PROCEEDINGS OF THE CLOSING CONFERENCE OF THE EU PROJECT “FOODMIGROSURE” QRLT-2001-2390

Modelling Migration from Plastics into Foodstuffs as a Novel and Cost Efficient Tool for Estimation of Consumer Exposure from Food Contacts Materials

G. Beldi, F. Franchini, C. Simoneau (eds)
The mission of the IHCP is to provide scientific support to the development and implementation of EU policies related to health and consumer protection.

The IHCP carries out research to improve the understanding of potential health risks posed by chemical, physical and biological agents from various sources to which consumers are exposed.
Table of contents

EXECUTIVE SUMMARY .................................................................................................................. 5
PROJECT PARTNERS ..................................................................................................................... 6
CONFERENCE PROGRAMME ........................................................................................................ 7
ORAL PRESENTATIONS ................................................................................................................ 9
POSTER PRESENTATIONS ............................................................................................................ 271
SNAPSHOTS OF THE EVENT ...................................................................................................... 325
ORGANISERS ............................................................................................................................. 333
SATISFACTION SURVEY ............................................................................................................. 335
Executive Summary

Food contact materials (FCM) such as plastic food packaging materials facilitate the preservation, protection and distribution of high quality foodstuffs, nowadays expected by the consumer. As food packaging materials come into contact with foodstuffs fairly extensively, it is also recognised that chemical substances can be released and migrate from the packaging materials into foodstuffs during processing distribution or storage.

One important aspect towards Health and Consumer Protection in the European Union’s is the exposure of the consumer to undesirable chemicals in the diet. FCM are one potential contamination source and therefore of particular interest for food exposure assessments. Scientific investigations concerning the migration potential and behaviour of food packaging materials have demonstrated that diffusion in and migration from FCM are foreseeable physical and in principle, mathematically describable processes.

The main objective of the project was to provide a novel and economic tool for estimation of consumer exposure to chemicals migrating from food contact materials. The tool was based on a physico-chemical migration model that describes mathematically the migration processes from plastics into actual foodstuffs under any foreseeable contact conditions. A further objective was to investigate the social acceptance of migration modelling versus chemical measurements, and its implications for exposure estimation.

The whole project work was divided into a design shown here.

The scientific conference exhibited the results of comprehensive sets of migration data for a wide range of substances and large sets of various foodstuffs. The data obtained allowed to validate a migration models on real foods both for compliance as worst case or refined for exposure assessment. The results also showed aspects of food chemistry that can influence migration, and initiated a new area of public risk perception specific to FCM safety. These systematic kinetics studies have a direct impact on Directive 85/572/EC on correspondence factors of foodstuffs and food simulants; The results obtained also showed an impact on 2002/72/EC on allowing the use of mathematical modelling, as the data is used to validate the current diffusion model for further applications; in addition The project showed an impact on risk assessment from systematic kinetics of migration related to real foods in different conditions for exposure assessment (EFSA), as well on standardisation (CEN) from the development of methods in foodstuffs. The conference was organised both around keynote speakers of the different workpackages as well as in day external speakers from different branches of stakeholders exemplifying the added value of the project for their field. The Conference also had a large expo area where the detailed scientific work was exhibited as posters around the coffee breaks. The event gathered 140 participants from both the EU as well as Norway, Switzerland, US, Japan, Thailand etc. The participants were also evenly distributed between industrial stakeholders (40%), National Reference Laboratories/Government (30%), and institutions, associations, academia etc (30%).
Project partners

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Further information:  
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Fraunfofer Institute for Process Engineering and Packaging IVV  
Giggenhauser Str. 35  
85354 Freising  
GERMANY
Conference Programme

27th September 2006

11:00-13:00 Registration
13:00-13:10 Welcome
13:10-13:50 Introduction and significance
   D. Bennink, European Commission - DG RTD
   R. Franz, IVV, D
13:50-14:10 Selection of plastics and migrants
   I. Cooper, PIRA, UK
14:10-14:30 Selection of foodstuffs
   I. Steiner, TUV, AU
14:30-14:50 Development of tailored methods for migrants in foods
   P. Paseiro, USC, ES
14:50-15:10 Systematic migration studies in foods
   R. Franz, IVV, D
15:10-16:00 Coffee and poster session
16:00-16:30 Development, verification and validation of migration model for foods
   R. Brandsch, FABES
16:30-17:00 Relation to exposure assessment
   L. Castle, CSL, UK
17:00-17:30 A risk management view towards the safety of food contact materials
   A. Schaefer, European Commission - DG SANCO
17:30-18:00 Summary and open floor
   R. Franz

Dinner Sponsored by Nestlé
28th September 2006

Public participation in the project

09:00-09:45 Consumer attitudes studies to packaging safety, migration and modelling
   C. Simoneau, European Commission - DG JRC

09:45-10:00 Video

10:00-11:00 Coffee and poster session

Implications of the project at the EU level

11:00-11:20 From migration to exposure: FDA experience
   T. Begley, FDA, USA

11:20-11:40 A EU consumer exposure assessment view
   P. Oldring, Exposure Vision Group

11:40-12:00 The additives and plastics industry view
   C. Gueris, representing CEFIC-FCA/Plastics Europe

12:00-12:20 The plastics converters view
   G. Tillieux, EUPC

12:20-12:40 The food industry view
   A. Mandanis, Nestlè

Lunch Sponsored by Cefic

14:00-14:20 National Enforcement Laboratory view
   X. Trier, DFVF

14:20-14:40 Risk communication and heritage from the EU TRUST project
   L. Pellizzoni, ISIG

14:40-15:00 Future support at DG RTD for research on food packaging
   D. Bennink, European Commission - DG RTD

15:00-15:30 Conclusions

15:30-16:00 Closing of the conference
Oral Presentations
Introduction and significance

D. Bennink,
European Commission - DG RTD

R. Franz,
IVV, D
FOODMIGROSURE
CLOSING CONFERENCE

Introduction

Baveno, 27 September 2006

Dyanne Bennink
European Commission
Directorate-General Research
Directorate E: Biotechnology, Agriculture and Food
Unit E.2: Food Quality

European Food Research Programmes
EU projects in FP5

5th Framework Programme QoL-KA1

- Electronic sensor system for the characterisation of packaging emissions (ESCAPE) 1.289,284
- Safe & eco-efficient packaging solutions for the food industry (ECO-PAC) 752,880
- Enhancement & indication of food quality by combinations of oxygen scavenger & quality indicator systems for polymer packagings (ACOSIC) 1.631,416
- Application of bioassays for safety assessment of paper and board for food contact (BIOSAFEPAPER) 998,879

EU projects in FP5

5th Framework Programme QoL-KA1

- Modelling migration from plastics into foodstuffs as a novel & cost efficient tool for the estimation of consumer exposure from food contact materials (FOODMIGROSURE) 1.186,270
- Food safety in Europe: Risk assessment of chemicals in food & diet (FOSIE) 754,000
- Ensuring the safety of consumers: Can coatings for direct food contact (CANCO-Workshop) 38,000
EU projects since FP5

5th Framework Programme MTI: Measurements, Testing and Infrastructure

• Calibration of sensory testing of food contact materials: Paper & Board 384,836

• Certified reference materials for specific migration testing of plastics for food packaging needed by industry & enforcement laboratories 624,781

EU projects in FP6

6th Framework Programme Food Quality and Safety

• Promoting Food Safety through a New Integrated Risk Analysis Approach for Foods (SAFEFOODS)
  www.safefoods.nl 11,376,001

• Novel Processing Methods for the Production and Distribution of High-Quality and Safe Foods (NovelQ) 10,900,000
harmonisation of risk assessment for chemicals in food

**FOSIE Coordination Action**

QLK1-CT-1999-00156:

to obtain ‘State of the Art’;
to examine means to improve principles;
to identify knowledge gaps;
to determine nature & level of testing & modelling.

---

**to demonstrate validity for realistic exposure linked to migration**

G6RD-CTG2000-00411:
to test plastics for specific migration;
to develop know-how to prepare & certify RMs for specific migration

QLK1-CT-1999-00156:
stochastic modelling of intake & exposure (MONTECARLO)

QLK1-CT-2002-02390:
to provide & validate an economic tool for estimation of consumer exposure to chemicals migration from food contact materials
Future ??
EU Project QLK1-CT2002-2390

Modelling migration from plastics into foodstuffs as a novel and cost efficient tool for estimation of consumer exposure from food contact materials

Roland Franz, Fraunhofer IVV, Freising GERMANY

EU Project QLK1-CT2002-2390

First of all:

Thank you all for your interest in our project and for coming!
Contents

- Why did we get into this project?
  [or:
   Why was the project unavoidable?]

- Intention & structure of the project

Why the project?

Migration tests should be

simple and reproducible

and have therefore been carried out so far using in the first place

food simulants
and not foodstuffs.
**EU Project QLK1-CT2002-2390**

**Why the project?**

On the other hand: EU legislation foresees that for compliance

the concentration (migration) *in food*

is the **crucial** one compared to the *food simulant test.*

---

**EU Project QLK1-CT2002-2390**

**Why the project?**

And:

Exposure estimation or assessment should (must) be based on

the concentration (migration) *in food*

and not on migration values in *food simulants.*

---
Why the project?

Consequently,

A correlation is needed between food simulants and foods, which is the intention of EU Directive 85/572/EEC.

However, problems were getting more and more evident over the years........

<table>
<thead>
<tr>
<th>Food item</th>
<th>RF</th>
<th>Relative migration default measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chocolate spread</td>
<td>X/3</td>
<td>0.33 up to 1</td>
</tr>
<tr>
<td>Chocolate</td>
<td>X/5</td>
<td>0.2 up to 1</td>
</tr>
<tr>
<td>Salami</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cocoa powder</td>
<td>X/4</td>
<td>0.25 up to 0.88</td>
</tr>
<tr>
<td>Mayonnaise (low fat)</td>
<td>X/3</td>
<td>0.33 up to 0.83</td>
</tr>
<tr>
<td>Mayonnaise</td>
<td>X/5</td>
<td>0.33 up to 0.80</td>
</tr>
<tr>
<td>Peanuts</td>
<td>X/2</td>
<td>0.2 up to 0.52</td>
</tr>
<tr>
<td>Butter</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cheese (low fat)</td>
<td>X/3*</td>
<td>0.33 up to 0.19</td>
</tr>
<tr>
<td>Whole milk</td>
<td>(A)</td>
<td>n.a. up to 0.27</td>
</tr>
<tr>
<td>Irish cream liqueur</td>
<td>C</td>
<td>n.a. up to 0.45</td>
</tr>
<tr>
<td>Flour</td>
<td>(NT)</td>
<td>0 up to 0.42</td>
</tr>
<tr>
<td>Biscuit (rich tea)</td>
<td>(NT)</td>
<td>0 up to 0.58</td>
</tr>
<tr>
<td>Bread</td>
<td>(NT)</td>
<td>0 up to 0.08</td>
</tr>
</tbody>
</table>

(Source: EU Report 19376 EN)
Why the project?

More recent examples from the ‘ITX crisis’ [photo initiator in printing inks transferred to food via set-off] revealed again weaknesses of the 85/572/EC system

More recent examples from the ‘ITX crisis’ [photo initiator in printing inks transferred to food via set-off] revealed again weaknesses of the 85/572/EC system

<table>
<thead>
<tr>
<th>Product</th>
<th>Pack size (ml)</th>
<th>ITX (µg/l)</th>
<th>EHDAB (µg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appleacerola juice</td>
<td>200</td>
<td>45</td>
<td>16</td>
</tr>
<tr>
<td>Apriicot nectar</td>
<td>200</td>
<td>63</td>
<td>33</td>
</tr>
<tr>
<td>Orange juice</td>
<td>200</td>
<td>115</td>
<td>48</td>
</tr>
<tr>
<td>Orange juice</td>
<td>200</td>
<td>294</td>
<td>86</td>
</tr>
<tr>
<td>Multivitamin drink</td>
<td>200</td>
<td>30</td>
<td>17</td>
</tr>
<tr>
<td>Multivitamin juice</td>
<td>200</td>
<td>249</td>
<td>125</td>
</tr>
<tr>
<td>Peach nectar</td>
<td>200</td>
<td>98</td>
<td>35</td>
</tr>
<tr>
<td>Pineapple juice</td>
<td>200</td>
<td>53</td>
<td>24</td>
</tr>
<tr>
<td>Pineapple juice</td>
<td>200</td>
<td>32</td>
<td>10</td>
</tr>
</tbody>
</table>

(Source: EFSA opinion on ITX and EHDAB in FCM, dated 7 December 2005)

... altogether 45 products measured with found concentrations for
- ITX between 20 ppb and 250 ppb
- EHDAB between 10 ppb and 125 ppb
**EU Project QLK1-CT2002-2390**

**Why the project?**

**ITX and EHDAB levels in “clear” drinks (fruit juices/nectars)**

(Source: EFSA opinion on ITX and EHDAB in FCM, dated 7 December 2005)

<table>
<thead>
<tr>
<th>Product</th>
<th>Pack size (ml)</th>
<th>Juice content (%)</th>
<th>ITX (µg/l)</th>
<th>EHDAB (µg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apple juice</td>
<td>1000</td>
<td>100</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Apple juice</td>
<td>1000</td>
<td>100</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Apple pure drink</td>
<td>1000</td>
<td>6</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Cherry drink</td>
<td>1000</td>
<td>15</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Cranberry &amp; Raspberry</td>
<td>1000</td>
<td>21</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Cranberry drink</td>
<td>1000</td>
<td>5</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Elderflower drink</td>
<td>1000</td>
<td>22</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Froot drink</td>
<td>1000</td>
<td>15% alcohol</td>
<td>&lt; 5</td>
<td>&lt; 5</td>
</tr>
</tbody>
</table>

... altogether 17 products measured and in all cases found concentrations were not detectable, i.e. < 5 ppb

**EU Project QLK1-CT2002-2390**

**Why the project?**

**ITX and EHDAB levels in milk and soy beverages**

(Source: EFSA opinion on ITX and EHDAB in FCM, dated 7 December 2005)

<table>
<thead>
<tr>
<th>Product</th>
<th>Fat content (%)</th>
<th>ITX (µg/l)</th>
<th>EHDAB (µg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UHT milk</td>
<td>3.8</td>
<td>142</td>
<td>71</td>
</tr>
<tr>
<td>UHT milk</td>
<td>1.5</td>
<td>177</td>
<td>92</td>
</tr>
<tr>
<td>UHT milk</td>
<td>0.1</td>
<td>54</td>
<td>27</td>
</tr>
<tr>
<td>Soy milk</td>
<td>1.5</td>
<td>219</td>
<td>134</td>
</tr>
<tr>
<td>Soy milk vanilla</td>
<td>1.5</td>
<td>170</td>
<td>90</td>
</tr>
<tr>
<td>Soy milk &amp; juice</td>
<td>0.6</td>
<td>137</td>
<td>71</td>
</tr>
<tr>
<td>Chocolate milk</td>
<td>2.9</td>
<td>295</td>
<td>148</td>
</tr>
</tbody>
</table>

It should be noted that water and 10% ethanol would give ‘not detectables‘ (< 5ppb)
Why the project?

Further advantageous circumstances were that...

... enormous progress has been made over the last decade in migration modelling, in particular for mono-plastics in contact with food simulants, but also for multi-layer plastics.

... essential developments have been made in the area of certified or certifiable reference plastics films in relation to migration relevant specifications.

EU Project QLK1-CT2002-2390

Modelling Migration from Plastics into Foodstuffs as a Novel and Cost Efficient Tool for Estimation of Consumer Exposure from Food Contact Materials

Project specification:

Contract: QLK1-CT2002-2390
Starting date: 01/03/2003
Duration: 36 months

Scientific Officer:

Dr. Achim Boenke
European Commission DG Research
E-mail: Achim.Boenke@cec.eu.int

Total project costs: 2.35 Mio €
EU contribution: 1.19 Mio €
The basic equation to calculate FCM related exposure:

$$ \text{Exposure} = \sum_{i} C_i \cdot P_i \left( M_i \right) \sum_{i} C_i $$

where:
- $C_i$ = consumption rate of a particular food $i$
- $P_i$ = relative packaging usage of a given FCM for a particular food $i$
- $M_i$ = migration rate from a given FCM into a particular food $i$
- $n$ = number of foodstuffs considered for the exposure estimation

via migration modelling
Main Objective

was to develop an into-food migration model as a novel and economic tool that may be applicable in two different ways:

1. As a stand alone tool to estimate exposure related migration within the conventional frame conditions of the EU food regulatory evaluation system thus applying a worst case exposure scenario, i.e.:

\[ \text{Exposure} = \sum M_i \]

2. In conjunction with statistical data obtained from food consumption and plastics packaging surveys to estimate realistic or worse-case exposure for any situation of interest, i.e.:

\[ \text{Exposure} = \sum C_i \cdot P_i \cdot M_i \]

\[ \sum C_i \]

---

Project structure

Ian Cooper
Perfecto Paseiro
Roland Franz
Rainer Brandsch
Laurence Castle
Ingrid Steiner
Catherine Simoneau
Expected Benefits

... for EU Commission and EFSA

**In general:**
Supporting present and future EU legislation for FCM

**In particular:**
- Scientific/technical basis for actualisation of Directive 85/572/EEC issues
- Bringing more light into the dark related to meet specific restrictions (SMLs) on the level of foodstuffs
- Supporting and plausibilisation of exposure estimations in context with petitions and setting specific restrictions

Expected Benefits

... for Industry

**Packaging industry:**
- Early (in the packaging development process) and food type specific compliance evaluation
- Cost efficient compliance ensurance

**Food industry:**
- Improving or even establishing independent house-internal quality assurance tools and this in a better product specific way
- More knowledge about diffusion and partitioning processes or organic compounds in foods
Expected Benefits

.... for the Consumer
including ‘consumer protectors’

- Better and more effective surveillance of FCM
- (Further) familiarisation with the idea of ‘Migration Modelling’ and increasing acceptance of calculated migration values
- (Somewhat) cheaper packed foodstuffs?

And with the possible effect of ...

decreasing the FCM related conflict potential and generating more confidence in FCM safety?

www.foodmigrosure.com

Thank you,
also on behalf of the FOODMIGROSURE project team
Selection of plastics and migrants

I. Cooper, PIRA, UK
Selection of polymeric films and model migrants to study the physico-chemical parameters influencing mass transport of packaging constituents into and within foodstuffs

Ian Cooper, Hélène Robin

Pira International

The experimental work

- **Kinetic migration studies WP3b**
  - Kinetic migration experiments carried out on polymer test films containing model migrants. Polymer test films need to be well characterised.

- **Diffusion/partitioning experiments WP3c**
  - Measurement of thickness related concentration profile of model migrants in food after exposure of the food to a release matrix containing model migrant.

  *5 test films and at least 15 model migrants needed*
### Desirable properties of the model migrants (1)

- Should represent a wide range of potential migrants in food packaging
- Should be measurable in a wide range of foods down to a concentration of about 30 ppb – straightforward analysis required
- Must not be present in foods, either naturally occurring or a food additive

### Desirable properties of the model migrants (2)

- Should cover a range of molecular weights, yet still have a propensity for migration
- Should cover a range of chemical types, chemical structures
- High stability in food required
- Ideally should be relevant to food contact materials
- $D_p > D_F$
Test films


• 16 plastics, containing potential migrants, were prepared and evaluated in 1st phase of project.

• 6 of these plastics selected for 2nd phase were produced in larger batches and evaluated by ring trial (4 labs) for $C_{P,0}$, $D_P$ and specific migration into food simulants.

• Homogeneity ($C_{P,0}$, density and thickness) stability, and diffusion characteristics established.

