaigns about 1/3 of the samples analysed exceeded the current maximum limits for phthalates (especially DBP and DEHP) in plastics or showed migration into the fatty food simulant above the specific migration limits. Critical sample types were conveyor belts, hoses and gloves.

Conclusions: Legal limits for phthalates were exceeded in many of the samples analysed in recent tests, including a large proportion of conveyor belts and gloves. The proportion of non-compliant conveyor belts, hoses and gaskets was lower in 2013 and 2014 than in 2008-2009, whereas the proportion of non-compliant gloves increased.

Keywords: Food Contact Materials (FCM), Official control, Phthalate, Migration, DEHP, DBP, DiNP, DiBP

Findings

This paper contains a collection of results from two targeted enforcement campaigns in 2013 and 2015, planned and carried out together with the Food Authorities in Denmark (Holm et al. 2013) and in the other Nordic Countries (Li et al. 2015), respectively. The Food Authorities were responsible for sampling of Food Contact Materials (FCM) and collecting documentation of compliance (DoC). The National Food Institute was responsible for the analytical work and migration tests. Similar campaigns were conducted in Denmark in 2008 and 2009 and it was expected to see a reduced number of non-compliances with the regulation.

Legislation

Since 2008, plastic FCM's in the EU market shall comply with the restrictions and specifications for the following phthalates: Dibutyl phthalate (DBP), Butylbenzyl phthalate (BBP), Di- (2-ethylhexyl) phthalate (DEHP), Di-isononylphthalate (DiNP) and Di-isodecylphthalate (DiDP). In some cases, the phthalates used in FCM are regulated by maximum permitted residual contents (weight % concentration in the FCM, here referred to as Quantum maximum, Qm) and in others by SML's, Specific Migration Limits (mg/kg food simulant). The interpretation of the restrictions is not straight forward and is presented in more detail in a guideline from the EU reference laboratory (EURL) for FCM (Hoekstra et al. 2011) where a table of the critical parameters to control in enforcement is shown

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(Table 1). Control of the migration limits for phthalates was performed with the official food simulant for FCM in contact with fatty foods according to the Plastics Regulation (EU) No 10/2011 (EU, 2011).

In the two recent control campaigns official food inspectors were asked to take samples of soft FCM plastics, which either were claimed by the producer, importer or retailer to be suitable for contact with fatty foodstuffs or which were found by inspections to be used for this purpose in the food industry. Detailed instructions were given with respect to sample types, number and size of items. Before selling their product on the European market, producers and importers of plastic FCM's are obliged to fill out a DoC claiming compliance with the EU regulation among other requirements stating any restrictions in the legal area of use for the final FCM. Information about acceptable time and temperature-range in use as well as limitations, if any, of food types in contact with the FCM are among the most important parameters. Such information is needed when the official control laboratory decides how to test the plastic material for compliance with the legal limits in the plastic and in the food simulant.

Analytical Strategy

The accredited analysis of the above five phthalates in our laboratory was described earlier in details by Petersen and Jensen (2010). In brief the following strategy was applied: the FCM was first analysed with infrared spectroscopy to verify that the sample was a plastic material. If not, the sample was discarded. Secondly, the phthalate concentration in the plastic was determined by dissolution/precipitation (e.g., PVC) or extensive extraction of the polymer (e.g., polyolefins) followed by analyses of the extract using gas chromatography with mass selective detection (GC-MSD). The sample was deemed acceptable if all phthalates were below the Qm-limits. If the phthalate concentration exceeded the Qm, the use intended by the supplier and the real use were checked to conclude on whether or not the FCM was compliant (Table 1). If so, no further testing was performed and the sample was deemed non-compliant. If the sample complied with Qm-limits but contained phthalates, migration tests were performed according to EU 10/2011, Annex V, with the food simulant olive oil or 50 % ethanol taking any restrictions mentioned in the DoC for contact time and temperature into consideration (EU 2011). The oil was analysed using the EURL-method of Bratinova et al. (2010). Phthalates in 50 % ethanol were transferred to an organic solvent using liquid/liquid extraction after dilution of the simulant with water. After testing the sample in one test (single use FCM's) or three times in a single sided test with new portions of food simulant (repeated use FCM's) it was concluded if the sample complied with the SML's or not. In Table 3 sample characteristics, type of test applied and result of the analysis is shown for each individual sample.