Results - $C_p$

![Graph showing $C_{p,0}$ of Chimassorb 81 in HDPE](image)
Four of these films selected for study in FOODMIGROSURE

Fifth film (well characterised) was sourced from Fraunhofer Institute National project.
<table>
<thead>
<tr>
<th>Film and Lab</th>
<th>Chemical name of model migrant</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 LDPE (JRC)</td>
<td>Benzenepropanoic acid-3,5-bis(1,1-dimethylethyl)-4-hydroxyoctadecyl ester (1076)</td>
<td><img src="image1" alt="Structure" /></td>
</tr>
<tr>
<td>2 LDPE (USC)</td>
<td>Diphenylbutadiene (DPBD)</td>
<td><img src="image2" alt="Structure" /></td>
</tr>
<tr>
<td>3 HDPE (PIRA)</td>
<td>2-hydroxy-4-n-octyloxybenzophene (C81)</td>
<td><img src="image3" alt="Structure" /></td>
</tr>
<tr>
<td></td>
<td>2,5-bis(5-tert-butyl-2-benzoxazoyl)thiophene (UOB)</td>
<td><img src="image4" alt="Structure" /></td>
</tr>
<tr>
<td>4 PA (CSL)</td>
<td>Caprolactam</td>
<td><img src="image5" alt="Structure" /></td>
</tr>
<tr>
<td>5 HDPE (TUV)</td>
<td>Benzophenone</td>
<td><img src="image6" alt="Structure" /></td>
</tr>
<tr>
<td></td>
<td>Diphenyl phthalate</td>
<td><img src="image7" alt="Structure" /></td>
</tr>
</tbody>
</table>

### Model migrants for WP3c

<table>
<thead>
<tr>
<th>Model migrant for WP3c</th>
<th>MW</th>
<th>PM REF No.</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bis(2-ethylhexyl)adipate (DEHA)</td>
<td>370</td>
<td>31920</td>
<td><img src="image8" alt="Structure" /></td>
</tr>
<tr>
<td>Styrene</td>
<td>104</td>
<td>24610</td>
<td><img src="image9" alt="Structure" /></td>
</tr>
<tr>
<td>2,2-Bis(4-hydroxyphenyl)propane (Bisphenol A)</td>
<td>228</td>
<td>13480</td>
<td><img src="image10" alt="Structure" /></td>
</tr>
<tr>
<td>1-octene</td>
<td>112</td>
<td>22660</td>
<td><img src="image11" alt="Structure" /></td>
</tr>
<tr>
<td>Limonene</td>
<td>136</td>
<td>63970</td>
<td><img src="image12" alt="Structure" /></td>
</tr>
</tbody>
</table>
### Model migrants for WP3c

<table>
<thead>
<tr>
<th>Model migrant for WP3c</th>
<th>MW</th>
<th>PM REF No</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diisopropyl naphthalene (DIPN)</td>
<td>212</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laurolactam</td>
<td>197</td>
<td>19490</td>
<td></td>
</tr>
<tr>
<td>Triacetin (GTA)</td>
<td>218</td>
<td>57760</td>
<td></td>
</tr>
<tr>
<td>Tri-n-butylacetyl citrate (ATBC)</td>
<td>402</td>
<td>93760</td>
<td></td>
</tr>
<tr>
<td>2,6-di-tert-butyl-p-cresol (BHT)</td>
<td>220</td>
<td>46640</td>
<td></td>
</tr>
<tr>
<td>2,4,4’ Trichloro-2”-hydroxy-diphenyl ether (Triclosan)</td>
<td>290</td>
<td>93930</td>
<td></td>
</tr>
</tbody>
</table>
Selection of foodstuffs

I. Steiner, TUV, AU
SELECTION OF FOODSTUFFS - Fundamentals

- To classify foodstuffs/food groups with respect to those physico-chemical properties which can be expected to influence the behaviour of food as a sorption matrix for the uptake of migrants.
- To select a list of approximately 15 to 20 (max. 25) foodstuffs which can be considered to be representative based on the following criteria:
  - with respect to the spectrum of migration relevant physico-chemical parameters
  - according to up-to-date and available food consumption statistics and market share. This should at least include the foodstuffs most frequently consumed and deserve therefore consideration at higher priority.
Establishing the final list of foodstuffs

- From the aspects considered before the two representative and established lists of foodstuffs may vary to a small or even large extent.
- Some harmonisation was necessary to end up with one representative list which fulfilled the best the purpose of this project idea which is to model migration into foodstuffs for exposure estimates as broad and general as possible.
Categories of food

- Aqueous and acidic foods
- Fatty foods
- Dry foods

Aqueous and acidic foodstuffs

- Orange juice, unsweetened, fresh with pulp
- Apple sauce
- Tomato ketchup
- Carbonated beverage (cola drink)
- Beer
- Wine
- Milk, consumer milk, min. 3.5% fat
### Fatty foodstuffs (I)

- Margarine (80% fat content)
- Mayonnaise (80% fat content)
- Cheese Philadelphia (~70% fat in dry matter)
- Cheese Gouda, 45% fat content (in dry matter)
- Cheese sauce (18.5% fat)
- Cottage cheese (fresh cheese, ~10% fat content in dry substance)

### Fatty foodstuffs (II)

- Whipping cream, UHT (~30% fat)
- Condensed milk (10% fat)
- Yoghurt drink, (0.9% fat)
- Chocolate, dark, milk free, min. 40% cocoa content (30% fat)
- Chocolate spread (25% fat)
- Meat, lean pork meat (minced, fat content ≤ 5%), mixed with lard to get fat contents up to 50%
- Salmon
Physico-chemical specifications of selected foodstuffs

- establish the representative physico-chemical parameters for the foodstuffs finally selected before
  - The measurements were done in the majority of cases according to standard or state-of-the-art methods
  - or were adopted from the labelling of the foodstuff.

⇒ basis for a representative and general applicability of the model

Dry foodstuffs

- Milk powder
- Butter toast (4% fat)
- Wheat flour
- Rice
- Honey
### Physico-chemical parameters in detail

- Water content
- Solids (dietary fibre)
- Carbohydrates
- Proteins
- Fat
- Alcohol
- Ash (minerals)
- pH
- Density
- Mass-to-volume ratio ("Schüttgewicht")
- Structure
- Particle size
  - Median particle diameter
  - Mean specific surface area

### Data of aqueous and acidic foodstuffs

<table>
<thead>
<tr>
<th>Food</th>
<th>Water (%)</th>
<th>Fat (%)</th>
<th>Solids (%)</th>
<th>CH (%)</th>
<th>Protein (%)</th>
<th>pH</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orange juice</td>
<td>87,1</td>
<td>0,15</td>
<td>12,9</td>
<td>12</td>
<td>---</td>
<td>3,95</td>
<td>1,05</td>
</tr>
<tr>
<td>Apple sauce</td>
<td>79</td>
<td>0</td>
<td>---</td>
<td>21</td>
<td>0</td>
<td>3,60</td>
<td>1,10</td>
</tr>
<tr>
<td>Tomato ketchup</td>
<td>64,5</td>
<td>0,20</td>
<td>35,5</td>
<td>29</td>
<td>1</td>
<td>3,20</td>
<td>1,17</td>
</tr>
<tr>
<td>Cola drink</td>
<td>89</td>
<td>---</td>
<td>11</td>
<td>10,6</td>
<td>---</td>
<td>3,10</td>
<td>1,05</td>
</tr>
<tr>
<td>Beer (EtOH 5%)</td>
<td>91,3</td>
<td>---</td>
<td>3,7</td>
<td>---</td>
<td>---</td>
<td>3,90</td>
<td>1,01</td>
</tr>
<tr>
<td>Wine (EtOH 11.5%)</td>
<td>86,7</td>
<td>---</td>
<td>1,8</td>
<td>---</td>
<td>---</td>
<td>3,25</td>
<td>1,00</td>
</tr>
<tr>
<td>Milk</td>
<td>88,5</td>
<td>3,5</td>
<td>11,5</td>
<td>4,7</td>
<td>3,3</td>
<td>5,35</td>
<td>1,03</td>
</tr>
</tbody>
</table>
### Data of fatty foodstuffs (I)

<table>
<thead>
<tr>
<th>Food</th>
<th>Water (%)</th>
<th>Fat (%)</th>
<th>Protein (%)</th>
<th>CH (%)</th>
<th>pH</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Margarine</td>
<td>19,3</td>
<td>80</td>
<td>0,2</td>
<td>0,3</td>
<td>4,20</td>
<td>0,88</td>
</tr>
<tr>
<td>Mayonnaise</td>
<td>16,8</td>
<td>82</td>
<td>0,9</td>
<td>0,3</td>
<td>3,60</td>
<td>0,86</td>
</tr>
<tr>
<td>Gouda</td>
<td>48,0</td>
<td>29</td>
<td>21</td>
<td>2</td>
<td>5,70</td>
<td>1,11</td>
</tr>
<tr>
<td>Cheese Philadelphia</td>
<td>61,8</td>
<td>28</td>
<td>6,5</td>
<td>3,1</td>
<td>5,00</td>
<td>1,02</td>
</tr>
<tr>
<td>Cottage cheese</td>
<td>82,0</td>
<td>2,2</td>
<td>12,7</td>
<td>3,1</td>
<td>4,30</td>
<td>1,06</td>
</tr>
<tr>
<td>Cheese sauce</td>
<td>62,4</td>
<td>19</td>
<td>9,5</td>
<td>7,9</td>
<td>5,00</td>
<td>1,14</td>
</tr>
</tbody>
</table>

### Data of fatty foodstuffs (II)

<table>
<thead>
<tr>
<th>Food</th>
<th>Water (%)</th>
<th>Fat (%)</th>
<th>Protein (%)</th>
<th>CH (%)</th>
<th>pH</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whipping cream</td>
<td>60,0</td>
<td>32,0</td>
<td>8,0</td>
<td>---</td>
<td>5,50</td>
<td>1,00</td>
</tr>
<tr>
<td>Condensed milk</td>
<td>28,6</td>
<td>9,0</td>
<td>8,2</td>
<td>54,2</td>
<td>5,55</td>
<td>1,29</td>
</tr>
<tr>
<td>Yoghurt drink</td>
<td>83,6</td>
<td>0,9</td>
<td>2,7</td>
<td>12,8</td>
<td>3,70</td>
<td>1,06</td>
</tr>
<tr>
<td>Chocolate dark</td>
<td>1,8</td>
<td>31,3</td>
<td>2,7</td>
<td>53,1</td>
<td>---</td>
<td>1,34</td>
</tr>
<tr>
<td>Chocolate spread</td>
<td>2,0</td>
<td>30,0</td>
<td>7,0</td>
<td>59,0</td>
<td>6,20</td>
<td>1,32</td>
</tr>
</tbody>
</table>
Data of fatty foodstuffs (III)

<table>
<thead>
<tr>
<th>Food</th>
<th>Water (%)</th>
<th>Fat (%)</th>
<th>Protein (%)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pork meat</td>
<td>65 - 72</td>
<td>1,4 - 5</td>
<td>17,5 – 21,6</td>
<td>5,80</td>
</tr>
<tr>
<td>Chicken meat</td>
<td>70 - 74</td>
<td>~ 6</td>
<td>~ 22</td>
<td>6,30</td>
</tr>
<tr>
<td>(breast)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Salmon</td>
<td>59 - 66</td>
<td>~ 14</td>
<td>~ 20</td>
<td>6,60</td>
</tr>
</tbody>
</table>

Pork meat: Correlation between fat content and density
Data of dry foodstuffs

<table>
<thead>
<tr>
<th>Food</th>
<th>Water (%)</th>
<th>Fat (%)</th>
<th>Protein (%)</th>
<th>CH (%)</th>
<th>Density &quot;Schüttgewicht&quot;</th>
<th>Med. particle diameter (µm)</th>
<th>Mean spec. surface area (cm²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Milk powder</td>
<td>3.0</td>
<td>26.0</td>
<td>26.0</td>
<td>39.0</td>
<td>0.48</td>
<td>125</td>
<td>13</td>
</tr>
<tr>
<td>Wheat flour</td>
<td>14.0</td>
<td>1.0</td>
<td>10.6</td>
<td>74.0</td>
<td>0.60</td>
<td>75</td>
<td>22</td>
</tr>
<tr>
<td>Butter toast</td>
<td>44.0</td>
<td>4.0</td>
<td>4.0</td>
<td>47.0</td>
<td>0.23</td>
<td>***</td>
<td>***</td>
</tr>
<tr>
<td>Rice</td>
<td>12.0</td>
<td>1.4</td>
<td>6.8</td>
<td>79.8</td>
<td>0.84</td>
<td>5,1-5,7 mm (76 %)</td>
<td>11</td>
</tr>
<tr>
<td>Honey</td>
<td>17.0</td>
<td>---</td>
<td>3.0 (incl. ash etc.)</td>
<td>80.0</td>
<td>1.44</td>
<td>***</td>
<td>***</td>
</tr>
</tbody>
</table>

Milk powder – particle size

Sieve Analysis (Air Jet Sieve, 200 LS-N, Hosokawa Alpine AG, Augsburg, Germany)
Special structures and matrices of some foodstuffs

- Orange juice
- Milk
- Cheese
- Other milk products
- Powders

Orange juice and other foodstuffs containing fibres

- Adsorption effects of pectin and other unsoluble substances
  ⇒ Higher migration values also of lipophilic substances than expected
Apple sauce and tomato ketchup

- **Apple sauce:**
  - Lower water content and higher carbohydrate content than the unsweetened juices.
  - Low pH value due ⇒ aggressive towards the packaging.
  - Viscosity of this suspension is considerably higher than that of juices.
- **Tomato ketchup:**
  - Representing important group of processed vegetable products.
  - Semi fluid food showing plastic behaviour.
  - High viscosity (suspension).
  - Lower water content, due to low pH value highly aggressive, packaged in considerable amounts in plastic packaging.

---

**Milk**

[Electron micrograph of homogenized milkfat globules](http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm)

*From: [http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm](http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm)*
Milk powder

Dents in the large particle have been caused by small hard dry particles

A small particle (surrounded by a 'crater' rim) has been captured by a large particle.

Pictures from: http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm

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Difference of milk powders depending on drying process

Roller-dried milk particles are produced as fragments of a dried milk sheet.

Instantization results in agglomeration of powder particles

Pictures from: http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm

Influence of cheese production on structure of milk particles

Junctions in stirred curd cheeses (e.g. Gouda)

Pictures from: http://www.foodsci.uoguelph.ca
Cream (soft) cheese

Microstructure of traditional Cream cheese Fat globule clusters (dark yellow) are covered with protein (dark blue) in the aqueous medium (light yellow).

Microstructure of Cream cheese made from high-fat cream and acid-coagulated hot milk Fat globules (brown), curd (which shows the core-and-shell ultrastructure of casein particles - dark blue), aqueous phase (yellow).

Picture from: http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm

„Cottage“ cheese

Distinct casein micell clusters; green: bacteria

Picture from: http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm
Structure of yoghurt depending on content of milk solids

- 10% milk solids content
- 15% milk solids content
- 20% milk solids content

Pictures from: http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm

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27.9.06

Casein micelle chains in yoghurt and micelle aggregates in cheese

Yoghurt

Cheese

Pictures from: http://distans.livstek.lth.se:2080/microscopy/foodmicr.htm

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27.9.06
Questions

- Is there a correlation between the migrational key parameters *diffusion coefficients and partition coefficients* of the selected model migrants and food categories specified by physico-chemical properties?
- Have food categories and their corresponding model migrants to be revised caused by measured migrational values?
Thanks for excellent cooperation to

- Peter Volansky
- Herbert Hafner
- Catherine Simoneau (WP 2 and 7)
- All project partners
- Coordinator Roland Franz and his team

Thank you for your attention!
Development of tailored methods for migrants in foods

P. Paseiro, USC, ES
Development of tailored analytical methods for migrants in foods

Perfecto Paseiro Losada
Dpto. Analytical Chemistry, Nutrition and Bromatology
Faculty of Farmacy
University of Santiago de Compostela
SPAIN

WP 3a: Objectives

• To develop analytical methods for the quantitative determination of model migrants in the selected foodstuffs.

• To establish the necessary analytical and experimental basis.

For:

– To carry out kinetic migration studies from plastic films into foodstuffs.
– To study migrant concentration profiles in foodstuffs.
Methodology

1. Compilation, classification and evaluation of the interesting analytical information of the selected substances.

2. Design and experimental development of analytical methods in foodstuffs.

1. Compilation, classification and evaluation

- Study of physical and chemical properties: bp, Log Kow, solubility, spectroscopic properties, derivatization reactions, etc.
- Review of current and available analytical methods.
- Discussion and evaluation of possible and more successful measurement techniques.
2. Design and experimental development (1/2)

1. Selection of model analytical matrices.
   - Orange juice (containing pulp, not filtered).
   - Minced turkey or Chicken breast.
   - Gouda cheese (near to 30% fat).

2. Preliminary trials.
   - Complicated analytical stage.
   - Optimization of the analytical method.
     • Sample preparation, purification and extraction steps.
     • Conditions for the measurement analytical techniques.
     • Evaluation of interferences from the food matrix.

2. Design and experimental development (2/2)

3. Validation.
   - Linearity.
   - Accuracy.
   - Precision.
   - Detection limit.
Deliverable D2: “Compilation of analytical methods for model migrants in foodstuffs”

- Introduction and general considerations.
- Collection of method descriptions in CEN format.
  - Including analytical protocols to determine 18 model migrants (Irganox 1076, DPBD, Chimassorb 81, Uvitex OB, Caprolactam, Benzophenone, DPhP, DEHA, Styrene, Bisphenol A, Octene, Limonene, DIPN, Laurolactam, Triacetin, ATBC, BHT and Triclosan) in selected model foods (Orange juice, minced turkey or chicken breast and Gouda cheese).
  - Methods are applicable to other foodstuffs.
The involved people

FABES: R. Brandsch and A. Zülch.
PIRA: I. Cooper and H. Robin.
TUV I FCT: I. Steiner and P. Volansky.
CEFIC-FCA: K. Hinrichs and C. Jassogne.

Thank you
Systematic migration studies in foods

R. Franz, IVV, D
Systematic Migration Studies into Foods

Roland Franz, on behalf of project partners in work packages 3b and 3c,

Project structure

WP 1: Sel. of Plastics & Migration

WP 2: Selection of Food

WP 3: Physic.-Chemical Parameters
   3a: Analytical methods in food
   3b: Migration kinetics in foods
   3c: Concentration profiles in foods

WP 4: Phys.-Chem. Food Specifications

WP 5: Development, Verification & Validation of the Migration Model

WP 6: Workability, Applicability & Validation of the Migration Model

WP 7: Investigation of Consumer Attitude towards Migration Modelling
Project structure

WP 3: Physic.-Chemical Parameters
3a: Analytical methods in food
3b: Migration kinetics in foods
3c: Concentration profiles in foods

Validation of the Migration Model

WP 6: Workability, Applicability & Validation of the Migration Model

Target parameters for migration

Migration $M$ from a polymer into food is mainly a function of:
- initial migrant concentration in the polymer, $C_{P,0}$,
- mobility of migrant in the polymer, i.e. its diffusion coefficient $D_P$,
- transfer of migrant from polymer surface into and its mobility in the food, both could be summarized as effective diffusion coefficient $D_F$,
- partition coefficient between polymer and food, $K_{P/F}$.

$M = f (C_{P,0}, D_P, D_F, K_{P/F})$
Target parameters for migration

3b: Migration kinetics

Migration from the same polymer sample into 2 different foods under the same conditions

Food 1: $K_{P,F} = 1$

Food 2: $K_{P,F} = 1000$

Migration

Food 1: $D_F$ is higher

Food 2: $D_F$ is lower

time
3b: Migration kinetics

- Food 1: $D_r$ is higher, $K_{P,F}$ ok
- Food 2: $D_r$ is much lower, $K_{P,F}$ ?

Migration from the same polymer sample into 2 different foods under the same conditions.

3c: Concentration profiles

- Food 1: $D_r$ is higher, $K_{P,F} \approx K_{P,F}$
- Food 2: $D_r$ is lower

Migration from the same polymer sample into 2 different foods under the same conditions.
Target parameters for migration

3c: Concentration profiles

- **Food 1**: $K_{PF}$ is higher
  - $D_P = D_F$
- **Food 2**: $K_{PF}$ is lower

Migration from the same polymer sample into 2 different foods under the same conditions
### Overview kinetic migration experiments

<table>
<thead>
<tr>
<th>Food source</th>
<th>Temp (°C)</th>
<th>Times</th>
<th>Test conditions</th>
<th>Test films no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orange juice</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Apple juice</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Milk, 0.5% lactose</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Tomato fleshy</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Tomato luscious</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Tomato paste</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Tomato skin</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Plum</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Pomegranate, crimson</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Pomegranate, yellow</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Pomegranate, red</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Nuts</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Fruits</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Vegetables</td>
<td>19</td>
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<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Cabbage, white</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Cabbage, red</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Broccoli</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Lettuce</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Tomato</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Cabbage</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Spinach</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Strawberries</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Wholemeal</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Bread</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Cereal</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Themmeholz</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
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<tr>
<td>Crackers</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Chocolate</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Yogurt</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Cheese</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Fish</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
<tr>
<td>Fruits</td>
<td>19</td>
<td>1, 4, 10, 16, 22, 28</td>
<td>1, 4, 10, 22, 28</td>
<td>x, x, x, x</td>
</tr>
</tbody>
</table>

- 5 reference films with
- 7 model substances in contact with
- 32 different foodstuffs
- mostly as single side exposure at
  - T = 5°C, 20°C, 25°C, 40°C, 70°C
- 235 kinetic curves
 migration kinetics - examples

Migration of diphenyl butadiene from LDPE into pork meat mixed with different fractions of lard

Migration of caprolactam from PA6 into pork meat mixed with different fractions of lard
Migration modelling of caprolactam from PA6 into pork meat mixed with different fractions of lard.

Overview conc. profile experiments

- 10 model substances, incorporated
- in LDPE films or PE wax (as high diffusivity release systems) in contact
- with 27 different foodstuffs using
- single side exposure at conditions
- \( T = 5^\circ C \text{ to } 70^\circ C \) and \( t = 1 \text{ to } 30 \text{ days} \)
- 175 conc. profiles
### Overview conc. profile experiments

**For example: schedule for Chimasorb 81 (in LDPE)**

<table>
<thead>
<tr>
<th>Foodstuffs</th>
<th>Temp (°C)</th>
<th>Time (days)</th>
<th>Chimasorb 81</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cheese Gouda</td>
<td>5</td>
<td>30</td>
<td>done</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>8</td>
<td>done</td>
</tr>
<tr>
<td>Soft cheese</td>
<td>5</td>
<td>20</td>
<td>done</td>
</tr>
<tr>
<td>Chocolate spread</td>
<td>20</td>
<td>5</td>
<td>done</td>
</tr>
<tr>
<td>Margarine</td>
<td>5</td>
<td>30</td>
<td>done</td>
</tr>
<tr>
<td>Mayonnaise</td>
<td>6</td>
<td>30</td>
<td>done</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>20</td>
<td>done</td>
</tr>
<tr>
<td>Pork neck</td>
<td>5</td>
<td>5</td>
<td>conc.</td>
</tr>
<tr>
<td>Fish (salmon)</td>
<td>6</td>
<td>10</td>
<td>done</td>
</tr>
<tr>
<td>Milk powder</td>
<td>40</td>
<td>10</td>
<td>done</td>
</tr>
<tr>
<td>Wheat flour</td>
<td>40</td>
<td>10</td>
<td>done</td>
</tr>
<tr>
<td>Bacon</td>
<td>5</td>
<td>10</td>
<td>done</td>
</tr>
<tr>
<td>Roasted chicken</td>
<td>5</td>
<td>10</td>
<td>done</td>
</tr>
<tr>
<td>Mature cheddar</td>
<td>20</td>
<td>10</td>
<td>done</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>20</td>
<td>done</td>
</tr>
<tr>
<td>Salmi 30% 1st</td>
<td>5</td>
<td>10</td>
<td>done</td>
</tr>
</tbody>
</table>

### conc. profiles - examples

**Migration of Chimasorb 81 from HDPE into chocolate spread after 20 days at 5°C**

- **Experiment 1**
- **Experiment 2**

---

**FOODMIGROSURE**

Final conference
Migration of Chimasorb 81 from HDPE into cooked chicken (sliced) after 10 days at 5 °C

conc. profiles - examples

Migration modelling of Chimasorb 81 from HDPE into cooked chicken after 10 days at 5 °C

Mean Concentration, \( <C_{PA}> \) (mg/kg) vs. Depth in Food, \( x_p \) (mm)
Conclusion

An extensive and unique collection of data sets were elaborated within WPs 3b & 3c for into-food migration modelling in WP5. This was only possible by the excellent cooperation of all partners and due to the incentiveness and imagination of partners with respect to the experimental design in measuring migration kinetics and concentration profiles.
Development, verification and validation of migration model for foods

R. Brandsch, FABES
Aim

Work package 5

► to establish on the basis of the experimental data set (from work package 3) an advanced migration model for the migration of low molecular weight components from plastic materials into foodstuff

► by adapting the validated diffusion model that exists for food simulants.
### Compliance tools

#### FCM

<table>
<thead>
<tr>
<th>Year</th>
<th>Regulation</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>1982</td>
<td>(82/711/EEC)</td>
<td>experimental testing under conventional test conditions (time/temperature &amp; simulants)</td>
</tr>
<tr>
<td>1985</td>
<td>(85/572/EEC)</td>
<td>substitute fat tests (ethanol 95%, iso-octane &amp; Tenax)</td>
</tr>
<tr>
<td>1997</td>
<td>(97/48/EEC)</td>
<td>migration modelling for plastics in contact with food simulants (diffusion models based on scientific evidence)</td>
</tr>
</tbody>
</table>

- overall migration
- specific migration
  - authorized substances from positive lists

#### Complience tools

<table>
<thead>
<tr>
<th>Year</th>
<th>Regulation</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>2004</td>
<td>(EC 1935/2004)</td>
<td>declaration of compliance (record-keeping of supporting documents)</td>
</tr>
<tr>
<td>future (Active &amp; Intelligent Packaging)</td>
<td></td>
<td>authorisation granted to the applicant (authorisation limited to application)</td>
</tr>
<tr>
<td>future (4. Amendment to 2002/72/EC)</td>
<td></td>
<td>functional barrier concept (maximum migration level of 0.01 mg/kg food for &quot;permitted substances&quot;)</td>
</tr>
</tbody>
</table>
### Compliance tools

<table>
<thead>
<tr>
<th>FCM</th>
<th>Future (4. Amendment to 2002/72/EC)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fat consumption reduction factor, FRF (fatty foods and lipophilic substances)</td>
</tr>
<tr>
<td></td>
<td>Ethanol 50% - new food simulant (for milk and milk products)</td>
</tr>
</tbody>
</table>

- Overall migration
- Specific migration (authorized substances from positive lists) (permitted substances migration below 0.01 mg/kg food)

### Compliance tools

<table>
<thead>
<tr>
<th>FCM</th>
<th>Future</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Estimation of consumer exposure from food contact materials</td>
</tr>
</tbody>
</table>

\[
\text{Exp} \left[ \frac{\text{mg}}{\text{kg}(\text{bw}) \cdot \text{day}} \right] = \sum_{\text{Food}} \frac{\text{kg}}{\text{day}} \cdot \text{SM} \left[ \frac{\text{mg}}{\text{kg}} \right] \cdot \text{body weight} \left[ \frac{\text{kg}}{} \right]
\]

- Specific migration (SM) is one of the key inputs
- To be estimated by modeling into foods
Migration Model

Steps to carry out a simulation:
► Define your objective and measures of performance
► Develop the conceptual model
► Create the mathematical model
► Quantify the input parameters
► Implement and solve the mathematical model using a computational tool
► Evaluate, explain and present the results

Migration Model

Objective and measures of performance

- objective (question to be answered):
  - estimation of specific migration from plastic food contact materials into food.
- measure of performance (model output):
  - specific migration from plastic food contact materials in food.
**Migration Model**

**Develop the conceptual model**
- conceptual model (current understanding of the system):
  - representation of significant features, events and processes.
  
  ⇒ - initial content of migrant
  - volume and contact area
  - time and temperature
  - diffusion process
  - partitioning
  - boundary conditions

**Migration Model**

**Create the mathematical model**
- mathematical model (represents the system quantitatively):
  - set of input assumptions, equations and algorithms describing the system.

**Diffusion equation (Fick's 2nd law):**
\[
\frac{\partial c}{\partial t} = D \cdot \frac{\partial^2 c}{\partial x^2}
\]
- \( c \) - concentration
- \( t \) - time
- \( x \) - space
- \( D \) - diffusion coefficient
### Migration Model

Create the mathematical model

#### Set of input assumptions:

- **Polymeric material**
  - homogenous on macroscopic scale
- **Migrant**
  - homogenously distributed in polymer
- **Interface**
  - no boundary resistance
- **Interaction**
  - no swelling of polymer
- **Reaction**
  - no loss of migrant
- **Diffusion coefficient**
  - remains constant over time

### Migration Model

Quantify the input parameters

- input parameters
  - to be quantified by specifying their values.

**Diffusion coefficient, \( D_p \):**

\[
D_p = D_0 \cdot e^{-\frac{E_A}{RT}}
\]

**Partition coefficient, \( K_{P,F} \):**

\[
K_{P,F} = \frac{C_{P,\infty}}{C_{F,\infty}}
\]

- \( D \): diffusion coefficient [cm²/s]
- \( D_0 \): pre-exponential factor
- \( E_A \): activation energy [J]
- \( R \): gas constant [8,314 J/molK]
- \( T \): temperature [K]
- \( K \): partition coefficient
- \( c \): concentration [mg/kg]
**Migration Model**

**Implement & solve the mathematical model**

- solve the mathematical model:
  - use a computational tool capable of solving the equations representing the system.

Discretisation of space:

\[
\frac{c_i^{n+1} - c_i^n}{\Delta t} = D \left[ \theta \left( \frac{c_i^{n+1} - 2c_{i-1}^{n+1} + c_{i-2}^{n+1}}{(\Delta x)^2} + (1 - \theta) \frac{c_i^n - 2c_{i-1}^n + c_{i-2}^n}{(\Delta x)^2} \right) \right]
\]

Discretisation of time:

\[
\frac{\partial^2 c_i}{\partial x^2} = \frac{\epsilon c_{i-1} - (1 + \epsilon)c_i + c_{i+1}}{(\Delta x)^2}
\]

**Migration Model**

**Evaluate, explain and represent the results**

- results:
  - migration of substances from plastic food contact materials into food under any conditions of use
**Migration Model**

Diffusion model used for migration modeling

\[ D/K/.../D \]

- \( D/K \) - polymer/simulant
- \( D/K/D \) - polymer/food
- \( D/K/D/K \) - polymer1/polymer2/simulant
- \( D/K/D/K/D \) - polymer1/polymer2/food
- \( D/D \) - polymer1/polymer1

**Fundamental constants**

Evaluation of experimental data

- Fitting of experimental data points with calculated migration curves under variation of the partition coefficients between polyolefine and food and diffusion coefficient in food.
**Fundamental constants**

Evaluation of experimental data

- fitting of experimental data points with calculated migration curves under variation of the partition coefficients between polyleine and food and diffusion coefficient in food.

### Concentration profiles

![Concentration profiles](image)

### Fundamental constants

Evaluation of experimental data (Gouda cheese)

<table>
<thead>
<tr>
<th>Gouda cheese</th>
<th>18°C</th>
<th>30°C</th>
<th>40°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low wax PE</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EVA</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>LDPE</td>
<td></td>
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<tr>
<td>LDPE-OPE</td>
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<tr>
<td>LDPE-OPE</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

Time dependent kinetic migration experiments

Concentration profiles

![Time dependent kinetic migration experiments](image)
**Fundamental constants**

**Partition coefficients (Gouda cheese)**

\[ K_{P,F} = \frac{C_{P,\infty}}{C_{F,\infty}} \]

**Diffusion coefficients (Gouda cheese)**

\[ D = D_0 \cdot e^{\frac{-E_A}{RT}} \]
**Fundamental constants**

**Food groups**
- Food groups based on migrant uptake properties (partitioning):
  - Liquid foods
  - Processed fruits and vegetables
  - Dry foods
  - Milk products
  - Meat products
  - Cheese products
  - Margarine/Mayonnaise
  - Chocolate products
  - Special cases (e.g. honey, etc.)

---

**Fundamental constants**

"Upper-level" partition coefficients (cheese products)
- "Upper level" partition coefficients between polyolefines and cheese products in dependence of the octanol/water partition coefficients log $K_{OW}$ (polarity of migrants):

![Chart showing partition coefficients](attachment:image.png)
Fundamental constants

"Upper-level" diffusion coefficients (cheese products)

\[ D = D_0 \cdot e^{\frac{-E_A}{RT}} \]
\[ D_0 = e^{A_F} \]
\[ A_F = \ln D + \frac{E_A}{RT} \]

"Upper limit" diffusion coefficients \( D_p^\ast \) through "upper limit" food specific constants, \( A_F^\ast \) in cheese products:

\[ \text{Safety Limit: } A_F = -1.8 \]

Average \( E_A = 30 \text{ kJ/mol} \)

Migration translation tool

Migration translation tool:

\[ D_p / K_{P,F} \text{ (simulants)} \]
\[ D_p / K_{P,F} / D_F \text{ (food)} \]

⇒ translation of migration data with food simulants under conventional test conditions, e.g. 10 days at 40°C into food migration values under real conditions of use, e.g. 180 days at 5°C.
Migration translation tool

**case A:** Initial concentration of migrant in FCM $c_{P,0}$ and diffusion coefficient of migrant in FCM $D_P$ and partition coefficient between FCM and food simulant $K_{P,S}$ is known

⇒ substitute $K_{P,S}$ with $K_{P,F}$ and calculate migration into food with MMF

**case B:** one of $c_{P,0}$, $D_P$, $K_{P,S}$ unknown

⇒ derive unknown value from migration data with food simulant by applying MMS

⇒ substitute $K_{P,F}$ with $K_{P,F}$ and calculate migration into food with MMF

**case C:** two of $c_{P,0}$, $D_P$, $K_{P,S}$ unknown

⇒ make reasonable assumptions concerning one of the unknown values

⇒ derive second unknown value from migration data with food simulant by applying MMS

⇒ substitute $K_{P,F}$ with $K_{P,F}$ and calculate migration into food by MMF

---

Migration translation tool - case A

Example (PA6/Caprolactam)

$D_P=7.075 \times 10^{-10}$ ($A_P=8.2$);
$K_{P,S}=45$

food simulant: well mixed liquid

water
**Migration translation tool - case A**

Example (PA6/Caprolactam)

Comparison between well mixed and semi-solid food (apple sauce)

- **food:** (semi-solid) apple sauce
- **food:** apple sauce

Migration:

\[ D_F = 7.075 \times 10^{-10} \quad (A_P = 8.2); \quad K_{P,F} = 160 \]
\[ D_F = 1.2 \times 10^{-5} \]
### Migration translation tool - case C

#### Example (LDPE/Triclosan)

**Food simulant:** (well mixed liquid) HB 307

#### 1. General Information on Final Article

<table>
<thead>
<tr>
<th>Value</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### 2. General Information on Substance Tested

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### 3. Experimental Migration Data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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---
Migration translation tool - case C

Example (LDPE/Triclosan)

migration soluble in HB 307

\[ K_{P,F} = 1 \text{ - known} \]

(reasonable assumption)

\[ D_P \]

food simulant: (well mixed liquid)

HB 307

Apply MMS:

- initial concentration: 1100 mg/kg
- thickness: 2400 µm
- density (HDPE): 0.95 g/cm³
- area: 0.954 dm²
- volume: 100 ml
- density (HAc 3%): 1 g/cm³

\[ D_P = 4.8 \times 10^{-10} \]

\[ K = 1 \]
**Migration translation tool - case C**

**HDPE**

- Apply MMF:
  - initial concentration: 1100 mg/kg
  - thickness: 2400 µm
  - density (HDPE): 0.95 g/cm³
  - area: 0.954 dm²
  - volume: 100 ml
  - density (gouda cheese): 1 g/cm³

**Migration translation tool - case C**

- Calculation of Triclosan migration after 150 days at 5°C into gouda cheese by use of MMF:
  - \( m_{F,t} = 0.8 \text{ mg/dm}^2 \)

- According to directive 85/572/EEC:
  - Reduction factor for HP307 migration at 40°C: \( X = 3 \)
  - \( m_{F,t} = 2.37 \text{ mg/dm}^2 : 3 = 0.79 \text{ mg/dm}^2 \)
Validation

"Upper-level" diffusion- and partition coefficients

Diffusion model for plastic materials in contact with food:

\[ D_P^*/K_{P,F}^*/D_F^* \]

\[ \Rightarrow \text{overestimation of migration for compliance purpose under real conditions of use} \]

Validation

Probability distributions of diffusion- and partition coefficients

Exposure estimation for plastic materials in contact with food:

\[ D_P/K_{P,F}/D_F \]

\[ \Rightarrow \text{probabilistic estimation of migration for exposure estimation purpose} \]
Conclusions & Outlook

- Diffusion properties of plastics (\(D_P\) based on \(A_p\) and molecular mass)
- Food groups according to partitioning (\(K_{P,F}\) based on log \(K_{OW}\))
- Diffusion like behavior in food (\(D_F\) based on \(A_F\))
- Migration modeling in food (upper limit values, \(D_P^* / K_{P,F}^* / D_F^*\))
- Migration translation tool (best values \(D_P / K_{P,S} + D_P / K_{P,F} / D_F\))
- Exposure Estimation (probabilistic values - \(D_P^* / K_{P,F} / D_F^*\))
Relation to exposure assessment

L. Castle, CSL, UK
Foodmigrosure - Relationship to exposure assessment

Work package 6a

Laurence Castle & Emma Bradley
Central Science Laboratory
York

WP 6a - objectives

1. To provide a demonstration of the workability and reliability of the modelling approach, by comparing it with estimates of exposure made by other, independent, routes.

2. To link the project with exposure research groups and investigate the feasibility of making the migration model compatible and linkable with computer programs established there.
Estimating consumer exposure

For a single food item:

\[ Exposure = Weight \times Concentration \]

For whole diet, add up the food items:

\[ Exposure = \sum_{\text{first item}}^{\text{last item}} Weight \times Concentration \]

Need data on diets and concentrations

Data needed for estimating exposure

a) food consumption data, describing the types and amounts of food and beverages consumed by individuals of the population of interest;

b) market share data, describing the materials that these food items are packaged in;

c) migrant concentration data, for the substance of interest, for these food-package combinations or for food simulants under the relevant conditions.
Meeting objective 1

TA says: “The utility of the modelling approach will be tested using at least 5 substances for which suitable and representative migration and exposure information is available to a greater or lesser extent”

- A step-wise approach was used for 9 substances, chosen to cover a variety of technological roles.

Variability between individuals

Calculate exposure for every person in survey

\[
Exposure = \sum_{\text{first item}}^{\text{last item}} \text{Weight} \times \text{Concentration}
\]
Testing the exposure estimates

- The technical annex required that the validity of the developed migration model, when coupled with estimated food chemical intakes, should be tested:
  - A = estimated intake (in mg / person / day)
  - B = ‘true’ intake
  - C = present ‘worst-case’ exposure assessments using common assumptions made by the Commission and EFSA

For validation the criteria is that: \( B \leq A < C \)

- That is, the estimated intake should be no less than the real intake (and so be conservative) but should be less than any unrealistic ‘worst-case’ assumptions made presently in exposure assessments performed by the Commission and EFSA.
Data needed for estimating exposure

a) food consumption data, describing the types and amounts of food and beverages consumed by individuals of the population of interest;

b) market share data, describing the materials that these food items are packaged in;

c) migrant concentration data, for the substance of interest, for these food-package combinations or for food simulants under the relevant conditions.

Data providers within this project

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
<th>Provider</th>
</tr>
</thead>
<tbody>
<tr>
<td>FIS</td>
<td>food intake statistics</td>
<td>CSL</td>
</tr>
<tr>
<td>MIS</td>
<td>modelled intake statistics (i.e. 1 kg food / person / day; 600 cm² packaging / person / day)</td>
<td>CSL</td>
</tr>
<tr>
<td>ACD</td>
<td>actual food concentration data</td>
<td>CSL</td>
</tr>
<tr>
<td>MCD</td>
<td>modelled food concentration data</td>
<td>FABES</td>
</tr>
<tr>
<td>SMD</td>
<td>simulant migration data (i.e. the worst-case concentration for simulants A, B, C, D/X).</td>
<td>CSL</td>
</tr>
</tbody>
</table>
Data sheets and step-wise exposure estimates were prepared for these 9 substances

<table>
<thead>
<tr>
<th>Substance</th>
<th>Technological role(s)</th>
<th>Main FCM application(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Di(2-ethylhexyl) adipate</td>
<td>Plasticiser, carrier solvent</td>
<td>Polyvinyl chloride (PVC)</td>
</tr>
<tr>
<td>Epoxidised soybean oil</td>
<td>Stabiliser and plasticiser</td>
<td>Polyvinyl chloride (PVC)</td>
</tr>
<tr>
<td>Irgafos 168</td>
<td>Stabiliser (antioxidant)</td>
<td>Polylefins</td>
</tr>
<tr>
<td>Styrene</td>
<td>Monomer</td>
<td>Polystyrenes</td>
</tr>
<tr>
<td>Caprolactam</td>
<td>Monomer</td>
<td>Nylon</td>
</tr>
<tr>
<td>Antimony</td>
<td>Polymerisation catalyst</td>
<td>Polylethleneterephthalate</td>
</tr>
<tr>
<td>AA quencher</td>
<td>Additive (AA scavenger)</td>
<td>Polylethleneterephthalate</td>
</tr>
<tr>
<td>PET cyclic trimer</td>
<td>Oligomer</td>
<td>Polylethleneterephthalate</td>
</tr>
<tr>
<td>Bisphenol A</td>
<td>Monomer</td>
<td>Polycarbonate, epoxyphenolic can coatings</td>
</tr>
</tbody>
</table>

Step-wise exposure scenarios used for the 9 test cases

- Migration = total migration of all of the substance
- Migration = total migration from a surface layer only (e.g. 250 um)
- Simulant data
- Simulant data & average packaging market share (e.g. USA PUFs)
- Estimated exposure from the diet (deterministic, food item consumption and food analysis results)
- Estimated exposure from the diet (probabilistic, food consumption and food analysis results)
- Estimated exposure from a biomarker study
**FIS – food intake statistics**

The CSL application utilises the UK National Dietary and Nutrition Surveys.


Migrants from food cans revisited - application of a stochastic model for a more realistic assessment of exposure to bisphenol A diglycidyl ether (BADGE). *Packaging Technology & Science*, 2006, 19, 121-137.

**MIS – modelled intake statistics**

Default parameters ('conventions') used for exposure assessment

For the general case:-

• 1 kg of packaged food eaten every day
• 600 cm² of packaging used each day
• this packaging always contains the substance (monomer, additive etc.)
• the substance always migrates at the highest level permitted (e.g. at the SML)
• no other significant sources of exposure
• a consumer has a body weight of 60 kg
ACD – actual concentration data

In a number of cases, concentration data for foods themselves were used if available

- market basket surveys
- enforcement campaigns

- total diet samples
- duplicate diet samples

SMD – simulant migration data

- More generally, the concentration data used were for simulants based on food classes A, B, C, D, D/X2 etc, rather than for individual food items or food groups.

- In some cases, the concentration data are based on solvent (total) extraction data.
MCD – modelled concentration data

Available from FABES:
Migration rate for:
- a given food type
- a given plastic composition
- a given temperature
- in units of mass / area / time

Conclusion on WP6a Objective 1

The ‘acid test’ is

- how well does the migration modelling fit the concentration data measured in foods?

Note: In WP6a we have concluded that:
Whereas modelling for compliance testing using simulants has been aimed deliberately to overestimate migration in the majority (95+%) of cases;
Modelling for exposure estimates should aim to give the best estimate of migration into foods along with a statement of any uncertainty.
Meeting objective 2

TA says:

➢ “To link the project with exposure research groups and investigate the feasibility of making the migration model compatible and linkable with computer programs established there”.

Available now

A way to use the modelled concentration data in estimating exposure at the level of the individual consumer – e.g. by deterministic or probabilistic modelling

➢ Models under development and testing at CSL, TCD, INRA, RIKILT, TNO, etc. But this cannot just be a one-way (input) process.
The migration model itself needs input information from these exposure models.
Calculate exposure for every person in survey

\[
Exposure = \sum_{\text{first item}}^{\text{last item}} \text{Weight} \times \text{Concentration}
\]

Plot as histogram

Variability between individuals

Modelling the concentration data

Available from Foodmigrosure:
Migration rate for:
- a given food item or food type;
- a given plastic composition;
- a given temperature;
- in units of mass / area / time.
Modelling the concentration data
– Input parameters

Plastic composition?

Initial concentration of the substance in the packaging material(s) at the point of use to pack the food

Temperature?

Contact temperature(s) for the period between packaging by the producer and unwrapping by the consumer
Modelling the concentration data
– Input parameters

Area ?

Surface area of packaging in contact with the portion of food (of known mass) that is consumed

Time ?