Results and discussion

In Table 2 results from the recent control campaigns are compared with results from the FCM control in 2008/2009 (Petersen and Jensen, 2010). The overall picture shows that the non-compliant FCM's belong to only a few sample types: food production equipment containing PVC like conveyor belts, hoses and gloves for single or repeated use. In these categories there continue to be a high proportion of illegal products on the market. In contrast, problems with high amounts of phthalates plasticisers in the gasket of screw caps for glass containers seen in 2008/2009 seems to be solved. However, it was reported that there are other problems with migration of substances substituting phthalates as primary plasticisers (McCombie et al. 2015). Samples of PVC cling film did not contain any of the five phthalate plasticisers.

For **conveyor belts** typical problems were a high concentration of DiNP and/or DiDP in the PVC plastic layers leading to a high migration into fatty foods. Many belts contained also DBP or DEHP in the plastic in

Table 1 Clarification of the critical parameter of "classical" phthalates for control in enforcement work (adapted and slightly simplified from Hoekstra et al. 2011)

PM-no	Substance	SML	Qm	Parameter to control in *single use* Food Contact Material *			Parameter to control in *repeated use* Food Contact Materials		
		mg/kg food simulant	% in plastic	Fatty food	Infant food	Non-fatty food	Fatty food	Infant food (non-fatty)	Non-fatty food
74640	Phthalic acid, bis(2-ethylhexyl)ester (DEHP)	1.5	0.1	Qm			Qm	SML	
74880	Phthalic acid, dibutyl ester (DBP)	0.3	0.05 #	Qm			Qm	SML	
74560	Phthalic acid, benzyl butyl ester (BBP)	30	0.1	Qm		SML	SML		
75100	Phthalic acid, diester with C8-C10 (DiNP)	9 $	0.1	Qm		SML(T)	SML(T)		
75105	Phthalic acid, diester with C9-C11 (DiDP)	9 $	0.1	Qm		SML(T)	SML(T)		

* Packaging made from glasses with lid containing a plasticized gasket is usually considered as a single use material; # only permitted in polyolefins; $ SML(T) is sum of DiNP and DiDP

Qm, maximum permitted quantity of the residual substance in the material; SML, specific migration limit

Table 2 Survey of results from three control campaigns about phthalates in food contact plastics. Total number of samples analysed in different types of FCM and the frequency of violations

Type of food contact material	Danish control 2008/2009 **)			Danish control 2013 ***)			Nordic control 2014 ****)		
	Samples analysed	Non-compliances No./%	Type of violation (Qm/SML)	Samples analysed	Non-compliances No./%	Type of violation (Qm/SML)	Samples analysed	Non-compliances No./%	Type of violation (Qm/SML)
Conveyor belt	6	6/100	6/0	12	8/67	4/4	3	2/67	1/1
Hose	5	4/80	4/0	7	2/29	1/1	3	1/33	1/0
Gloves	20	5/25	5/0	5	2/40	2/0	3	3/100	3/0
Gasket in screw cap/lid	28	8/33	8/0	6	0	0/0	4	0/0	0/0
Various sample types *)	41	0/0	0/0	1	0	0/0	6	0/0	0/0
Total	100	23/23		31	12/39		19	6/32	

*) plastic film, cling film, bottles, containers, cutlery, kitchen and tableware
) Petersen and Jensen, 2010; *) Holm et al., 2013; ****) Li et al. 2015

amounts below 1 % but above the Qm-limits, presumably because they are used as a production aid in an inner adhesive layer. It was shown earlier that phthalates readily diffuse into the food contact layers after production of the laminated conveyor belt since it contained no functional barrier against migration (Petersen and Jensen, 2010). The non-compliant PVC-**hose** from 2014 contained DEHP as the primary plasticiser. It could be expected that DEHP would have been substituted with the less toxic DiNP or DiDP, which was the case in the 2013 campaign where one PVC hose contained DiDP as primary plasticiser.