Period of time elapsed between packing by the producer and unwrapping by the consumer
Input parameters needed to calculate concentrations in the whole diet

- food types
- plastics composition
- contact temperatures
- contact areas
- migration times

all are distributions

and not fixed points

Ways forward for exploitation

Integrate the estimation of concentration using modelling

- into exposure modelling programmes
  - CSL
  - CREME
WP6a Conclusion

Obj.1. ✓
Tools, understanding and data were provided to allow a verification of the reliability of mathematical migration modelling in estimating consumer exposure

Obj.2 ✓
Made links with existing exposure modelling programmes and pointed the way forward to full integration of mathematical modelling into the exposure assessment process

(end)

Laurence Castle
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l.castle@csl.gov.uk
A risk management view towards the safety of food contact materials

A.Schaefer, European Commission - DG SANCO
Migration into food
EU point of view

Dr. Annette Schäfer

27/09/2006 Foodmigrosure, Baveno

Warning

The content of this lecture does not necessarily represent the position of the European Commission

27/09/2006 Foodmigrosure, Baveno
Migration

- Food
- Contact time
- Migration
- Contact temperature
- Packaging

The Golden Rule


No transfer of constituents into food in concentrations that could
- endanger Human Health
- change in Characteristics of Food
  - Taste
  - Odour
  - Composition
Translation for specific materials

<table>
<thead>
<tr>
<th></th>
<th>RCF</th>
<th>Ceramic</th>
<th>Plastics</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Substances</strong></td>
<td>Authorisation/</td>
<td>Restriction</td>
<td>Authorisation/</td>
</tr>
<tr>
<td></td>
<td>Restriction</td>
<td></td>
<td>Restriction</td>
</tr>
<tr>
<td><strong>Limits</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Residual content</td>
<td>QM</td>
<td>QM</td>
<td>QM</td>
</tr>
<tr>
<td>Migration</td>
<td>SML</td>
<td>SML</td>
<td>OML</td>
</tr>
<tr>
<td>Testing</td>
<td>Simulant</td>
<td>Food Simulant</td>
<td></td>
</tr>
</tbody>
</table>

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Plastics

2002/72/EC

- OML 60 mg/kg **food** or 10 mg/dm²
- SML mg/kg or mg/dm²
- Verification of compliance according to 82/711/EEC and 85/572/EEC

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Plastics

2002/72/EC

- Migration testing
- Residual content below SML
- Residual content and migration modelling below SML (simulants)

82/711/EEC

- LIMITS
  - OML
  - SML
- TEST MEDIA
  - into or onto food
  - into or onto food simulant
Plastics

82/711/EEC

VERIFICATION

- FOOD
  Under most extreme conditions of time and temperature foreseen in actual use
- FOOD SIMULANT
  Conventional testing following basic rules

27/09/2006

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82/711/EEC

<table>
<thead>
<tr>
<th>Simulant</th>
<th>or</th>
<th>or</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Destilled water</td>
<td>equivalent</td>
</tr>
<tr>
<td>B</td>
<td>3 % Acetic acid (w/v)</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>10 % Ethanol (v/v)</td>
<td>Adjusted to higher EtOH content</td>
</tr>
<tr>
<td>D</td>
<td>Rectified Olive oil</td>
<td>Corn oil, Sunflower oil, HB307</td>
</tr>
</tbody>
</table>

27/09/2006

Foodmigrosure, Baveno
Plastics

85/572/EEC

<table>
<thead>
<tr>
<th>Simulant</th>
<th>or</th>
<th>or</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Destilled water</td>
<td>equivalent</td>
</tr>
<tr>
<td>B</td>
<td>3 % Acetic acid (w/v)</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>15 % Ethanol (v/v)</td>
<td>Adjusted to higher EtOH content</td>
</tr>
<tr>
<td>D</td>
<td>Rectified Olive oil (specification)</td>
<td>Sunflower oil, HB307 (specification)</td>
</tr>
</tbody>
</table>

Dry foods and ready to eat food

- 82/711/EEC
- No simulant assigned for dry foods
- Test carried out on foods

- 85/572/EEC
- No simulant assigned for foods like: Cereal, pasta, flour, dried fruit, egg powder, milk powder, deep frozen food, spices
- Packaging
Plastics

Fatty foods
- 82/711/EEC
- Simulant D
- Substitute simulants
- Alternative simulants

Testing conditions for simulants 82/711/EEC

<table>
<thead>
<tr>
<th>Time</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,5 h</td>
<td>5 °C</td>
</tr>
<tr>
<td>1 h</td>
<td>20 °C</td>
</tr>
<tr>
<td>2 h</td>
<td>40 °C</td>
</tr>
<tr>
<td>4 h</td>
<td>70 °C</td>
</tr>
<tr>
<td>24 h</td>
<td>100 °C or reflux</td>
</tr>
<tr>
<td>10 days</td>
<td>121 °C</td>
</tr>
<tr>
<td></td>
<td>130 °C</td>
</tr>
<tr>
<td></td>
<td>150 °C</td>
</tr>
<tr>
<td></td>
<td>175 °C</td>
</tr>
</tbody>
</table>
**Plastics**

Translation into CEN standards

CEN
- EN 1168 Overall migration plastics
- EN 13130 Specific migration plastics
- EN Fatty food contact
- EN 14235 polymeric coating on metal substrate

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**Plastics**

Future Changes 4th amendment 2002/72/EC

- Fat consumption reduction factor (FRF)
- Total consumption factor (TRF = DRF + FRF)
- Simulant for milk in 85/572/EEC
  - Simulant D 50% Ethanol

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Plastics

Future Changes Plastics Regulation

- Compilation of all texts on plastics into 1 text
- Restrictions/specification/migration testing
- Differentiation between legal provisions and provisions in guidelines
- Applicability for other materials such as plastic coated multilayers

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Thank you for your attention

JRC website
http://crl-fcm.jrc.it

EFSA website
http://www.efsa.europa.eu/

SANCO website
http://ec.europa.eu/comm/food/food/chemicalsafety/foodcontact/index_en.htm

27/09/2006  Foodmigrosure, Baveno
Public participation in the project
Consumer attitudes studies to packaging safety, migration and modelling

C. Simoneau, European Commission - DG JRC
Food Safety perception

★ Explosion of projects on risk perception, risk communication (FP5 and FP6)

★ All question methodologies or find methodologies are crucial to results

★ All point to importance of risk communication

★ All warn about approaches are not developed yet
Difficulties unique to foodmigrosure

★ introducing the concept of risk in an area that has never been associated with risk in any preceding studies.
   ➢ not a product being presented (e.g. actipack, organic food) or
   ➢ Not a technological risk (e.g. food irradiation);
   ➢ topic can appear confusing even to risk perception experts, therefore the concepts from other studies are not really applicable and need to be revisited and better understood.

The challenges

★ Packaging not normally associated as source of risk
★ The exact nature of risk is not known
★ The topic is unfamiliar or not perceived
★ It will be perceived as an imposed risk (rather than voluntary, as the individual has no choice)
★ It will be perceived as under government control rather than individual, therefore uncontrollable

★ Education, communication and transparency are CRUCIAL
What can risk from packaging be measured against?

★ microbiological contamination,
★ mycotoxins,
★ pesticides,
★ antibiotics and growth promoters (hormones),
★ Industrial pollution (dioxins, heavy metals),
★ bovine spongiform encephalopathy.

Plan of investigation

★ Need expert advice
  ➢ Expert meeting on risks and studies
★ Need education support
  ➢ Brochure/leaflet
  ➢ Presentation/video
★ Need polling support
  ➢ Focus group
  ➢ Questionnaire
Strategy: 3 approaches

★ Focus group – based on protocols developed in the project TRUST
   ➢ 8 people, balanced group, moderator but no lead
   ➢ Qualitative, more time (2hrs)
   ➢ Natural discussion evolving in points of interest.

★ Quantitative
   ➢ Polling, questionnaire
   ➢ Many people, visual support, poll after communications

★ Stakeholder questionnaires

Approach One: focus group

★ Italy; 4 women 4 men; age 29-75; high school to college
★ Team 2 facilitators, 1 observer and 3 JRC researchers.
★ Laboratory tools and a portion of video showing the laboratory was available
★ 2.5 hours; all participants got immediately involved

★ General comments
   ➢ All participants showed high level of interest
   ➢ Interventions usually appropriate and comments often sophisticated,
     o Ability to capture numerous aspects of the food packaging issue, from research to regulation and controls.
     o Some participants touched upon complex issues such as the scope and limitations of scientific research and the researchers’ ethics, making a case for the importance of humility and recognition of uncertainty.
Focus Group: Most relevant points

★ food selection: quality emerges as a shared key criterion, though somewhat conditioned by time and budget constraints. The idea of quality includes a number of meanings: freshness, taste, naturalness, pleasure, healthiness and safety. Low price is taken as a cue to low quality (and possibly reduced safety).

★ The issue of quality controls is of the utmost importance for participants and keeps coming up in different associations during the discussion. There is widespread awareness of and basic confidence in regulation and controls, both at the national and European level.

★ The great availability of information is appreciated, despite the difficulty in detecting useful and honest messages and advice from manipulative ones.

★ The issue of packaging is raised spontaneously as a matter of concern. Normally, packaging is mentally associated with long shelf life and elaborate processing, while it is rarely spontaneously considered in relation to fresh food.

★ Regarding possible problems related to packaging, the following two are identified: migration and food decay. Whereas the latter is attributed to inappropriate packaging, the former is ascribed to non-compliance with norms or inappropriate use.

★ Participants speculate that industry is less preoccupied of consumers’ safety than its own profit. Accordingly, they are afraid of low compliance with packaging regulations and even fraud.
Focus Group: Most relevant points

★ Overall, participants recognise the benefits of packaging in terms of hygiene and safety, also due to constant improvements in packaging technologies. There is no principled objection to any specific type of packaging, including plastics. “Appropriateness” emerges as an umbrella criterion, including safety as well as visual attractiveness, easy handling, transporting and storing, etc.

★ Some food-packaging combinations are particularly disliked, for a number of different reasons, ranging from safety to visual appearance, from taste to limited choice. Some participants sometimes recognise theirs as idiosyncratic and not rationally grounded. Again, judgement criteria are referred to an encompassing idea of “appropriateness”.

★ Participants are satisfied with the demonstrations and explanations of laboratory testing offered by the JRC researchers. Their satisfaction is grounded in a positive image of chemistry as a highly respected discipline, relying on solid procedures and producing reliable data. Also they appreciate the work documented and the experts’ willingness to dialogue with them.

Focus Group: Most relevant points

★ The main and most frequently expressed preoccupation is that laboratory results are not taken in due account by industry and retailers. Questions are raised and doubts are expressed repeatedly about the connection between problems detected in the laboratory and practical actions implemented to prevent risk for the consumers.

★ Participants seem to grasp the general idea of computer simulation and are satisfied with the illustrations provided.

★ There are no principled objections to the use of simulation in the investigation of food packaging safety. Computer modelling is deemed efficient and useful, provided that input data are of good quality. Some cautionary remarks are raised about simulation being a proxy of the real world and not its exact reproduction. Simulation is appreciated also in so far as its use reduces the need for animal testing. Neither diminished use of chemicals nor any other benefits are mentioned.
Focus Group: Most relevant points

- Although the rationale of the “worst cases” in risk assessment doesn’t seem hard to understand or accept, the idea that non conformity to legally established standards is not equivalent to lack of safety is difficult to grasp and agree to.

- In general, expressions of concerns for health and safety are accompanied by others pointing to the benefits of current packaging technologies. This results in a balanced assessment, combining common sense and notions acquired either during the discussion or previously.

- The discussion shows that there is a largely shared consensus in attributing the main value of research to its applications for the public benefit. The use of computer simulation is neither objected to nor deemed inferior to laboratory testing. In both cases the basic concern is about the ability of techniques and procedures to generate a positive and robust spin-off in terms of consumers’ protection.

Approach 2: Questionnaire

- Also see our posters
- Idea: to take advantage of an Open Day at the JRC on May 13 2006 (about 3000 visitors)
- Conduct a large scale citizen perception study.
- Challenge: goal become 3-fold
  - Packaging = not risk: Need a part on risk education and communication;
  - Open day: exhibiting in simple terms the type of research and the means for doing it that the JRC possesses.
  - Highly focalized nature of the foodmigrosure project we had to test people on their perception of mathematical modelling as a mean to simulate and predict migration from food contact materials.
Approach (cont’d)

- We developed a questionnaire based on the one used by EFSA and questions raised in the EU TRUST project.
- We prepared visual stimuli with:
  - an art piece made of packages
  - posters
  - tour programme (explained our goal within the project)
  - A short movie (12 min) on the general risk issues and tests associated with packaging
  - Quick tour of the laboratories for visual impact where the same scientists were welcoming visitors and answering questions
- Logistics: The tour was of a total of 20 min and about 5 min to compile the questionnaire in a shaded and seated area.
- The questionnaire was given upon exiting and completed questionnaires were rewarded with a small gadget.

Posters
Approach

- A test trial was run on consumer associations with 35 representatives of the Lombardia region.
- The experiment was then conducted on citizens in full scale during the JRC Open Day which was highly publicised regionally.
- The event also involved the presence of the consumer association representatives.
- Questionnaires and comments were collected for 700 units which represented about 1400 visitors to the food contact activities.

Results: the open day
Question: What importance do you give to the characteristics of the packaging when you shop for food?

Keys:
1: not important – 2: little important; 3: important; 4: very important; expressed in number of people.

people considered safety the most important, followed by resistance and convenience as important. Shape and color were not considered important.
On the question of an opinion on the use of modelling as helping tool to investigate safety food packaging, almost 90% people were either favourable or strongly favourable to modelling when they understood what it meant; only 3% were against.

The data also reflected comments obtained by focus groups, which was a completely different approach with no prior risk education.

Some comments showed that people inherently trusted the simulations because they assumed or knew that scientists would have tested the model compared with experimental data.

Ranking “partially favourable” seemed to indicate a lack of certainty on the relation between experimental data and modelling.

On the question of in case of a problem associated with packaging and whom one trusts for information, the answer showed that people would like to receive relevant safety related information primarily from scientists, followed by public authorities and newspaper/TV, followed by consumer associations and last by food producers or supermarkets.
The level of concerns of citizen towards packaging showed that packaging contaminants were perceived much like food additives, whereas higher concerns are still towards microbiological contaminations, antibiotics /hormones in meat, pesticides in fruits and vegetables and new viruses like bird flu.

The answers highlighted that consumers felt that migration modelling was most relevant as a value added tool to point out more quickly worst cases and as support to science.

A source of positive feeling from migration modeling seemed to be the help to faster science communication and consumer (unbiased) reassurance.

The level of concerns of citizen towards packaging showed that packaging contaminants were perceived much like food additives, whereas higher concerns are still towards microbiological contaminations, antibiotics /hormones in meat, pesticides in fruits and vegetables and new viruses like bird flu.
Other remarks

★ People felt much reassured regarding the safety of packaging simply from the fact that they did not previously know that such research and controls existed.

★ Most spontaneous written comments were to have this type of research much more visible at the level of both consumer associations and consumers themselves.

★ People were also extremely enthusiastic and grateful for experiencing an entertaining science short production, and made enthusiastic comments on the humanity and added-value the initiative represented for the consumer’s understanding of science.

Conclusions general on consumer perception

★ The responses were echoing quite interestingly many answers also obtained in the focus group, although a completely different methodology.

★ There is a fundamental trust from the public in the scientists to distinguish and understand safety issues.

★ The consumer wants sincerely to be approached and informed by scientists for this reason and is also ready to favor new approaches such as migration modelling if it can be an additional tool for better consumer protection.

★ However, the consumer needs to be sure that at the root for use are experimental data which demonstrate the applicability of the model.
Approach 3: stakeholder survey

- 25 responses; about 15 countries
- Balance between MS CA, NRL, institutes, industries
- Questions from legislation, use of modelling, practices, confidence, future applications

Thank you for your attention
Implications of the project at the EU level
From migration to exposure : FDA experience

T.Begley, FDA, USA
From Migration to Exposure: FDA experience

Tim Begley
Office of Food Additive Safety
Center for Food Safety and Applied Nutrition
Food and Drug Administration

CONCENTRATION IN THE DAILY DIET

\[ <M> = f_{\text{aqueous and acidic}}(M_t 10\% \text{ Ethanol}) + f_{\text{alcohol}}(M_t 50\% \text{ Ethanol}) + f_{\text{fatty}}(M_t \text{ fatty}) \]

Dietary Concentration = \( <M> \times CF \)
**History**

1977-1983 AD Little Study of Indirect Food Additive Migration

**Polymers Studied:** HDPE, LDPE, PS, IPS, PVC, EVA.

**Migrants (\(^{14}\)C radiolabelled):** BHT, Irganox 1010, Styrene, organo tin stabilizer, di(2-ethyl hexyl) adipate.

**Food simulants:** Water, Water/Ethanols, Heptane, Corn oil, 3% acetic acid.

---

**Foods Tested in 1977-1983 Study**

whole milk, skim milk, orange juice, margarine, whipped topping, pickle juice, mayonnaise, vegetable shortening, dry milk, dry chicken soup, bread, wine, cheese, bologna, white rice, brown sugar, squash, sour cream, yogurt, cottage cheese, vanilla pudding, beef liver, gelatin, apple jelly, chocolate, lean beef, chicken breast, cola, fish, whiskey
History

1988 A.D. Little Study High Temperature Migration Testing of Indirect Additives

Polymers Studied: LDPE, HDPE, PP.

Migrants ($^{14}$C radiolabelled): Irganox 1010, Irganox 1076.

Temperatures up to 130°C.

History 1990 A.D. Little Study on Migration at High Temperature to Food

- Aqueous Foods with No or Low Fat
- Aqueous Foods with Emulsified Fat
- Aqueous Foods with Free Fat
- Water and Corn oil
Conclusions of these Migration studies

- Oil and water tend to represent the extremes of migration for non-polar polymer / non-migrant combinations.
- Polar polymer/migrants not studied (i.e. PET or polyamide).
Current migration/exposure issue:

Migration of perfluorochemicals and fluorochemicals.

Why are we interested in perfluoro chemical migration?

- Perfluoro chemicals found in human serum (Olsen et al. 1999).
- May 2000, 3M Company announce phase out of perfluorooctyl chemistry.
- Elderly in Seatle WA are found to have perfluoro chemicals in their serum (Olsen et al. 2003).
Why are we interested in perfluoro chemical migration?

- PFOA is biopersistent / bioaccumulative
- **Half-life** in human serum is 4.4 years (Butenhoff et al. 2004).
- Potential concerns for carcinogenesis; developmental/reproductive and immunotoxic.
- Many fluorochemicals regulated for food-contact contain PFOA as an impurity and/or have chemical moieties similar to PFOA.

---

Structures of perfluorochemicals found in human serum

- **Perfluorooctanesulfonate** = PFOS
- **Perfluorooctanoic acid** = PFOA
Food package types that contain fluorochemicals

- Polymers
  - cookware
  - tubing
  - gaskets

- Paper
  - microwave popcorn
  - muffin bags
  - french fry bags
  - pizza liners
  - sandwich wrappers

Where are perfluorochemicals for paper regulated?

- In US approximately 15 materials are regulated
- BfR also has a number of perfluorochemicals regulated
Types of perfluorochemicals added to paper

- Perfluoro telomer type
  \( C_6 \quad C_8 \quad C_{10} \quad C_{12} \quad C_{14} \) based

- Polymeric type
Fluorochemical Paper coatings

- Fluorochemical paper concentrations can be up to 0.5% or 5000 mg/kg.
- Temperature range for paper applications -5°C – 200+°C.
- Most perfluoro telomer based paper treatments have molecular weights >1000 but the molecular size is similar to much smaller molecules.

Migration of chemicals with MW > 1000?

\[ \text{F versus H} \]

MW = 1121

MW = 510
Is PFOA in Perfluorochemical Paper Coatings and treated Paper Products?
Migration of PFOA from microwave popcorn bags?

None detected into oil


<table>
<thead>
<tr>
<th>Material</th>
<th>C_{p0}</th>
<th>PFOA (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Perfluoro paper coating</td>
<td></td>
<td>88 - 160</td>
</tr>
<tr>
<td>(not applied)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Popcorn bags</td>
<td></td>
<td>0.3</td>
</tr>
<tr>
<td>Muffin bag</td>
<td></td>
<td>0.5</td>
</tr>
<tr>
<td>Sub Sandwich wrapper</td>
<td></td>
<td>0.5 – 1.2</td>
</tr>
<tr>
<td>Hash brown potato bag</td>
<td></td>
<td>0.9</td>
</tr>
</tbody>
</table>
Test for perfluorochemical migration from commercially produced paper
Test conditions for perfluorochemical migration

- Food simulating liquids/foods at 100°C
- Single-sided contact with paper
- Contact time 15 minutes
- LCMS analysis for perfluorochemical

Typical structures of Perfluoro telomer based paper coatings

A  B  C
Migration results for incidental contact, Coating A

Migration results for incidental contact, Coating B
Problem for exposure estimates

Water and oil are not the extremes!
Migration into Emulsions

- Butter = water-in-oil Emulsion (20% / 80%)
- Non-ionic surfactant (polyoxyethylene sorbitan / oil / water)
- Ionic surfactant (lecithin / oil / water)
Migration of Fluorochemical is Kinetic to food at 40°C

Do Perfluoro telomer Paper Coatings Migrate Under Actual Conditions of Use?
Migration from microwave susceptor into oil

Results for migration of fluorotelomer from popcorn bags

| Concentration in Popcorn Oil before Heating | 1.4 mg/kg | 4 µg/ dm² |
| Concentration in Miglyol after 2 min. microwave heating | 2.1 mg/kg | 7 µg/ dm² |
Migration of perfluorotelomer into microwave popcorn

<table>
<thead>
<tr>
<th>Brand</th>
<th>mg/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1.2</td>
</tr>
<tr>
<td>B</td>
<td>0.8</td>
</tr>
<tr>
<td>C</td>
<td>3.8</td>
</tr>
<tr>
<td>D</td>
<td>1.2</td>
</tr>
<tr>
<td>E</td>
<td>2.1</td>
</tr>
</tbody>
</table>

LCMS analysis of Popcorn for the Migration of Fluorotelomer from Popcorn bag
Conclusions of Migration Tests

- The food simulants water and oil are not the extremes for these fluorochemicals.
- The soy emulsifier (lecithin, 0.05%) can change oil into a potent solvent for migration.
- Fluorotelomers do migrate to food in the mg/kg (ppm) range.

Migration to oil = none

Migration to oil + emulsifier = huge
A EU consumer exposure assessment view

P. Oldring, Exposure Vision Group
WHY IS ESTIMATING EXPOSURE IMPORTANT?

• The current EU approach to estimating exposure to migrants from FCMs over-estimates exposure in many cases but may under-estimate exposure in some cases.

• This could arguably restrict consumer choice by de-selection of certain materials.

• Thus it is in the interests of regulators, authorities and the CONSUMER to have good exposure assessments.

• This would help focus efforts on any areas where there may be a real and not an imaginary risk of an unacceptably high exposure.
HOW COULD AN ESTIMATE OF EXPOSURE BE MADE?

There are different approaches to estimating exposure to migrants from FCMs:

- Deterministic (typically point values)
  - Per capita
  - EU
  - FDA
  - Refined deterministic (could use distributions)
- Probabilistic (stochastic - Monte Carlo)

ALL APPROACHES NEED CONCENTRATION DATA

DATA NEEDED FOR AN EXPOSURE ASSESSMENT

- In order to undertake an estimate of exposure the following data are required
  - Amount of food eaten
  - Type of packaging for each food item consumed
  - Amount of migrant(s) in each foodstuff
- The availability of data and type of estimate required dictate which approach(es) can be used to estimate exposure.
CONCENTRATION DATA

We will concentrate on concentration data for the remainder of this presentation.

- Concentration data can be obtained in three distinct ways:
  - Analysing simulants that are intended to mimic foods
  - Analysing foodstuffs as they would be consumed
  - Mathematical modelling of migration levels into simulants or into foods

CONCENTRATION DATA (Cont.)

- With simulants and modelling it is VITAL to ensure that the use of the plastic with the foodstuff is properly represented i.e.
  - Physical and chemical composition of the foodstuff
  - Time / temperature during all stages of the contact
- It is also necessary to know which fraction of each packaging type is used for particular end uses e.g.
  - Chilled foodstuffs
  - Fatty foodstuffs
  - etc
The FOODMIGROSURE project offers an alternative to analysing simulants or foodstuffs for migrant concentrations in order that realistic estimates of exposure can be made.

In fact modelling may be the only way to represent fully all the different permutations of:
- Packaging composition
- Food type
- Time and temperature conditions of use

All sources of data used for any exposure assessment must be transparent to all.
They must be clearly cited and available to the general public.
There has to be a simple explanation for the non-experts as to how the data were obtained, how they have been used with any assumptions and limitations.
The challenge for mathematical modelling is to explain to the non-expert how they arrive at the numbers.
Note the same applies to probabilistic modelling.
This is because all these data would be too expensive to obtain by detailed chemical analysis of foodstuffs or simulants.

However for the consumer, regulator and food industry to be convinced, it is necessary that no-one mistrusts the ‘black box’ approach.

Full transparency is fundamental to the process of acceptance.

This workshop is a first important step in opening up this approach to scrutiny.
The additives and plastics industry view

C. Gueris, CEFIC-FCA/Plastics Europe
FoodMigrosure, a perspective from a producer and user of additives in FCM’s

- Role of CEFIC-FCA during the 3 years research
- FCA position regarding research on FCM’s
- Positive findings from the conducted research
- A few critical remarks
- A few suggestions
- Conclusions
Role of CEFIC-FCA

- Mainly an observer
- Having a strong interest in the scientific developments related to the safety of FCM’s
- A more scientific understanding of the migration behavior of substances from a FCM to a food simulant or even better food, is essential knowledge to producers of such substances
- Provide samples of additives or FCM’s during the research
- Provide expert information in relation to the FCM’s and market applications, when possible

FCA position on scientific research

- FCA represents approx 70 companies producing, and frequently also using, additives in FCM’s
- FCA strongly supports new scientific developments on FCM’s as defined in the Food Law and the Frame Work Regulation
- FCA as well as many of their members regularly conduct their own scientific investigations (migration studies, toxicological studies, development of data in support of exposure assessments
- FCA, as well as all other associations involved with FCM’s, strongly support research towards exposure based legislation and risk assessment of new substances.
- This research includes the support for a Tiered Threshold of Toxicological Concern (ILSI Europe), as well as applying well established tools to estimate toxicological critical endpoint (QSAR, Topcat, Derek)
- Most essential aspect is the inclusion of the recent scientific research in the legislation on FCM’s as well Note for Guidance on new substances
Positive findings

- Scientific evidence that the already existing migration modeling to food simulants can be extended to the prediction of the concentration in food
- Extensive new data generated on the partitioning to food
- Already existing data on dietary intake (UK, Italy, etc.) and Packaging Use Data, has been used (FDA, M. Palmer)
- A workable mathematical model has been designed
- Results already being used to make adjustments for some of the prescribed food simulants for actual migration testing (milk products), which changes can effectively prevent too high migration to milk, resulting in media covered issues (ITX)

A few critical remarks

- Is sufficient validation created, to have the scientific findings accepted by EFSA and Commission to be included in the applicable Commission documents, as an extension to migration modeling (food simulants) and in support of exposure assessment?
- The migration prediction to food simulants is rather conservative to ensure that no inappropriate estimations are being calculated, as the food simulant is a surrogate for food. Is the model now developed, using the same conservative mathematical model? Many uncertainties seen with food simulants are now over come.
- Can the model be adjusted when PlasticsEurope, FCA, EuPC come with a more detailed packaging use data base? (Matrix > Correlation Factors, instead of FDA)
A few critical remarks

- What is the application area of the model? Food types or applications/conditions where the model cannot be applied?
- e.g. deep frozen food?
- How can the model be used in the current petition concept of new substances?
- Example: Current petitions are based on the highest found migration in food simulants (mostly with simulant D). The market requires that FCM’s can be used for “all food types”. One single migration result above 5 mg/kg leads to tremendous costs/time problems. Mostly the FCM shows lower migration in actual foods, especially aqueous foods.
  - Can, with the new scientific evidence predicting concentrations in food, the toxicological data package be adjusted, to the levels predicted in food?
  - Can the new scientific evidence be used to modify the current food simulants for certain food types, in a way to use less aggressive food simulants?

A few suggestions

- Publication of the FoodMigrosure work in a scientific magazine
- Ensure that the missing modules are being completed, and finalize the report.
- Carefully review and assess the module on “consumer acceptance” of compliance verification by modeling to simulants/food. What is the conclusion? Many consumer have a mistrust to science they may not fully understand.
- Ask EFSA AFC for their opinion on the completed work, including their suggestions for inclusion of this new science in the respective EFSA (Note for Guidance) and Commission documents (Directive 2002/72/EC, 85/572/EEC, Practical Guide)
Conclusions

• Excellent scientific work
• Will further support the safety of FCM’s
• Can lead to reduced data packages for petitions of new substances
• Can demonstrate that concentrations in actual food (t/T depending) can be lower as well higher than seen with food simulants
• Work accomplished should be made available to a broad audience.
The plastics converters view

G. Tillieux, EUPC
Food contact: exposure to plastics converters view

Food migrosure conference
Baveno, 27 September 2006
Geoffroy TILLIEUX

PRESENTATION OUTLINE

I. Political background/upcoming legislation
II. Exposure: where we are now
III. An outlook to the future
I. Political background/upcoming legislation
II. Exposure: where we are now
III. An outlook to the future
Plastic Packaging: the overall picture

Unfavorable economical trend for plastic packaging producers will continue in 2006

EX: cost of production of Std. Laminate 70% PA-6 + 30% PE
Where is the legislator in this picture?

- Importers are in principle responsible for products manufactured outside EU, but as a matter of fact control is totally absent!
- Costs of compliance for EU converters is 000€ per product, and will increase >100% with NIAS testing;
NIAS: Non-Intentionally-Added-Substances

Substances present in plastics and other materials on top of regulated components (i.e. monomers and additives), derived from degradation or reaction during the production process.

Plastic Packaging: the overall picture

- Increasing compliance burden is easy for the legislator but scarcely effective
- More efficient and frequent control is the only way to hit illegal behaviors
Risk Perception often drives decision making

- Authorities’ Risk Management often based on “Risk Perception”, esp. at national level;
- …despite EFSA;
- Striving for sound Risk Communication, but the objective is often failed.

Rules for compliance are often complicated or unclear

*Surface-to-Volume* (4th am. Art. 2.1 (g) (iii)) to be identified in the Declaration of Compliance

*Ex: chicken breast vs. whole bird*
Subject to misinterpretations

Example: substance with SML=30 mg/kg
Experimental migration = 5 mg/sq.dm
(i.e. 30 mg/6 sq.dm)
Packaging 100 g food in 2 sq.dm means 10 mg of migrant in 100 g food

- Eating 100 g of food ➔ intake of 10 mg of migrant
- **But**
- Eating 1 kg of food ✗ intake of 100 mg of migrant!!

Fourth amendment

- Fat reduction factor concept introduced but hardly useable (not in conjunction with 6 dm² convention)
- Simulant D reduction factor not allowed anymore for thin films
I. Political background/upcoming legislation
II. Exposure: where we are now
III. An outlook to the future
Current tools to assess Exposure

- Exposure assessment made by EFSA
  a person consumes every day in his lifetime 1 kg of food packaged in 6 dm² plastics from which each authorized substance is migrating up to its maximum migration into the food

\[
\text{Exposure} = \frac{\text{Migration (mg} / 6\text{dm}^2)}{1(\text{person} \cdot \text{day} / 6\text{dm}^2)}
\]

Consequences

- Need for migration evaluation
  - Testing (involves cost)
  - Testing in simulants (more practical approach, reduces testing cost, but imperfect: often over-conservative, sometimes not well predictive)
  - Modelling of migration into food: food migrosure, positive step for making compliance more
    - cost efficient (less testing)
    - closer to reality (better picture of migration into food)
    - need to apply new findings disturbing as less as possible current practice (PROPORTIONATILITY), not introducing new restrictions when not justified from a safety point of view, (consider other sides of the exposure equation)
  - Still work to be done in multilayers, set-off behaviour (inks)
I. Political background/upcoming legislation
II. Exposure: where we are now
III. An outlook to the future

3 Red Points

1. Facing food contamination scares: defensiveness vs. proactiveness
2. Adequate response tools needed for unproportionate measures;
3. Next steps.
1. Need to switch to a PROACTIVE approach

- Set-up and execute crisis prevention programmes;
- Establish crisis management processes and responsibilities.
- Communication and education (FCM users, regulatory authorities, retailing companies, consumers);
1. Developing and “full exposure-based” approach for FCM

\[ \text{RISK} = \text{HAZARD} \times \text{EXPOSURE} \]

- Quantify exposure to FCM migrants, and foster it as the main criterion for risk assessment
- The MATRIX Project
  - Objective: To establish “Correlation Factors” enabling to assess the consumer exposure to substances migrating from plastic packaging materials and articles into the food groups of his daily diet.

2. Lack of responsiveness tools triggers unproportionate measures

- Increased attention on risk assessment of NIAS;
- Heavy obligations on Declaration of Compliance
  - Ruling Surface/Volume for compliance;
  - Disclosure of confidential information.
- Address multilayers and multi-material multilayers
2. Expoplast Project

**Expoplast**

**Plastics Converters Consortium for exposure in Food Contact:** The EuPC consortium set-up to develop exposure tools for converters

- **CSL** stochastic model: a software for assessing exposure (developed by packaging industry consortium)
- **Matrix** project: Coordinated effort of EuPC, Plastics Europe, CEFIC-FCA, FPE, CIAA;

*Investing in “exposure” means investing on the future*

---

**Extract from the Matrix**

---

Geoffroy Tillieux / 27-09-2006
3. Impact of incorporation of “exposure” in the current legislation

Current legislation + Exposure

SWOT analysis

Which rules? Which scientific tools are missing?

3. Next steps

- Organize Packaging value chain and authorities in research in order to:
  - Fill in knowledge gaps in risk assessment
  - Harmonize approaches at EU level
  - Develop practical cost/efficient solutions
Areas of research for potential project(s) to be organized in modules

Summary

- Risk assessment of NIAS is the N. 1 challenge;
- Taking initiative, master the context instead of being mastered by it;
  \textit{If you don’t create powder, you will end up to eat powder} (Clint Eastwood)
- Let’s prepare today the team for the next game.
Thank you for your attention!

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The food industry view

A. Mandanis, Nestlè
The food industry view

A. Mandanis, Nestlé

Nestlé’s View on Packaging Safety & Compliance and Benefits from the FOODMIGROSURE project
Outline

- Quality by Nestlé
  - Our perception of packaging issue today
  - Managing the Packaging Safety and Compliance
    - Monitoring
  - Food packer’s expectations
    - The Certificate of Compliance (CoC)
    - Upstream traceability
  - Where the modelling tools do apply

What is Quality by Nestlé?

[Diagram showing a pyramid with various layers labeled Consumer Satisfaction, Safety, Compliance, Health, Social, Sensory, and other aspects related to food quality and safety.]
Product quality & Consumer satisfaction

Non Negotiable

Food Safety
Legal compliance

Competitive Quality

Consumer preference
Consistency
Value for money

Quality must be managed throughout the whole supply chain
Every employee must understand and play his or her role in delivering value to customers and consumers

The Nestlé Quality System is translating the Quality Policy into practice

Materials in contact

Materials in contact (processing)
- conveyor belts
- ion exchange resins
- plastic moulds
- rubber
- etc.

Packaging materials
- polymers
- coatings
- jute
- wood
- glass
- metals
- paper/board
- etc.

Promotional items
- toys
- premiums
- pacifiers
- accessories
- measuring spoons
- ice cream sticks

Auxiliary items
- etc.

The Nestlé Quality System is translating the Quality Policy into practice

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WHY?

- ITX
- Inks
- Plasticizers
- Adhesives
- Coatings
- Additives
- ?

Nestlé Research Center
Packaging Safety & Compliance
We know that … !

*We are responsible for delivering safe packs to our consumers*

To ensure that, we need:

- Early identification and elimination of chemical contaminants in packaging.
- Awareness on the risks linked to packaging being a source of chemical contaminants into foods,
- Getting transparency from our packaging suppliers on material composition,
- Packaging specs that include relevant safety requirements, considering types of food, process conditions, target consumers and specific use.
The Packaging Supply Chain
→ A “communication” challenge ←

The Packaging Supply Chain

Resin Manufacturers
Adhesive Manufacturers
Converter
Food Packer
Trade
Authorities
Consumers
Excl. Chemical Industry

EU Legislation - Current

DIR 2002/72/EC
non-exhaustive Positive list: monomers and other starting substances which may be used in the manufacture of plastic materials and articles

Currently non targeted compounds

But NON INTENTIONALLY ADDED SUBSTANCES (e.g. impurities)

Processing aids:
- Mixing
- Additives machinability
- Lubricants

Breakdown / Reaction products

Materials and articles do not transfer their constituents to food in quantities which could:
(a) endanger human health; or...
(b) bring about an unacceptable change in the composition of the food; or...
(c) bring about a deterioration in the organoleptic characteristics thereof
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“Non-issue“ Packaging

- Safe
- Compliant
- No negative perception
- Agreed – Acknowledged by all parties
Achieving “Non-issue“ Packaging

Quality & Safety by design applies to packaging

From concept → Early Warning
through product and process development → Safety requirements
raw material supply → Specifications
manufacturing → Monitoring
and supply chain → Supplier audit
to consumer plate → Migration – Exposure
Setting Safety Requirements for Packaging Materials

- Safety requirements for packaging materials describe:
  - The requirements to ensure that packaging materials are safe and comply with legislation and Nestlé policy.
  - Nestlé's duty and the vendor's duty that allow specifying the requirements properly.
  - An approach (decision tree) to the safety evaluation of a material and its acceptance for use.

Monitoring

- To conduct a planned sequence of observations or measurements to assess whether all quality parameters are under control.

In the frame of Packaging Safety, monitoring means the measurement of the migration of material components into the food in contact in order to verify that this migration is below the legal limit.
What to monitor?

- There are hundreds, even thousands, of material components.
- Alone, the Synoptic Document of the European Union lists more than 2700 substances, 367 having a restriction (e.g. specific migration limit).
- The fact that there is a legal limit or restriction does imply that verification of compliance, e.g. through monitoring, must be carried out.
- Packaging monitoring plans must be established following a risk-based approach.
- Worst case calculations can ease the monitoring load.
- **Refined modelling can be applied if necessary. FOODMIGROSURE project is essential.**

Reactive monitoring

- Verification driven by a current issue or issue that occurred in the past:
  - Unsaponifiable in jute sacks
  - BADGE in coatings
  - SEM and ESBO in metal closures
  - ITX in UV-printed material

  **Easy to define, less easy to implement!**
Pro-active monitoring

- Consists in predicting which are the components whose migration could exceed the limit.

As there are hundreds of substances in packaging materials, a selection has to be made according to some criteria.

A preliminary list of substances for monitoring

- ESBO (in metal closures)
- Phthalates
- Other plasticisers (citrates, adipates, etc.)
- Styrene monomer
- Residual solvents
- Photoinitiators
- Mineral hydrocarbons
- Unsaponifiables (jutes sacks)
- Halogenophenols
- PAA
- BFDGE, NOGE, BADGE chlorohydrines
- BPA
- Olfactory check
- Taint transfer test
Food packer’s expectations

"Acquisition of warranties and assurances from suppliers can contribute to a due diligence system."

but ...

"Reliance cannot be placed on warranties nor on general assurances from suppliers."

Butterworths, Law of Food & Drugs, Issue 44
**EU Directive 1935/2004**

**Food packer's expectations**

* Art. 6 (5): "The specific directives shall require that such materials and articles be accompanied by a written declaration attesting that they comply with the rules applicable to them."

**EU Draft "4th Amendment" of Directive 2002/72/EC**

**Food packer's expectations**

* "The declaration shall contain the following information:
  
  [...] 
  
  Adequate information relative to the substances used for which restrictions are in place under relevant Community legislation to allow the downstream business operators to ensure compliance with those restrictions and list of substances used for which an SML or specifications are established".
...the future CoC as required by EU regulation...

Food packer's expectations

Specific migration
List of additives having a restriction in EC 2012/72 (as amended by date).

<table>
<thead>
<tr>
<th>Name of Additive</th>
<th>PM/REF No</th>
<th>Restriction, e.g. SML*</th>
</tr>
</thead>
</table>

Specific migration limit

Additives having multiple functions
List of additives that have a double function (e.g. food additive and packaging additive)

<table>
<thead>
<tr>
<th>Name of Additive</th>
<th>PM/REF No</th>
</tr>
</thead>
</table>

...the future CoC as required by food industry

Food packer's expectations

- As above, plus:
  List of substances that do not have a restriction in the positive list (exceptions are defined)
The past Nestlé Policy

Food packer's expectations

- Certificate of compliance sufficient for most simple applications,
  - e.g.: - Non-printed plastic film,
            - Virgin cardboard

The past Nestlé Policy

Foodpacker's expectations

Certificate of compliance + detailed specifications giving the qualitative composition for:

- Can and closure coatings,
- Mineral hydrocarbons,
- Plasticisers,
- Auxiliary items (when contact with mouth occurs)
The past Nestlé Policy

- Foodpacker's expectations

- Dossier of compliance for:
  - Ovenable packs,
  - Inner printing,
  - Recycled materials (plastics, paper & board)

The new Nestlé Policy

- Food packer's expectations

- Certificate of Compliance giving the qualitative composition (secrecy agreement if necessary) for all materials in contact with food.
Traceability is a legal requirement in Europe

Article 17
Traceability

1. The traceability of materials and articles shall be ensured at all stages to ensure full trace control, the recall of defective products, consumer information and the attribution of responsibility.

2. With due regard to technological feasibility, business operators shall have in place systems and procedures to allow identification of the batches from which and to which materials or articles are delivered. The information should be made available to the competent authorities on demand.

3. The materials and articles, which are placed on the market in the Community shall be identifiable by an appropriate system which allows their traceability by means of labelling or other documentisation or information.
### Outline

- Quality by Nestlé
- Our perception of packaging issue today
- Managing the Packaging Safety and Compliance
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- Food packer’s expectations
  - The Certificate of Compliance (CoC)
  - Upstream traceability
- Where the modelling tools do apply

### The modelling tools do apply

At different levels:

- Migration and exposure Assessment (development level; issues)
- Setting Safety Requirements for Specifications and CoC
- Monitoring decision & Early Warning
The modelling tools do apply to the Quality & Safety by design

From concept → Early Warning
through product and process development → Safety requirements
raw material supply → Specifications
manufacturing → Monitoring
and supply chain → Supplier audit
to consumer plate → Migration – Exposure

The modelling tools do apply

For the different players of the packaging value chain:

- Chemical industry
- Material manufacturers
- Packaging manufacturers
- Food packers
- Retailers
- Consumers
- Authorities

Complexity of the packaging supply chain means many potential safety issues!
Expected benefits for all

- Easier evaluation of migration and exposure
- More accurate migration assessment (less detrimental to some valuable pack systems; warning for others)
- Less analytical resource needs
- Focus on essential and real improvement needs
- Contribution to raise awareness and common understanding of contaminants from packaging
- Contribution to speak the same language
- ...

Thank you for your attention!
National Enforcement Laboratory view

X. Trier, DFVF
An Enforcement Perspective on Migration Modelling into Foods

Xenia Thorsager Trier, Chemist, M.Sc.
FoodMigrosure conference, Baveno, Italy
September 28th 2006

FCM Group at Dept. of Food Chemistry

• Develop research methods
• Enforce compliance according to EU/DK legislation
• Investigate health hazard situations / consumer complaints

using:
- chemical analyses (migration tests + detection)
- migration modelling
- intake estimates
- toxicological in-vitro/in-vivo tests (Dept. of Toxicology)

• Advise policy making authorities
This presentation

• Aim of Food Contact Material Legislation
• Enforcement Issues related to Migration Modelling in Foods
• Benefits of Migration Modelling
• Other Implications of the FoodMigrosure Project
• Conclusions

Aim of EU Food Contact Material Legislation

• To protect all consumers from exposure to substances detrimental to human health, including consumers with extreme eating habits (e.g. children)

\[
\text{Exp} \left[ \frac{\text{mg}}{\text{kg (bw) - day}} \right] = \sum_{\text{Food}} \left( \frac{\text{mg}}{\text{bw - weight (kg)}} \right) \]

• For compliance/enforcement controls worst case testing conditions should be applied (migration experiments and intake)
Enforcement Issues

1. Can MMF protect all consumers => calculate worst case scenarios?
2. Does design of experiments give worst case constants?
3. Can the information on input parameters be obtained?
4. Are supplied input parameters trust-worthy?
5. Will the model be applied properly (risk of misuse)?
6. Legislative status of MMF compared to other tests?
7. Who will look for substances not covered by MMF?

How can protection of Consumers be achieved?

• Setting toxicologically based migration limits (EFSA) => migration data and exposure data needed
• Compliance control by packaging and food industry
• Inspire industry to keep (or start!) doing compliance controls – through enforcement controls
• Education at all levels about principles governing migration
• Development of new products with less (hazardous) migration
• Migration modelling can aid at all levels - if applied properly!
Enforcement Issues

1. Can MMF protect all consumers => calculate worst case scenarios?

It seems as YES if worst case parameters are chosen – but validation data need to be presented / evaluated

2. Does design of experiments give worst case constants?

- Crucial that constants really are worst case
- Are the $M_n$’s tested, low enough?
- Setup of kinetic experiments
  - enough data points at equilibrium?
  - measurements at enough relevant usage temperatures?
- Are assumptions OK?
  - swelling issues
  - homogeneity of plastic
- Other mass transport phenomena?
Uncertainties In MMF

- Analytical methods: Result + uncertainty should comply with limits
- MMS and MMF: Result - uncertainty should comply with limits???