Suppliers of food production equipment like conveyor belts and hoses are reluctant to set restrictions for the legal area of use for their products. If needed, the DoC should state to the user of the FCM that it is not suitable for direct contact with fatty foodstuffs. Clear statements of the maximum temperature and contact time for safe use of the FCM is needed. However, in many cases the food inspectors did not find any relevant documentation, when they took the samples (Table 3). Annex 5 in the plastics regulation (EU 2011) says that: "verification of compliance of migration into foods with the migration limits shall be carried out under the most extreme conditions of time and temperature foreseeable in actual use". For hoses and conveyor belts there is a risk that the contact time with food can be quite long in the case there are minor stops in the production, and the FCM must be tested accordingly. Food is considered to be the primary source of human intake of phthalates like DBP and DEHP and food production equipment is a potential source of such food contamination. Many of our foodstuffs contain relatively low levels of DEHP, a substance which has a rather low tolerable intake per kg bodyweight (TDI). Because of the high intake of food per kg bodyweight for small children it could be shown e.g., by Fierens et al. (2012) that the DEHP exposure can be close to the TDI. Since endocrine disruptors like DEHP and DBP easily can be substituted with other less toxic substances, there is no reason to use them in such critical applications.

With respect to **gloves** the situation remains very unsatisfactory. It was shown years ago that use of DEHP plasticised gloves in catering could lead to an increased intake of DEHP e.g., for patients in hospitals (Tsumura et al. 2003). Single use PVC gloves, often in 100 pcs boxes, are cheap and frequently used but have a very high content of plasticiser. DEHP-plasticised gloves are still on the market without statements in the DoC and on the packaging, that they are unsuitable for contact with infants foods and fatty foods. In one case the gloves were labelled correctly but used in catering for fatty foods.

In the 2008/2009 campaign the food inspectors were asked to sample all kind of plastic samples provided they were claimed to be suitable for fatty food contact. It transpired that all findings of phthalates were in PVC plastics and in the following campaigns inspectors were urged to concentrate sampling on this type of polymer. However, over the whole period a total of 48 samples made from polymers other than PVC were analysed and in no case were phthalates present.

Conclusions

The overall conclusion from the control is that with 23 to 39 % non-compliant samples in the recent campaigns there is still a lot of room for improvement. Importers and producers must be better at compiling the DoC correctly to guide the users of the FCM to avoid excessive migration of endocrine disruptors like phthalates – which could easily be replaced with less toxic substances. Findings of phthalates in plastic FCM was often connected to the polymer PVC. Plasticised PVC and phthalates is a problematic cocktail, when used in contact with fatty foods.

Table 3 Detailed sample data and test results from enforcement campaigns in 2013 (Denmark) and 2014 (Nordic Countries)

			Concentration in the plastic (%)					Limitations in use (from suppliers DoC etc.)			Test conditions (laboratorys assessment)			Reduction factor D2 applied	Results, migration into food simulant (mg/kg)		
Sample no. (13-x = DK; 14-x = Nordic)	Country of origin	Sampling Type	DBP	BBP	DEHP	DiNP	DiDP	Foods excluded	Max Time (hours)	Max Temperature (°C)	Food Simulant	Time (hours)	Temperature (°C)		DEHP	DiNP	DiDP
13-13		Bag in Box	<0.02	<0.02	<0.01	<0.03	<0.03										
13-1	Germany	Conveyor belt				high content			0,25	40	oil	0,5	40	3		**17.8 (12.8-22.0-18.5)**	
13-5		Conveyor belt				high content			short	100	oil	0,5	70	3		6.6 (5.7-7.7-6.2)	
13-12		Conveyor belt				high content					oil	0,5	70	3		**30 (30.8-31.7-26.7)**	
13-21		Conveyor belt	<0.02	<0.02	<0.01	<0.03	<0.03										
13-22		Conveyor belt	<0.02	<0.02	<0.01	<0.03	<0.03	some fatty	2	70							
13-23		Conveyor belt			0,026	high content			2	40	oil	2	40	3		**9.1 (9.1-9.5-8.7) #**	
13-24		Conveyor belt	**0.26 (0.27-0.27-0.25)**	<0.02	<0.01	high content											
13-25		Conveyor belt	<0.02	<0.02	**0.52 (0.50-0.63-0.42)**	high content											
13-26		Conveyor belt	**0.78 (0.50-0.96-0.89)**	<0.02	<0.01	high content	3.7										
13-27		Conveyor belt	<0.02		<0.01	high content$				70							
13-28		Conveyor belt				>0.1 %				70	oil	0.5	70	3		**28 (25.3-26.4-33.3)**	
13-29		Conveyor belt	<0.02	high content	**0.25**	high content											
13-2		Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03										
13-3		Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03										
13-4		Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03										
13-11	Germany	Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03										
13-19	Italy	Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03		1	100							
13-20	Czech Republic	Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03										

Table 3 Detailed sample data and test results from enforcement campaigns in 2013 (Denmark) and 2014 (Nordic Countries) *(Continued)*