- Many contributions to uncertainties from experiments + model:
  - kinetic experiments
  - migration concentration measurement
  - model equation
  - constants
  - assumptions – are they fulfilled?
  - choice of exposure conditions

=> difficult to overview for non-expert

- Uncertainty budgets are needed to create
  - transparency and trust in MM!
Enforcement Issues
4. Are supplied input parameters trust-worthy?
5. Will the model be applied correctly?

- Have $C_{p,0}$’s been measured in the final article?
- Are they trustworthy?
- Criteria to judge trustworthyness? eg.
  - history of violations?
  - updated references to regulations?
  - reference to relevant Batch numbers?
- Risk of wrong worst-case conditions
  (food simulants, contact area, K)

Enforcement Issues
6. Legislative status of MMF compared to other tests?

More precise data in food
(=> higher legal power)

The food inspector must be able to decide if a product is legal or not

=> Enforcement can only model substances for which validated exp. methods exist under current EU legislation
Benefits of Migration Modelling

- MMF and MMT could be a valuable tool to “fix” underestimating nature of food simulants / technical dir.
- Possible to enforce many substances at once
- Improved understanding of important parameters for migration for personnel involved in compliance testing
- Especially useful tool
  - when all information is readily available, e.g. during product development
Conclusions

• EU legislation aims at protecting all consumers
  => Compliance / enforcement testing means worst case testing

• MMF apparently works both for exposure and compliance testing

• Uncertainty budgets would increase non-expert trust in MM

• Obtaining trustworthy input information is crucial challenge

• Experimental testing still needed for compounds exempted from modelling, eg. NIAS, ionic and blooming substances

• Current food simulants, FRF and K values underestimate migration => need to be updated fast!

• MM can create better understanding of migration processes
  => prerequisite to increase safety level of consumers!
Ladder of Uncertainties

- For validated analytical methods, uncertainty budgets are mandatory

Organisation

Ministry of Family and Consumer Affairs

Danish Institute for Food and Veterinary Research
- Risk assessment

Danish Veterinary and Food Administration
- Risk evaluation

Region East

Region North
- (inspection + enforcement)

Region South

Dept. of Food Chemistry & project groups

Dept. of Toxicology and Risk Assessment

Dept. of Nutrition

Dept. of Microbial Food Safety

Dept. of Epidemiology and Risk Assessment

Dept. of Veterinary Diagnostics and Research

Dept. of Virology

Dept. of Poultry, Fish and Fur Animals

DFVF - vigilance and foresight

Who is DFVF and what do we do?
Compliance testing of plastics

- Exposure conditions for testing of plastics are chosen according to the Technical directives (82/711/EEC + 85/572/EEC)
- Instead of testing real foods (many tests!!), testing is allowed on
  - food simulants (water, olive oil, 3% acetic acid, 10% ethanol)
  - alternative tests (isooctane, 95% ethanol, MPPO)

Key parameters determining migration from plastics to food

- the material
  (initial concentration of chemical in FCM)
- the food
  (chose the most aggressive)
- the contact temperature, T
  (chose the highest foreseeable temp. in use)
- the contact time, t
  (chose the longest time foreseeable)
- the exposed area of the FCM in contact with the food
  (chose the largest FCM area in contact with smallest food volume)
- stresses that the FCM has been exposed to
  (e.g. during sterilisation – chose combination of exposures)
Underlying principle of EU legislation

• The principle behind the EU food contact material legislation: All citizens should be safe under the legislation, also those with extreme eating habits (e.g. children)

=> Worst case conditions are applied when choosing which substances, materials and testing conditions to enforce:
   - the most toxic chemical substances (e.g. carcinogenic)
   - materials giving the highest exposure (e.g. used for basic foods)
   - the most vulnerable groups (e.g. baby foods)
   - the highest likeliness of violations (e.g. new rules / 3rd countries)
   - highest risk of migration (e.g. high food contact temperatures)
   - worst case testing conditions are applied for compliance testing
Risk communication and heritage from the EU TRUST project

L. Pellizzoni, ISIG
RISK COMMUNICATION - EVOLUTION

“All we have to do is get the numbers right”
“All we have to do is tell them the numbers”
“All we have to do is explain what we mean by numbers”
“All we have to do is show them that they’ve accepted similar risks in the past”
“All we have to do is show them that it’s a good deal for them”
“All we have to do is treat them nicely”
“All we have to do is make them partners”

(Fischhoff)
COMMUNICATION AND TRUST

'Trust in communication refers to the generalized expectancy that a message received is true and reliable and that the communicator demonstrates competence and honesty by conveying accurate, objective and complete information' (Renn and Levine).

Components of trust in communication:
- a) perceived competence (degree of technical expertise assigned to a message or a source)
- b) objectivity (lack of biases in information)
- c) fairness (acknowledgement and adequate representation of all relevant viewpoints)
- d) consistency (reliability of arguments and behaviour based on past experience)
- e) faith (perception of 'good will' in composing information, no misleading intentions)
- f) congruence (internal: congruence within a given set of statements, external: congruence between statements and the addressees' world)
THE TRUST STUDY

TRUST
‘Food risk communication and consumers’ trust in the food supply chain’.

EC Fifth Framework Programme

Five countries: Italy, France, Germany, the Netherlands and UK
Eight research teams
2003-2005

More information: www.trust.unifi.it

CONTEXT AND AIMS OF THE PROJECT

CONTEXT
• Several food scares
• Loss of consumers’ trust in food safety
• Loss of trust in information sources

AIMS
Better understanding of features, determinants and processes of social diffusion of trust in food risk communication (with particular relation to food scares):
• Understanding cognitive models of persons exposed to food risk information
• Analyzing the role of social interaction and culture in the building of trust
• Analyzing the differentiation of trust among consumers
• Evaluating the economic impact of alternative strategies of risk communication
• Advising on effective communication strategies
QUESTIONS AND GOALS

- What? Consequences of ineffective information
- When? Evolution of food crisis & communication
- Why? Psycho & sociologic determinants of trust
- How? From mistrust & risk perception to consumer behaviour
- Define communication approaches before, during & after emergency
- Differentiate population segments and crisis contexts
- Compare costs of food crisis & info campaign

STRUCTURE OF THE PROJECT - 1

Phases:
Analysis, modelling, simulation, and policy advice.

Approaches:
- Psychology: individual determinants of trust - experiment
- Sociology: socio-cultural determinants of trust - focus groups
MAJOR FINDINGS - 1: QUALITY, SAFETY, AND TRUST

Quality: includes taste, freshness, nutritional value, ingredients origin and typology, ways of processing, ethical acceptability, etc. Quality and safety are strictly intertwined notions.

Consumers' information: labelling is a basic right rather than all that is needed. Labels however can be of little use and do not replace information (and public debate) on regulatory and corporate choices.

Perceived difficulties of effective controls related to the growing complexity of food chain, bureaucracy and costs.

Trust in information sources is relevant for risk-related behaviour. Socio-demographic factors are not relevant, and consumption styles are not directly related.

MAJOR FINDINGS - 2: EFFECTS OF FOOD CRISES

Behaviours (in order of relevance):
- temporary change of food habits (reduced or interrupted consumption of ‘hazardous’ food);
- durable change: new consumption styles adopted are considered healthier;
- no change: too late for preventive measures or usual behaviour considered protective enough.

Positive aspects of food crises:
- consumers' enhanced attention to safety and quality
- stricter rules and improved controls
- lessons for crises prevention and management

Crisis management:
- crises framed by mass media criteria
- unclear, contradictory, scarce or over-abundant information
- reticence or delays in diffusing information for risk avoidance
- lack of unambiguous expert standpoints
- lack of official communication on end of crises
- basic reliance in official information sources
- relevance of ‘two step’ information flow
MAJOR FINDINGS - 3: TYPES OF TRUST

**Interpersonal trust:** Undermined by scarce direct contact with food dealers. Difficulty for shopkeepers and dealers to guarantee quality and safety of products. Competence + access to information + good will as basic aspects for evaluating interpersonal communication.

**Systems trust:** Diffused scepticism about competence, efficiency, effectiveness, integrity of institutions and food chain organization structures, though basic trust in public and scientific authorities.

**Self-reliance:** Some consumers trust no one but themselves and believe they are able to make safe choices by their own.

**Confidence:** Some consumers say they 'cannot but' trust, as they cannot check the goodwill, competence, equity and integrity of food chain actors.

INSIGHT FOR RISK COMMUNICATION - 1

**What is known and what is not:**
Describe problems using colloquial words; no formulas or statistical data. Acknowledge uncertainty, identify its features and explain how it is faced.

**Context and framing of issue:**
- Who, what, where, when, why and how.

**Practical indications:**
- What to do and why.

**Sources and liability:**
- Who provides information and who is accountable for indications provided.

**Management:**
- Measures adopted, who is doing what, ‘forecast’.

**What comes next:**
- Who is going to provide further indications, and when.
INSIGHT FOR RISK COMMUNICATION - 2

- Consideration of impacts of previous experiences on existing relationships of (mis)trust
- Design and dissemination of messages according to target audiences (adopt different styles and adjust contents according to the public’s concerns)
- Contents consistent throughout
- Relevance of independent agencies and experts
- Honesty about the existence of scientific and technological uncertainty
- Dissemination through different media and channels
- Consideration of social amplification of risk (informal networks)
- Reiteration and continuity
- Monitoring and collection of feedback in order to apply corrections to following messages

SUMMARY OF FINDINGS

Trust is a central aspect in the food safety issue.

Food scares are neither the only nor possibly the prevalent reason for consumers’ decreasing trust.

Distrust and low sense of agency prevail, accompanied by recognition of consumers’ underexploited potential to influence the market and protect their own safety.

Evidence of efficiency, effectiveness, good will and integrity in preventing and managing emergencies is key for promoting consumers’ active orientation (action), rather than a passive one (hope) and restoring public trust.

The effectiveness of risk communication is conditional to the application of a sound approach and the previous building and nurturing of a trust relationship between citizens and authorities.
CONNECTIONS WITH FOODMIGROSURE STUDY

- No consideration of packaging in Trust (and other research). Thus Foodmigrosure provides major integration to previous research.
- Relevance of consumers’ involvement in the assessment of risk policies and risk communication.
- ‘Synthetic’ assessment of food quality and safety.
- Trust investments recognised as necessary.
- Basic trust in scientific expertise.
- Relevance of a ‘sound’ risk communication (including acknowledgement of uncertainties).
- Consumers’ ‘pragmatic’ orientation: scientific inquiry should be clearly connected with practical implications for risk prevention (including practical indications for behaviour).
Future support at DG RTD for research on food packaging

D. Bennink, European Commission - DG RTD
Current status of FP proposal:
- Modified COM proposal (28 June 2006)
- Political agreement on Common Position (24 July 2006)

Next steps:
- 2nd reading of European Parliament (end November)
- Council decision on 05 Dec 2006
FP7 Specific Programmes

Cooperation – Collaborative research

Ideas – Frontier research
People – Human Potential
Capacities – Research Capacity

+ JRC (non-nuclear)
  JRC (nuclear)
  Euratom

9 Thematic Priorities

1. Health
2. Food, agriculture and biotechnology (€1935 million)
3. Information and communication technologies
4. Nanosciences, nanotechnologies, materials and new production technologies
5. Energy
6. Environment (including climate change)
7. Transport (including aeronautics)
8. Socio-economic sciences and the humanities
9. Security and space

+ Euratom: Fusion energy research, nuclear fission and radiation protection
2. Food, Agriculture and Biotechnology

Sustainable production and management of biological resources from land, forest, and aquatic environments

“Fork to farm”: Food, health and well being

Life sciences and biotechnology for sustainable non-food products and processes

Cooperation

Theme 2 will support trans-national research cooperation through:

Collaborative research will constitute the bulk and the core of EU research funding (strongly industry driven)

International Cooperation between the EU and third countries is an integral part of this action.

In addition:

Coordination of non-Community research programmes and Research to support policies is also integrated into the themes of the cooperation programme.

Cooperation programme contributes financially to Risk-Sharing Finance Facilities (RSFF) …

…and other Community initiatives (COST, EUREKA, etc.)
Need to prioritise

“More with less”

- Theme 2 FP7 “Food, Agriculture, Biotechnology” has broader scope than priority 5 “Food Quality & Safety” of FP6 (added activity 3 on biotech products and processes for non-food applications)
- Research to support policies, international cooperation and coordination of national research is integrated into the themes
- Budget for first calls of theme 2 - FP7 comparable (or lower) than for priority 5 - FP6

Need to prioritise along the following criteria:

- New areas/topics not (little) covered in previous FPs.
- Continue/follow-up on successful EU research activities in order to achieve maximum impact.
- Preparatory actions for identifying priority topics/activity areas for future calls, i.e. analysis of certain research/technology options for addressing specific goals

Consultation and external input

Input into work programme from:
- Advisory Group of this theme
- the research community,
- interested organisations (EFSA, etc.)
- Standing Committee for Agricultural Research (SCAR)
- expert workshops, outputs from conferences
- studies, analysis of ongoing research,
- policy needs (other Commission directorates)
- strategic research agendas (SRA) of the European Technology Platforms relevant to Theme 2 (Plants for the Future; Sustainable Farm Animal Breeding and Reproduction; Global Animal Health; Food for Life; Forestry; Biofuels; the Industrial Biotechnology section of the Sustainable Chemistry Platform)
Activity 1: Sustainable production and management of biological resources from land, forest and aquatic environments:

**OBJECTIVES**

- Exploitation of new and emerging technologies
- Social and economic challenges
- Rural contexts, animal welfare
- External threats: climate change

**Sustainable production**

- Healthier, safer and higher quality foods. Non-food production
- Controlling the risk of zoonotic, epizootic and food-related disease
Activity 1: Sustainable production and management of biological resources from land, forest and aquatic environments: AREAS

- "Enabling" research
  - Micro-organisms; plants; animals
  - "-omics": bioinformatics; systems biology; tools and technologies

- Sustainable production
  - Agriculture; horticulture; forestry; fisheries; aquaculture
  - Improved crops; plant health; control of pests, disease and other threats

- Optimised animal health, production and welfare
  - Exploitation of genetics knowledge; breeding, physiology, behaviour
  - Control of infectious diseases; epizootics, zoonoses; management of by-products

- Building of the KBBE
  - Tools for policy makers in support of community policy
  - Rural and coastal development; International development
  - Socio-economics and cost-benefits. Farming systems, including non-food

Activity 1: European Technology Platforms

- Plants for the future
  - Exploitation of genomics for improved crops

- Sustainable farm animal breeding and reproduction
  - Exploitation of genomics and breeding technologies

- Forestry based sector
  - Exploitation of forest-based products

- Global animal health
  - Vaccines and diagnostics

Other ETPs
- » Food for life
- » Industrial Biotech
- » Biofuels
Activity 1: Sustainable production and management of biological resources from land, forest, and aquatic environments

- Enabling research (‘omics’, converging technologies, bio-informatics, biodiversity)
- Sustainable, competitive and multifunctional agriculture, forestry, fisheries, and aquaculture
- Animal welfare, breeding and production; Infectious diseases in animals, including zoonoses
- Policy tools for the knowledge-based bio-economy, agriculture, fisheries, rural and coastal development

2. Food, Agriculture and Biotechnology

Sustainable production and management of biological resources from land, forest, and aquatic environments

“Fork to farm”: Food, health and well being

Life sciences and biotechnology for sustainable non-food products and processes
Activity 2: Fork to Farm

Consumers
- Nutrition
- Processing
- Safety
- Environment

Activity 2: “Fork to farm” - Food, health and well being

**CONSUMER**
- Consumer, societal, industrial and health aspects of food and feed

**NUTRITION**
- Nutrition, diet related diseases and disorders, including obesity.

**TECHNOLOGIES**
- Innovative food and feed technologies, including packaging

**ENVIRONMENT**
- Food and feed safety and environmental impact

**FOOD CHAIN INTEGRITY**
- Control of the food chain

**TOTAL FOOD CHAIN**
- Traceability and chain management
Activity 2: European Technology Platforms

Food for life
Full chain – principally post-harvest

Sustainable production
» Plants for the future
» Forestry based sector
» Sustainable farm animal breeding and reproduction
» Global animal health

Activity 2: “Fork to farm”- Food, health and well being

- Consumer and societal aspects of food
- Nutrition, diet-related diseases and disorders, nutrigenomics, food development
- Innovative food and feed processing and packaging, smart control, waste management
- Improved quality and assured microbiological and chemical safety of food and feed, detection methods, risk governance
- Environmental impacts on/of the food chain, total food chain concept
Activity 3: Life sciences and biotechnology for sustainable non-food products and processes

- Improved biomass for energy,
- Bio-catalysis; new bio-refinery concepts
- Forestry and forest based products
- Cleaner processing

Fig. 3. The fully integrated agro-biofuel/biobased-products-bioenergy cycle for sustainable technologies.

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Activity 3:
European Technology Platforms

- Sustainable production
  - Plants for the future
  - Forestry based sector
  - Sustainable farm animal breeding and reproduction
  - Global animal health

- Industrial Biotech
  - Micro-organisms and enzymes

- Bio-fuels
  - Primary production and processing

www.suschem.org

Activity 3:
Life sciences and biotechnology for sustainable non-food products and processes

- Improved crops, feedstocks, marine products and biomass for energy, environment, and high added value industrial products; novel farming systems
- Bio-catalysis; new biorefinery concepts
- Forestry and forest based products and processes
- Environmental remediation and cleaner processing
The call approach

Work programme 2007

• CALL KBBE-2007-1
  • Published Dec 2006/early 2007
  • CSA, SCP, LCP & NoE covering all activities and areas
  • Covering full 2007 budget (appr. €190m)
  • 1 step evaluation procedure (exceptionally also for LCP&NoE)
  • Deadline for submission: April 2007

• CALL KBBE-2007-2A
  • Published Dec 2006/early 2007
  • LCP & NoE covering all activities and areas
  • Covering part of 2008 budget
  • 2 step evaluation procedure
  • Deadline for submission: Sep 2007
  • Final evaluation (Feb 2008)
Poster Presentations
WP 1: Plastics&Migrants

1a: Selection of plastics reference film
1b: Selection of model chemicals as migrant
Selection of polymeric reference films and model migrants to study the physico-chemical parameters influencing mass transport of packaging constituents into and within foodstuffs

Jan Cooper, Rainer Brandsch, Roland Franz, and Emma Bradley

Introduction

The FOODMIGROSURE project (EU Contract No. QLK1-CT-2002-2390) aims to develop a physico-chemical migration model that describes mathematically the migration processes from plastics into foodstuffs. To achieve this aim a significant amount of experimental work was conducted involving both migration kinetics as well as transport and partitioning studies using model migrants and real foodstuffs. The model migrants (11 altogether) were chosen to represent a broad spectrum of potential migrants found in food packaging, covering a wide range of molecular weights, chemical groups, polarities etc.

In Work Package 1 of FOODMIGROSURE project, four test films for the migration kinetics experiments have been selected from EU project CPRM for Specific Migration (EU Contract No. 50545-CB-CT-2000-0001). These films, which contain 5 of the model migrants, are candidate CRMs with established homogeneity, stability, composite migration and diffusion properties and were ideal for the kinetic migration experiments. A fifth film (HOPE) was selected from a Fraunhofer Institute National project which contained benzenophene and diphenylmethane.

Test films for kinetic migration experiments

<table>
<thead>
<tr>
<th>Test film</th>
<th>Density (kg/m³)</th>
<th>Thickness (mm)</th>
<th>Chemical name of model migrant</th>
<th>Structure</th>
<th>Function</th>
<th>Mw</th>
<th>Pw/Ref</th>
<th>Partner</th>
</tr>
</thead>
<tbody>
<tr>
<td>LDPE 1</td>
<td>0.915±0.001</td>
<td>131±18</td>
<td>Benzenepropionic acid 3,5-bis(1,1- dimethyl-4-hydroxyoctyl) ester (10%)</td>
<td>Artioxidant</td>
<td>618±29</td>
<td>532</td>
<td>68±32</td>
<td>JRC</td>
</tr>
<tr>
<td>LDPE 2</td>
<td>0.915±0.003</td>
<td>44±47</td>
<td>Diphenylbutadiene (DPBD)</td>
<td>Not used</td>
<td>12±1±2</td>
<td>206</td>
<td>-</td>
<td>USC</td>
</tr>
<tr>
<td>HDPE 3</td>
<td>0.042±0.004</td>
<td>51±4</td>
<td>2-hydroxy-4-n-octylbenzenophene (COB)</td>
<td>Light stabiliser</td>
<td>80±16</td>
<td>326</td>
<td>616±00</td>
<td>Pira</td>
</tr>
<tr>
<td>HDPE 4</td>
<td>1.530±0.01</td>
<td>107±12</td>
<td>Caprolactam</td>
<td>Monomer</td>
<td>21±14±1</td>
<td>113</td>
<td>142±00</td>
<td>CSL</td>
</tr>
<tr>
<td>HOPE 5</td>
<td>0.97±0.004</td>
<td>154±10</td>
<td>Benzenophene</td>
<td>Photobleaching</td>
<td>45±8±1</td>
<td>182</td>
<td>262±40</td>
<td>TUV</td>
</tr>
</tbody>
</table>

Substances selected for diffusion in food (Df) measurements

<table>
<thead>
<tr>
<th>Model substance</th>
<th>Structure</th>
<th>Acronym</th>
<th>MW</th>
<th>SML (mg/kg)</th>
<th>PM/REF</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bis(2-ethylhexyl)adipate</td>
<td>DEHA</td>
<td>370</td>
<td>16</td>
<td>31920</td>
<td>Plasticiser</td>
<td></td>
</tr>
<tr>
<td>Styrene</td>
<td></td>
<td>104</td>
<td>-</td>
<td>24510</td>
<td>Monomer</td>
<td></td>
</tr>
<tr>
<td>2,3,8,9-tetrahydroxyphenylpropane</td>
<td>Bisphenol A</td>
<td>229</td>
<td>9.6</td>
<td>13460</td>
<td>Monomer</td>
<td></td>
</tr>
<tr>
<td>1-octene</td>
<td></td>
<td>112</td>
<td>15</td>
<td>22660</td>
<td>Monomer</td>
<td></td>
</tr>
<tr>
<td>1-methyl-4-(1- methylphenyl)cyclohexene</td>
<td>Limonene</td>
<td>136</td>
<td>-</td>
<td>63970</td>
<td>Recycling</td>
<td>impurity</td>
</tr>
<tr>
<td>Disopyrphthalene</td>
<td>DIPN</td>
<td>212</td>
<td>-</td>
<td>-</td>
<td>Recycling</td>
<td>impurity</td>
</tr>
<tr>
<td>Lauric acid</td>
<td></td>
<td>191</td>
<td>5</td>
<td>19490</td>
<td>Monomer</td>
<td></td>
</tr>
<tr>
<td>Triacetin</td>
<td></td>
<td>216</td>
<td>-</td>
<td>57760</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Tri-n-butyliclacetate</td>
<td>ATBC</td>
<td>&lt;0.02</td>
<td>93760</td>
<td>Plasticiser</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,5-di-tet-butyl-p-terphenyl</td>
<td>BHT</td>
<td>220</td>
<td>3</td>
<td>469±40</td>
<td>Antioxidant</td>
<td></td>
</tr>
<tr>
<td>2,4,4'-Triphenyl-2'- hydroxydiphenyl etter</td>
<td>Triphenyl</td>
<td>290</td>
<td>-</td>
<td>93±20</td>
<td>Antimicrobial</td>
<td></td>
</tr>
</tbody>
</table>

Results

In addition to the 7 model migrants present in the test films, a further 11 model migrants were selected in Work Package 1 to investigate the parameters, diffusion in food (Df) and partitioning effects (Kw) also within food. The model migrants were chosen to represent a wide range of different physico-chemical properties, such as molecular weight, polarity and functional groups with emphasis on substances, representative of migrants from food contact materials. Other desirable attributes of the selected substances were that they should be stable and measurable at low levels in foodstuffs.

Acknowledgments

For further information about this project visit the website: www.foodmigrosure.com

Contact person: Roland Franz, Fraunhofer Institute

Appendix A: Table 1: Migration parameters

For more information about this project visit the website: www.foodmigrosure.com

Contact person: Roland Franz, Fraunhofer Institute
WP 2: Selection of foods

2a: Physico-chemical classification
2b: Consumption & related classification
Modellierung der Migration von Kunststoffen in Lebensmittel
Auswahl der Prüfliebemittel nach ihren physikalisch-chemischen Eigenschaften und der Verzehrhäufigkeit

Steiner L.1, Volansky P.1, Simoneau C.2 und Mandanis A.3
1 Institut für Verfahrenstechnik, Umwelttechnik und Technische Prozesswissenschaften (Abteilung für Naturstoff- und Lebensmittelchemie), Technische Universität Wien, Österreich
3 Nestlé Research Center, Lausanne, Schweiz

Einleitung
Ein wichtiger Aspekt innerhalb der Europäischen Union ist darauf ab, die Belastungen der Verbraucher und Verkäufer durch unwünschte chemische Substanzen in Lebensmitteln auf unvermeidliche Mengen zu reduzieren oder überhaupt auszuschließen. Lebensmittelverpackungsmaterialien stellen dabei eine nicht zu vernachlässigende Kontaminationquelle dar, die daher bei der Risikobeurteilung berücksichtigt werden muss.

Die Lösung der nachfolgenden Fragestellung wird ausgewählt: Lid Migratoren aus dem Verpackungsmaterial in das spezielle Lebensmittel, Zahl der Lebensmittel, die für die Risikobeurteilung in Betracht gezogen werden.

Grundlagen
Die grundlegende Gleichung für Belastungen durch verseuchte Lebensmittel lautet:
Belastung = (Lid M.) Lid Migratoren aus dem Verpackungsmaterial in das spezielle Lebensmittel, Zahl der Lebensmittel, die für die Belastungsbeurteilung in Betracht gezogen werden.

Eine weitere mathematische Voraussetzung für die Berechnung der Migration ist das lineare Diffusionsgesetz, das aufgrund der Annahme, dass Konzentration und Migrationsgeschwindigkeit der migrierenden Substanz, die Dichte des Kunststoffs, die Zeit und die Diffusionskoeffizienten im Kunststoff und im Lebensmittel bei der Erstellung eines Berechnungsmodells essentielle Größen darstellen. Weitere Faktoren können jedoch die Verteilungskoeffizienten zwischen Kunststoff und Lebensmittel eine entscheidende Rolle.

Da die Berechnungsmethode nur für Lebensmittel mit der gleichen Konzentration des Lebensmittels zum Lebensmittel einen wesentlichen Einfluss hat, wird sie unabhängig von der Konzentration der Lebensmittel in Extraktionslösungen verfeinert.

Ausgewählte Lebensmittel
Wasserige Lebensmittel
- Frischgemüse
- Alkohol
- Cola-getränk
- Bier
- Wein
- Milch

Fett Lebensmittel
- Margarine (80% Fett)
- Mayonnaise (80% Fett)
- Nüsse
- Käse Philadelphia (~10% Fett)
- Käse Gouda (45% Fett)
- Käsesauce (~15% Fett)
- Cottage Cheese (Frischkaltes mit 10% Fett)
- Feta
- Schlagsahne (~3% Fett)
- Kondensmilch (~10% Fett)
- Joghurt (~3,5% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)
- Schokolade (oder Schokolade, min. 40% Kakao, max. 30% Fett)

Trockene und wasserarme Lebensmittel
- Nüsse
- Butter (4% Fett)
- Würstchen
- Reis
- Fasöl

Anmerkungen
The work is supported by the EU-commission QLRT-2000-72956 "FOODMIGROSURE". The conclusions are the responsibility of the author(s) and do not necessarily reflect the opinion of the European Commission.
WP 3: Study physico-chemical parameters
COMPILATION OF ANALYTICAL METHODS TO DETERMINE MIGRATION KINETICS OF SELECTED ADDITIVES/MONOMERS IN PLASTICS TO FOODSTUFFS

A. Sanchez Silveira, T. Benedito Gacela, P. Pacheco Losada, O. Franca, L. Castell, R. Brandalise, I. Cooper, C. Simonse, J. Steinle, A. Mandani

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Introduction

The European Food Safety Authority (EFSA) has selected some reference plastics and model monomers of different chemical and physical properties as a basis for their optimization and relevance as food contact materials. The aim is to obtain reliable information to develop analytical methods in order to estimate the consumer exposure to substances that migrate from food contact plastics into food. The present work regards the final model monomers selected by the project: polyvinyl chloride (PVC), Diphénylhexafluorocéthylène, Chlorsorbin 31, Unitol OB, Caraplex, Desaphenon, Diphenyl phosphinate and BPA in two analytical procedures (1).

Table: Results of preliminary experiments in order to choose monomers to be used in analytical procedures.

<table>
<thead>
<tr>
<th>Additives</th>
<th>Type</th>
<th>Initial concentration</th>
<th>Migrated concentration</th>
<th>Migration conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVC</td>
<td>Fett</td>
<td>100 ppm</td>
<td>30 ppm</td>
<td>100 ppm</td>
</tr>
<tr>
<td>Diphenylhexafluorocéthylène</td>
<td>Fett</td>
<td>50 ppm</td>
<td>10 ppm</td>
<td>50 ppm</td>
</tr>
<tr>
<td>Chlorsorbin 31</td>
<td>Fett</td>
<td>20 ppm</td>
<td>5 ppm</td>
<td>20 ppm</td>
</tr>
<tr>
<td>Unitol OB</td>
<td>Fett</td>
<td>10 ppm</td>
<td>2 ppm</td>
<td>10 ppm</td>
</tr>
<tr>
<td>Caraplex</td>
<td>Fett</td>
<td>0.5 ppm</td>
<td>0.1 ppm</td>
<td>0.5 ppm</td>
</tr>
<tr>
<td>Desaphenon</td>
<td>Fett</td>
<td>20 ppm</td>
<td>5 ppm</td>
<td>20 ppm</td>
</tr>
<tr>
<td>Diphenyl phosphinate</td>
<td>Fett</td>
<td>50 ppm</td>
<td>10 ppm</td>
<td>50 ppm</td>
</tr>
<tr>
<td>BPA</td>
<td>Fett</td>
<td>10 ppm</td>
<td>2 ppm</td>
<td>10 ppm</td>
</tr>
</tbody>
</table>

Results

A bibliographic review of the physical and chemical properties of model monomers were made. Table 1 summarizes the available information on structural and physical properties. Due to the incomplete data found, some speciﬁc properties were evaluated in our laboratory. The results are shown in Table 2.

An extensive search of the available literature was also carried out. The conclusions of the reviewed literature on analytical methods in polymers, food simulants and foodstuffs were collected in a document which makes part of the Week 2 (2), and it is summarized in three points:

1. The literature of this subject is scarce, and has done more emphasis on the results concerning migration it’s less important and amount in polymers than on food analysis.
2. Analytical methods are in all the research and development stage and no particular set of methods has achieved generalised acceptance. It seems that there is not has studies that agree on the same procedure to determine a given compound in a ﬁxed type of sample.
3. Some of the protocols found in the literature are not described in sufficient detail to allow conﬁdent reproduction by other laboratories. This is a common situation in the early stage of the development of the analytical methodology.

To sum up, at present it is not possible to draw up reliable analytical protocols for the determination of all the selected compounds in food. For most of the monomers, any protocol drawn up on the basis of the literature, general analytical principles and experiences is necessarily a proposal which requires extensive evaluation.

Within the workshop 3a and taking in account all the needed data, the IFS has established the analytical and experimental basis for the whole package 3a and 3b. The guidelines suggested to propose analytical procedures to determine monomers in foods are shown in the Table 3.

References


For more information about this project visit the website

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Acknowledgements

The authors would like to thank all the participants of the Workshop 3a and 3b for their fruitful contributions in this work. Special thanks to the IFSA for the financial support.

283
Analytical methods for the determination of benzene propanoic acid-3,5-bis(1,1-dimethylethyl)-4-hydroxyoctadecyl ester in foodstuffs

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Introduction

In the frame of the EU funded project QLRT-2002-02340 “FOODMIGROSURE”, analytical methods for the determination of benzene propanoic acid-3,5-bis(1,1-dimethylethyl)-4-hydroxyoctadecyl ester (Irganox 1076) were developed. Irganox 1076 is commonly used as antioxidant in polymers for food contact materials and a specific migration limit is established for its migration into foodstuffs. Most of the methods developed for the determination of Irganox 1076 use food simulants as substitute of food matrices, yet are not applicable to food matrices due to their inherent complexity. Therefore for enforcement purposes in food and fluctuating studies comparing food to simulants, simple analytical strategies were developed to identify and quantify Irganox 1076 in a widest variety of selected foodstuffs.

Experimental

Chemicals, Standards and quantification

Hexane (Sigma Aldrich, Milano) was used as extraction solvent for all foods. In same cases (meat, fish, cheeses), hexane/n-hexane 50/50 mixture was added to food in order to increase sample homogenisation. For dairy products sodium chloride (Sigma Aldrich, Milano) saturated solution was used to avoid emulsion after hexane extraction. A stock solution of Irganox 1076 (Sigma Aldrich, Milano) was prepared in a concentration of 500 mg/l in hexane. The standards for calibration curves were obtained for each foodstuff by fortifying the blank food at appropriate levels (0.5-4 ppm).

Samples preparation

Foods with different nutritional and physical state characteristics were obtained from partners of “FOODMIGROSURE” project and from national markets. The samples were prepared based on 10g or 10ml, of each sample matrix (either blank or spiked with 10 ppm Irganox 1076) put in hermetically sealed glass tubes with 10 ml of hexane. In the case of fatty foods, the amount of hexane for extraction was increased (100ml) and the fat was separated by crystallization following the hexane extraction in order to dilute the samples and avoid interferences with Irganox 1076. The samples with extraction solvent were hemogenised by Ultra Turrax for 2 min, manually shaken for 2 min and then centrifuged at 2500 rpm for 10 min if necessary. The hexane phase was separated, filtered through a PTFE 0.45 µm filter and analysed by GC-MS.

Gas Chromatography coupled to mass spectrometry (GC-MS)

The extraction solutions of all the samples were analysed by GC-MS (Varian 6890 GC) under the following conditions:

GC unit: Carrier gas, Helium, flow, 1.2 ml/min. Splitless. Column, 30 m x 0.25 mm i.d., 0.5% Phenylmethylpolysiloxane (DB-1741), injection block, 280°C. Oven program: 70°C (6 min), 20°C/min to 250°C, 10°C/min to 350°C, 5°C/min to 350°C. Injection volume: 1.0 ml. Mass Detector: Electron impact ionisation (EI). SIM mode (m/z 530, 515).

Results and Discussion

As foods are complex matrices, different protocols (solvent, solvent to food ratio, extraction time, extraction mode, extraction temperature) were tailored for each. Chromatographic methods were compared for the various foodstuffs. The simplest approach consisted in dilution/extraction with hexane followed by direct analysis by gas chromatography, coupled to mass spectrometry (GC-MS). Laboratory experiments were first conducted to assess the absence of possible interferences for the food matrices with Irganox 1076, and to check the general acceptability of chromatograms. For this purpose blank chromatograms of each food matrix were compared with the same spiked food matrix as shown in figure 1 as example for Chocolate.

Fig. 1. Comparison between 0.5 ppm Irganox 1076 in Chocolate and a blank of Chocolate.

A recovery study was performed fortifying each foodstuff with the model compound. As shown in table 1, the average recovery over six replicates for each food was > 85.5% regardless of the concentration of the model migrant.

Table 1. Recovery obtained for each food

<table>
<thead>
<tr>
<th>Food</th>
<th>R</th>
<th>Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minced Meat</td>
<td>1</td>
<td>86.79</td>
</tr>
<tr>
<td>Chicken Breast</td>
<td>1</td>
<td>86.79</td>
</tr>
<tr>
<td>Milk</td>
<td>1</td>
<td>84.15</td>
</tr>
<tr>
<td>Cottage Cheese</td>
<td>5</td>
<td>85.10</td>
</tr>
<tr>
<td>Parmesan</td>
<td>5</td>
<td>83.10</td>
</tr>
<tr>
<td>Gorgonzola</td>
<td>3</td>
<td>83.10</td>
</tr>
<tr>
<td>Chocolate</td>
<td>3</td>
<td>83.10</td>
</tr>
</tbody>
</table>

Conclusion

Different protocols were carried out to develop adequate analytical methods for the detection of Irganox 1076 in each food matrix. The GC-MS (SIM mode) method was suitable to achieve the detection limit of 10 ppt and recoveries average over six replicates were higher than 85.5% for each foodstuffs.

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Acknowledgments

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Determination of Butylated Hydroxytoluene (BHT) by HPLC-UV and GC-MS in Food Samples

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Introduction

BHT is a synthetic phenolic antioxidant (SPA) which is commonly used as preservatives in a wide range of food products. It improves product stability and aids in the prevention or, at least, delay lipid oxidation. Nevertheless, BHT can also be used as indirect additive through diffusion from plastic packaging or be added to commodities to avoid degradation reactions in every stage of the cycle life of a polymer for long-term protection.

When BHT is used in food packaging, it migrates into foodstuffs during the processing or storage because it is rather a small molecule which can move easily [1]. In the recent Spanish legislation imposed specific migration limits regarding potential migrant substances from plastics to foodstuffs. The specific migration limit imposed to BHT is 3 mg BHT/kg foodstuff [2].

The aim of this work is to develop an analytical method for the determination of BHT as a result of migration from packaging plastics into foodstuffs.

Two representative food items were chosen to test the analytical complexity of the food matrices: chicken breast meat (rich in a high protein content and low fat content) and Gouda cheese (rich with a high lipid content). Several trials were carried out to optimise the sample preparation procedure and the chromatographic conditions.

Materials and methods

10 g guinea cheese + 20 mL hexane

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Soxhlet 10 min + centrifugation</td>
</tr>
<tr>
<td>2</td>
<td>Residue is extracted with 2 x 10 mL ACN</td>
</tr>
<tr>
<td>3</td>
<td>Evaporate ACN phases to dryness</td>
</tr>
<tr>
<td>4</td>
<td>Re-dissolve in 10 mL ACN and filter</td>
</tr>
</tbody>
</table>

HPLC-UV

GC-MS

10 g chicken breast + 2 mL ACN

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Shake 10 min + centrifugation</td>
</tr>
<tr>
<td>2</td>
<td>Evaporate hexane phases</td>
</tr>
<tr>
<td>3</td>
<td>Add the 1.3 and fill it to 10 mL</td>
</tr>
<tr>
<td>4</td>
<td>Dry hexane with Na2SO4</td>
</tr>
</tbody>
</table>

HPLC-UV

GC-MS

Results and Discussion

Table 1: Calibration curve parameters and detection limit.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>HPLC</th>
<th>GC-MS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slope</td>
<td>5.645</td>
<td>0.0917</td>
</tr>
<tr>
<td>Intercept</td>
<td>1.71</td>
<td>0.120</td>
</tr>
<tr>
<td>Correlation coefficient</td>
<td>0.9995</td>
<td>0.9992</td>
</tr>
<tr>
<td>Range</td>
<td>0.1-10 µg/mL</td>
<td>0.1-10 µg/mL</td>
</tr>
<tr>
<td>Detection limit</td>
<td>5 µg/L</td>
<td>25 µg/L</td>
</tr>
<tr>
<td>GC-MS</td>
<td>20 ng</td>
<td>6.5146</td>
</tr>
<tr>
<td>Correlation coefficient</td>
<td>0.9997</td>
<td>0.9973</td>
</tr>
<tr>
<td>Range</td>
<td>0.1-10 µg/mL</td>
<td>0.1-10 µg/mL</td>
</tr>
<tr>
<td>Detection limit</td>
<td>20 ng</td>
<td>6.5146</td>
</tr>
</tbody>
</table>

Table 2: HPLC and GC results for recovery and repeatability.

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Spiking level</th>
<th>Mean (µg/mL)</th>
<th>Recovery (%)</th>
<th>Repeatability (RSD, %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chicken breast</td>
<td>1</td>
<td>0.671</td>
<td>112.9</td>
<td>6.1</td>
</tr>
<tr>
<td>Gouda cheese</td>
<td>0.5</td>
<td>0.671</td>
<td>113.2</td>
<td>0.4</td>
</tr>
<tr>
<td>Gouda cheese</td>
<td>0.5</td>
<td>0.685</td>
<td>83.9</td>
<td>0.8</td>
</tr>
<tr>
<td>Gouda cheese</td>
<td>0.5</td>
<td>0.621</td>
<td>103.4</td>
<td>18.0</td>
</tr>
<tr>
<td>Chicken breast</td>
<td>1</td>
<td>0.882</td>
<td>85.6</td>
<td>4.9</td>
</tr>
<tr>
<td>Gouda cheese</td>
<td>1</td>
<td>0.713</td>
<td>71.3</td>
<td>5.1</td>
</tr>
</tbody>
</table>

References


Acknowledgement

This work is supported by EU contract EGF2-CT2002-2301 “FOODMICROSURE” and the Ministry of Science and Innovation (Spain) (PIE-07/073/RUG-2007/PIE). The authors are grateful to the "Plan Nacional de I+D" Program financed by the Ministry of Education and Science and to FIS (P08/00234) and F 

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EVALUATION BY HPLC-UV OF TWO METHODS TO CONTAMINATE POLYOLEFIN FILMS WITH MODEL MIGRANTS

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Introduction

In order to ensure the packaging food safety, it is necessary to carry out migration and partition experiments, which require, suitable migrant(s) release systems. For this purpose, a plastic film sufficiently permeant to perform the migration and partition experiments are, generally, not suitable commercially. Therefore, it is useful to use an adequate contamination method in order to obtain plastics contaminated with a known concentration of the migrant(s) target of the study. In our case, we wanted to have a plastic contaminated with linoleum and LDPE. The polyethylene, a LDPE film (450 μm of thickness and 2.02 of density) was not contaminated with the substances we wanted to study. Therefore, in order to contaminate the LDPE film, two methods were tested: the full immersion method and the polyethylene (PE) wax method.

Materials and Methods

1st METHOD: THE FULL IMMERSION METHOD

2nd METHOD: THE PE-WAX METHOD

Results and Discussion

Table 1: Contamination level of three different LDPE films using the PE-wax method.

References

Acknowledgments

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286
Determination of Diphenylbutadiene Migration in Foodstuffs from Packaging Materials

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Introduction

In the last years, food packaging has gained a widespread importance in the food industry. An essential investigation field within food packaging is focussed on the possibility of migration of chemicals from these materials. 1,4-Diphenylbutadiene (DPBD) CAS No. [131-64-3] is an optical brightener incorporated into a wide range of polymeric materials.

Framed in Foodmigrosure project, DPBD was chosen as a model; it is able to study migration kinetics from polymeric materials so that the results obtained could be extrapolated to other substances of similar characteristics.

As an analytical method was developed and optimised to determine DPBD in orange juice and chicken as representative samples of aqueous and solid and proteins foods.

Materials and methods

A stock standard solution was prepared by dissolving 100 mg DPBD in 100 ml of ethanol and was kept at -4°C in the refrigerator. Intermediate standard solutions of DPBD were prepared by dissolution of appropriate amounts of stock standard solution in acetonitrile. These solutions were stored at -4°C in the refrigerator.

Chromatographic conditions

HPLC:
Column: Kromasil 100 C18 column (100 mm x 4.6 mm i.d. 5 µm particle size).
Flow rate: 1 ml/min, injection volume: 50 µl.
Detection: 330 nm.
Fluorescence detection: λex 333 nm, λem 375 nm.

GC/MS:
Column 30 m x 0.25 id, 1 µm film thickness (DB-5 MS).
Temperature program: 160°C for 1 min, at 15°C/min to reach 260°C and then held at 260°C for 14 min.
Injections of 1 µl of samples were in split mode injector T: 290°C; Carrier gas: He at 1.9 ml/min.

Extraction procedure:

- 10 g of orange juice + 10 ml hexane
- 16 g of chicken breast + 2 ml acetonitrile
- Centrifugation 3000 rpm 10 min.
- Repeat extraction with 10 ml of hexane twice
- Collect all hexane phase and evaporated by dryness using a rotary evaporator
- Redissolve residue in 10 ml acetonitrile 50%/v/v and then homogenize by ultrasonics.
- Filter the solution and inject.