13-15	Thailand	Gloves, repeated use	<0.02	<0.02	<0.01	<0.03	<0.03										
13-16	Japan	Gloves, repeated use	<0.02	<0.02	<0.01	<0.03	<0.03										
13-17	Monaco	Gloves, repeated use				detected		fatty			50 % EtOH	2	40			<0.33	
13-9		Gloves, single use	<0.02	<0.02	<0.01	**13.9¤**	<0.03	fatty									
13-30	China	Gloves, single use	<0.02	<0.02	<0.01	>0.1 %	**3.0 (3.3-3.4-2.3)¤**										
13-10		Hose					25.7 (27.3-24.7-25.0)	fatty		60	50 % EtOH	2	60				**95 (97.8-98.4-87.7)**
13-14	Turkey	Hose	<0.02	<0.02	**0.62 (0.54-0.68-0.64)§**	<0.03	<0.03	fatty									
13-18	Ireland	Hose	<0.02	<0.02	<0.01	<0.03	<0.03										
13-31	France	Hose	<0.02	<0.02	<0.01	<0.03	<0.03			60							
13-6		Milk hose	<0.02	<0.02	<0.01	detected	<0.03				50 % EtOH	0.5	40			<0.33	
13-7		Milk hose	<0.02		'0.04	<0.03	<0.03										
13-8		Milk hose	<0.02	<0.02	<0.01	<0.03	<0.03										
14-2	Italy	Conveyor belt	**0.28**	<0.02	<0.01	high content	<0.03		2	80							
14-3	Germany	Conveyor belt	<0.02	<0.02	<0.01	high content	<0.03		short term	90	oil	0.5	70	3		**27 (27-25-28)**	
14-13		Conveyor belt	<0.02	<0.02	<0.01	<0.03	<0.03										
14-7	Germany	Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03			121							
14-10	France	Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03		1	100							
14-16	Italy	Gasket in Lid	<0.02	<0.02	<0.01	<0.03	<0.03	fatty	1	100							
14-18	Italy	Gasket in Lid for wine	<0.02	<0.02	<0.01	<0.03	<0.03										
14-14	Taiwan	Gloves, repeated use	<0.02	<0.02	**28.2 (30.8-24.6-29.2)**	<0.03	<0.03										

Table 3 Detailed sample data and test results from enforcement campaigns in 2013 (Denmark) and 2014 (Nordic Countries) *(Continued)*

14-5	China	Gloves, single use	<0.02	<0.02	<0.01	**34**	<0.03	fatty*							
14-15	Taiwan	Gloves, single use	<0.02	<0.02	<0.01	**43**	<0.03	fatty							
14-6	Italy	Hose	<0.02	<0.02	0,08	max 0,1 %	<0.03		2	40	oil	2	40		<0.3 (3x < 0.3)
14-8	Finland	Hose	<0.02	<0.02	<0.01	<0.03	<0.03		1	40					
14-19		Hose	<0.02	<0.02	**25.8 (25.9-24.3-27.3)**	<0.03	<0.03	None		60					
14-1	Finland	Polyolefin film	<0.02	<0.02	<0.01	<0.03	<0.03		0.5	85					
14-4	France	PVC cling film	<0.02	<0.02	<0.01	<0.03	<0.03		0.25	100					
14-9	Finland	PVC cling film	<0.02	<0.02	<0.01	<0.03	<0.03	pure fats		40					
14-11	Germany	PVC cling film	<0.02	<0.02	<0.01	<0.03	<0.03	Food with D2 < 4	0.25	100					
14-12	Germany	PVC cling film	<0.02	<0.02	<0.01	<0.03	<0.03								
14-17	Italy	PVC cling film	<0.02	<0.02	<0.01	<0.03	<0.03		2	70					

Results shown in bold are above the legal limits
*In practice used in contact with fatty food, although this was not recommended by the supplier
$ Production stopped
§ Declared as phthalate free hoses
¤ Qm value for infant food violated
Non-significant violation of the migration limit

Competing interests
The author declare that they have no competing interests.

Authors' contributions
LKJ carried out the experimental and analytical work. JHP made the analytical strategy, planned and coordinated the control campaigns in collaboration with the Food Authorities in Norway, Sweden, Finland, Island, Faroe Islands and Denmark. JHP designed and drafted the manuscript which was read and approved by both authors.

Received: 2 December 2015 Accepted: 3 March 2016
Published online: 09 March 2016

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