Results

Method validation parameters

<table>
<thead>
<tr>
<th></th>
<th>Linear range µg/ml</th>
<th>Slope</th>
<th>Intercept</th>
<th>LOD (µg/ml)</th>
<th>LOQ (µg/ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UV</td>
<td>0.1-10</td>
<td>0.51</td>
<td>3.26</td>
<td>0.269</td>
<td>0.529</td>
</tr>
<tr>
<td>FLD</td>
<td>0.1-10</td>
<td>0.56</td>
<td>3.26</td>
<td>0.267</td>
<td>0.517</td>
</tr>
</tbody>
</table>

Fluorescence excitation spectra of DPBD standard solution of 1 µg/ml.

Conclusions

- Calibration data showed excellent correlation coefficients indicating suitability for DPBD quantification.
- The method was able to detect at least 0.005 µg/ml DPBD when UV detections is applied and 0.012 µg/ml DPBD in case of FLD detection.
- There is any literature reviewed which develops an RP-HPLC method for the determination of this compound, therefore, the method presented is innovated and novel.
- Samples were spiked at three concentration levels (5, 1 and 0.5 µg/ml) before extraction. Recoveries were evaluated on the basis of six determinations for each sample. Mean recoveries are listed above and were always satisfactory, higher than 93% for UV detection and higher than 71% for FLD detection.
- However, as a result of the instability of DPBD to light, it has been checked that the FLD signal is more affected, originating this way that curing the extraction process recoveries obtained were lower for the fluorescence signal.
- The proposed RP-HPLC method was sensitive and may be considered a good analytical tool for the routine determination of DPBD in not fully foodstuffs as result of migration from food packaging.
DETERMINATION OF TRICLOSAN MIGRATION IN FOODSTUFFS FROM PACKAGING MATERIALS

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Introduction

During the last three decades, triclosan has been widely used as a broad spectrum antibacterial and antifungal agent. It has been incorporated in various consumer goods (hand soaps, toothpastes, soap bars, deodorants, household products, etc.) as well as in textiles, plastic consumer goods, and toys. As an additive in plastics it prevents the growth of micro-organisms, avoiding the formation of stains and odors and, therefore, allowing to extend the polymer’s shelf life. For this reason, its inclusion in food-contact polymeric materials has been considered. However, lately, triclosan safety has been questioned in what concerns to environmental impact and human health risk. In line with these facts, it is emergent the study of the levels of triclosan migration into foodstuffs in contact with triclosan-containing plastics. Framed in Foodmicrosure project, triclosan was chosen as a model migrant to study transport/migration processes into/within foods. An analytical method was developed and optimized to determine triclosan in orange juice, chicken and cheese as representative samples of aqueous and acidic proteins and fatty foods. This work describes an HPLC-LV method used for the quantification of triclosan in food matrices. Positive identification is carried out by HPLC-MS and GC-MS.

Fig 1 - Three-dimensional thiazole chemical structure.

Fig 2 - Three-dimensional plot of the UV-visible spectra of a triclosan standard solution of 1 mg/ml.

Materials and methods

EXTRACTION PROCEDURE FOR CHEESE

10 g homogenized cheese
3 x 10 ml hexane
hand-shaken (10 min), centrifugation

Soluble residue is discarded
Collectable phases are taken to dryness in a rotary evaporator
Residue is extracted with 2 x 10 ml acetonitrile
Proced phrases are taken to dryness in a rotary evaporator
Residue is re-dissolved with 10 ml acetonitrile (90%)
Filtration

Ingest 50 µl in the HPLC-LV.

Calibration curves showed excellent correlation coefficients (r² 0.999), indicating suitability for triclosan quantification.

The method was able to detect at least 0.25 µg/ml triclosan.

To evaluate the recovery, samples were spiked at three concentration levels (0.1, 0.5, and 1 µg/ml) before extraction. Recoveries were estimated on basis of six determinations for each sample. Mean recoveries are listed above and were always satisfactory, having a percentage error between 8.3% and 10.3%.

The proposed RP-HPLC method was sensitive and may be considered a good analytical tool for the routine determination of triclosan in foodstuffs. However high lipid content foods, such as Gouda cheese require an external sample preparation method.

Fig 3 - RPLC chromatogram of a Gouda cheese spiked with triclosan (1 µg/ml).

Fig 4 - Full scan 10.0 mass spectra of triclosan.

References


Acknowledgments

The Foodmicrosure project is supported by the European Commission under the Framework Programme 6. © Universidade de Santiago de Compostela.
Analysis of benzophenone and diphenyl phthalate as migrating substances from plastic food packaging materials into representative foodstuffs

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Introduction

To establish a model to predict the migration of substances from plastic food packaging materials into foodstuffs it is necessary to analyse the amount of the migrating substances into real foodstuffs instead of food simulants. At least 20 foodstuffs were selected representing the most important food groups, considering also the consumption rates in the European Union. This selection includes dairy products, fruits and vegetables, meat and carbohydrate containing foods (baked hard processed).

At least the following foodstuffs were selected:

- Acidic and acidic foodstuffs: orange juice, apple sauce, tomato ketchup, cola, milk, wine, beer.
- The fatty foodstuffs: margarine (80% fat), mayonnaise (80% fat), cheese (Philadelphia), (63% fat), chicken liver (45% fat), cheese sauce (19.8% fat), cottage cheese (fresh cheese with 10% fat), whipped cream, 1% (30% fat), iced cream (10% fat), milk (min 3.5% fat), yogurt drink (0.0% fat), smoothie (max, milk part min 40%), soya content (20% fat), chocolate spread (25% fat), meat (lean pork meat, minced, fat content 5% salmon, chicken).
- Dry foodstuffs: milk powder, butter toast (4% fat), wheat flour, rice, honey.

Materials and methods

General method for aqueous foodstuffs (wine, beer, apple sauce, juice, cola, kekshaup, etc.):

After adding the test substances and the internal standard (ISTD) the sample (wine, beer, juice, cola) is hand-shaken with dichromate in a separator funnel, centrifuged; the organic phases collected into an Erlenmeyer flask with 1.2 g of anhydrous sodium sulphate to dry the extract. The extract is evaporation of the residuum to enhance recovery, then filtered through a glass filter (02), and analyzed by GC-MS.

For ketchup and apple sauce 50ml of water are used to dilute the sample before extraction with dichromate.

General method for foodstuffs with intermediate fat content (meat, milk, etc.):

After adding the test substances and the ISTD milk is extracted (hand-shaken) with diethyl ether/potassium ether (1:1 v/v), centrifuged. A defined part of the upper layer is evaporated to dryness at 30°C. After adding hexane and acetonitrile the mixture is mixed for 30 seconds on an overhead mixer (vortex), the phases separated and the lower (acetonitrile) phase taken for GC-MS analysis.

Meat is cut into small pieces and then the test substances and the ISTD are added in case of meat treatment with 25% MCI is necessary prior to extraction. The following procedure is done as described before.

General method for foodstuffs with high fat content (butter, mayonnaise, cheese etc.):

A defined weight of butter and chocolate (depending on the fat content) and the ISTD are suspended in hot distilled water prior to extraction. In case of cheese treatment with 25% MCI at elevated temperature (30°C, 9040 min) is necessary prior to extraction in order to open the matrix. The extract solvent is dichromate for mayonnaise, diethyl ether/potassium ether (1:1 v/v) for butter, chocolate and cheese. After analysis internal a definite figure of the extract is evaporation to dryness. The residue is re-weighed with hexane/acetone (1:1 v/v), the volume of the residue is different for each foodstuff. The extraction with hexane/acetone is repeated a second time (this way a great part of the fat is transferred from the extract into the hexane phase).

For example in mayonnaise after one extraction with hexane/acetone is 97.7% of the fat originally present transfer into the hexane phase and 2.3% remains in the acetonitrile extract. The combined acetonitrile phases are evaporated under nitrogen at 40°C and taken for GC-MS analysis.

General method for dry foodstuffs (rice, milk powder etc.):

Rice is ground in a pestle greater prior to weighing. After extraction with dichromate and shaking intensity using a vortex shaker the mixture is left unbaked for 2 hours to separate the phases. The extract is filtered through a glass filter (02). After evaporation of the final extract (if necessary) it is analyzed by GC-MS.

GC-MS analysis

The analytical measurements of the test substances and the internal standard substances diocetyl phthalate (DOP, only for orange juice) and 4-methylbenzophenone (4-MUB) were carried out with a gas chromatograph (HP 5890 Series II) in connection with a mass selective detector (HP 5970).

Analytical conditions: Optimus 5-8. 30m x 0.25mm I.D. x 0.5um film.

Carrier gas and pressure: Helium at 300mL/min.

Injection temperature: 275°C; injection volume: 1ul; split flow 25ml/min.

Oven temperature program: 200°C, 0.1min → 260°C/min → 320°C, 4mins.

Detection: MS/MS module.

Results

The first analytical procedures were carried through with DOP as ISTD which proved to be unstable against some sample preparation treatments, so DOP was used for the subsequent experiments.

The following table summarizes the recovery values of benzophenone (BP) and phthalic phthalate (CPP) for a concentration level of 7.5 ppm.

<table>
<thead>
<tr>
<th>Foodstuff</th>
<th>BP recovery (%)</th>
<th>CPP recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orange juice</td>
<td>105</td>
<td>105</td>
</tr>
<tr>
<td>Cola drink</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Alue</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Beer</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Apple sauce</td>
<td>92</td>
<td>92</td>
</tr>
<tr>
<td>Ketchup</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Milk</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Constrained milk</td>
<td>102</td>
<td>102</td>
</tr>
<tr>
<td>Yoghurt drink</td>
<td>98</td>
<td>98</td>
</tr>
<tr>
<td>Butter</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Mayonnaise</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Cheese</td>
<td>92</td>
<td>92</td>
</tr>
<tr>
<td>Cheese sauce</td>
<td>105</td>
<td>105</td>
</tr>
<tr>
<td>May</td>
<td>95</td>
<td>95</td>
</tr>
<tr>
<td>Chicken breast</td>
<td>112</td>
<td>112</td>
</tr>
<tr>
<td>Fish</td>
<td>103</td>
<td>103</td>
</tr>
<tr>
<td>Butter toast</td>
<td>119</td>
<td>119</td>
</tr>
<tr>
<td>Milk powder</td>
<td>117</td>
<td>117</td>
</tr>
<tr>
<td>Rice</td>
<td>116</td>
<td>116</td>
</tr>
<tr>
<td>Note</td>
<td>95</td>
<td>119</td>
</tr>
</tbody>
</table>

The limit of quantification for most of the analyzed foodstuffs is below 30 ppb, for orange juice, cola drink, wine, beer, apple sauce and ketchup below 10 ppb, for butter and mayonnaise below 50 ppb.
Introduction

The aim of work package 3 was to establish the physico-chemical parameters for the mobility of substances in food (D) and packaging materials (A) from plastic/poly(styrene) films and other materials to foodstuffs used in food and packaging materials. This was based on a better understanding of the mechanisms and physico-chemical properties which influence the release of contaminants from packaging materials and the transfer of contaminants into foodstuffs.

Migration experiments

The foodstuffs were exposed to the release films (polyester and PA nylons) for different periods, and the migration of substances into the foodstuffs was assessed under different storage conditions.

Preparation of the release film for migration experiments with Lauroylactam and DIPN

For soaking the Polyethylene film (LDPE) in a solution of the substance, the substance was first dissolved in an organic solvent depending on the chemical properties of the substance (Lauroylactam, Dibenylpropylphthalate).

Concentration profiles of migration experiments with Lauroylactam

By cutting the foods into smaller pieces and analyzing the content of the migrant, a concentration profile was obtained. In order to facilitate the slicing process, most of the foodstuffs were stored in the freezer after the migration experiment. The level of the migrant in the foodstuffs was determined by solvent extraction using a mixture of acetonitrile in hexane. The extraction solutions were analyzed by gas chromatography with flame ionization detection.

Concentration profile measurements with Lauroylactam

For more information about this project, visit the website:

www.foodmicrosure.com

Acknowledgments

The microplastic pollution project was supported by the European Community, and the Steering Committee Board and the Project Team worked in cooperation with the BMBF (Munich, Germany) and the E.U. (Brussels, Belgium).
Migration kinetics of Irganox 1076 in relevant foods for refined exposure assessment

Sarah Pastorelli, Giorgia Beldi, Philippe Hannaert and Catherine Simonneau
European Commission DG Joint Research Centre, Institute for Health and Consumer Protection, Physical and Chemical Exposure Unit, Ispra

Introduction
The migration of Irganox 1076 (2,6-di-tert-butyl-4-methylphenol) in various foods was investigated for a variety of relevant foodstuffs (Frish carbohydride, high protein, high fat) at different time-temperature conditions.

Experimental
Chromatographic methods were compared for the various foodstuffs. The simplest approach consisted in dilution extraction with hexane followed by direct analysis by gas chromatography coupled to mass spectrometry (GC-MS). For fat foods (>10%) the fat was separated by crystallization following the hexane extraction. Kinetics were carried out on a thin film interferometer material fortified with the substance. Independent replicates of specimen film were placed in contact with the various food matrices at various time and temperature conditions and at a number of independent time points. Liquid food samples were placed in glass bottles with a hermetic cap lined with the fortified polymer. The bottles were incubated upside down to ensure contact, at different different temperatures.

Small migration cells were built for kinetic studies of semi or solid foods. The fortified polymer was cut to cover the internal surface of the cell and food was held in contact with the surface by a glass ring. The cell was closed with a glass cover pressed by a weight on the top. The cells were incubated at different conditions.

Results and Discussion
As foods are complex matrices, different protocols were tailored for each food type. Interference problems between matrix and analyte were more easily resolved, and stability during time-temperature experiments was adequate, the extraction yield and sensitivity were independent of the model compound.

Recoveries for each food were >90.5% regardless of the concentration of the model migrant.

<table>
<thead>
<tr>
<th>Food</th>
<th>Role (mg/cm²)</th>
<th>Recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cheddar Cheese</td>
<td>1</td>
<td>100.0</td>
</tr>
<tr>
<td>Whey Cheese</td>
<td>1</td>
<td>88.20</td>
</tr>
<tr>
<td>Chicken Breast</td>
<td>3</td>
<td>98.00</td>
</tr>
<tr>
<td>Milk</td>
<td>3</td>
<td>84.13</td>
</tr>
<tr>
<td>Cottage Cheese</td>
<td>5</td>
<td>84.04</td>
</tr>
<tr>
<td>Salami</td>
<td>5</td>
<td>92.58</td>
</tr>
<tr>
<td>Philadelphia</td>
<td>5</td>
<td>93.10</td>
</tr>
<tr>
<td>Gouda Cheese</td>
<td>5</td>
<td>92.90</td>
</tr>
<tr>
<td>Chocolate</td>
<td>5</td>
<td>83.30</td>
</tr>
</tbody>
</table>

The only samples where a migration of Irganox 1076 was detected were foods with high fat content (chocolate and gouda cheese) due to the chemical affinity between model migrant and fatty compounds.

Conclusion
The kinetic migration experiments confirmed the importance of physical-chemical properties of both model migrant and food matrices. The physical state of the sample seems also to influence the mass transport process.
Migration kinetics of Irganox 1076 from Food Contact Materials: influence of food chemistry and temperature

Giorgia Boldi, Sarah Pastorelli, Fabio Franchini and Catherine Simoneau

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Introduction

Food contact materials (FCM) are plastic packaging materials that protect the presentation, protection and distribution of high quality foods. Filled food packaging may contain additives to reduce degradation during processing and storage. Since packaging materials are commonly in contact with foodstuffs, chemical substances, such as antioxidants, stabilizers and plasticizers, even if generally present at low levels, can migrate from packaging materials into foodstuffs while stored.

Experimental

The concept of migration used in the present kinetic study selected by the FOODMIGROSURE project was the Cyclicadex-3-(3,5-di-buty-4-hydroxyphenylpropiolate) (Irganox 1076), normally used by the industry in FCM as antioxidant. The reference film chosen was the most widely used food packaging material, a low density polyethylene (LDPE), and the food samples used were olive oil, bottled with Irganox 1076 at a concentration around 81 mg/kg. A special migration cell was developed to hold the glass ring and film closed by a lid using Food with different nutritional characteristics and physical state were selected from the most popular or purchased from local national markets. Measurements were performed at different times and temperatures, in order to evaluate the real and most severe storage conditions that might occur. Once launched the migration free, Irganox 1076 was extracted from food with hexane, and analyzed by GC-Ms, to determine the concentration migrated from LDPE into food, expressed as µg/m² of reference film.

Results and Discussion

Kinetic in foods

34 kinetics were performed in 20 different food matrices at several times and temperature exposure conditions. The values reported in the table below represent the maximum migration obtained from the kinetic data per time of each food.

<table>
<thead>
<tr>
<th>Food sample</th>
<th>Fat content (%)</th>
<th>Temperature (°C)</th>
<th>Retained I</th>
<th>Retained 2</th>
<th>Retained 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cheese</td>
<td>17.0</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Ham</td>
<td>3.0</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Ham prod.</td>
<td>11.1</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Pecorino</td>
<td>30.1</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Prosciutto</td>
<td>7.6</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Tomasso</td>
<td>5.1</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Hamprod.</td>
<td>11.6</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Hamprod.</td>
<td>27.1</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Hamprod.</td>
<td>30.6</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Hamprod.</td>
<td>52.9</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Hamprod.</td>
<td>6.0</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Milk</td>
<td>2.63</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Milk</td>
<td>2.63</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
<tr>
<td>Milk</td>
<td>2.63</td>
<td>20</td>
<td>3</td>
<td>10.36</td>
<td>10.71</td>
</tr>
</tbody>
</table>

Conclusions

In all the kinetics performed on foods, the combination of a high temperature and a high fat content concentration was helping the migration of Irganox 1076. The migration was increasing when raising the temperature and the fat content. The results have also demonstrated the strong influence of food chemistry on migration of substances from FCM.
Kinetic studies on the migration of caprolactam from nylon-6 into foodstuffs

E. Bradley, W. Read, D. Spack and L. Castle
Central Science Laboratory, Sand Hutton, York, YO41 1LZ, UK

Introduction
Caprolactam is a monomer used in the manufacture of polymers (nylon-6) and is intended to come into contact with foods. A method for the analysis of caprolactam in foods has been developed and validated in-house. This method has been applied to the analysis of caprolactam migrating into foods from a reference film produced within EU project 6RD-CT-2000-00411 (Certified Reference Materials for specific migration). Three foodstuffs were selected based on their properties (fat content, pH, etc.) such that a range of parameters were investigated.

The migration of the monomer over a defined period of time at one or more temperatures has been determined for a number of foodstuffs to allow physico-chemical parameters (diffusion in the polymer, diffusion in the food, partitioning between the plastic and the foodstuff) to be determined. This work has been carried out such that the scope of validated mathematical modelling may be extended to include migration into foods rather than simply into food simulants (EU Contract QLRT-2001-02390). Modelling migration from plastics into foodstuffs, FOODMIGROSURE).

Materials and methods
Migration testing
The kinetics of the migration of caprolactam from the reference film have been determined for a range of foodstuffs at defined times and temperatures including:
- Cola drink: 2, 4, 8, 16, 24, 48 and 96 hours at 20°C and 40°C
- Minced pork (0, 10, 20, 30 and 50% added fat): 2, 4, 8, 16, 36, 48 and 96 hours at 5°C and 20°C

Extraction and analysis of caprolactam in foods
The foodstuff was extracted with acetonitrile (1:2) containing caprolactam as internal standard and the extract diluted using brine. The extracts were analysed by liquid chromatography coupled with mass spectrometry (LCMS). The ions monitored were m/z 69, 75 and 114 for the analyte caprolactam and m/z 142 for the internal standard caprolactam.

Results
The kinetic curves obtained for the migration of caprolactam into a cola beverage before and after degassing are shown in Figure 1. No effect was observed on the migration by degassing the cola. It may have been expected that gas bubble formation on the film could have reduced the effective surface area of the film exposed. Degassing a carbonated beverage could also increase the pH (decrease the acidity) and this might be expected to influence the migration of a nitrogen-containing chemical such as caprolactam. However, in the case of a cola the beverage would remain strongly acidic even on removal of the carbonation because of the phosphoric acid used.

Figure 1: Migration kinetics for caprolactam into cola at 20°C

The kinetic curves obtained for the migration of caprolactam into a pork mince with varying levels of added fat are shown in Figure 2. The migration was observed to be influenced by the amount of land higametised with the pork mince prior to carrying out the migration experiments. At both temperatures investigated (5°C and 20°C) the rate of the migration decreases as the fat content increases as may be expected for this water-soluble monomer.

Figure 2: The effect of fat content on the migration of caprolactam at 5°C

Similar kinetic curves have been generated for thirteen foodstuffs at two or more temperatures from time zero until the migration has reached an equilibrium. Migration modeling has been applied to the data and physico-chemical parameters (diffusion coefficients for caprolactam in the polymer and in the food as well as the partitioning coefficients of the monomer between the plastic and the foodstuff) have been determined.

For more information about this project visit the website: www.foodmigrosure.com
Contact person: reed.luc@csl.gov.uk

Acknowledgements
The food samples were purchased from local supermarket and the authors are grateful to Zotterlly and Health for their support.
STUDY ON THE MIGRATION BEHAVIOUR OF DIPHENYL BUTADIENE FROM LPDE INTO DRY FOODS

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2 - Fraunhofer Institut für Verfahrenstechnik und Umwelt, Gippsauerstr.35, D-38134, Freising, Germany.

Introduction
Plastic food packages may contain additives in order to minimize degradation during processing and storage. These additives, such as antioxidants, whiteners and preservatives, are generally present at low levels but may migrate into food packaged with these materials. The most widely used food packaging material is low-density polyethylene (LDPE).

In the present work, the kinetics migration of an optical brightener, diphenylbutadiene (DPBD), for LPDE, was determined by the FOODMIGROSURE project. In this work, the experiments were carried out using one-side contact migration cell for honey and one-side contact migration sandwicths for the other foods. Measurements were made at different times and temperatures in order to study the real and reliable storage conditions.

Materials and methods

Migration tests:

Sandwich method
1. Samples (flour, rice, fat, and powder milk) were accurately weighed in order to fill a glass vial of 0.1 ml of diameter and 0.5 mm of high and then they were filled into contact with the plastic containing the DPBD after turning over the cell. Samples were stored at different conditions (Table 1). All analyses were conducted by duplicate.

<table>
<thead>
<tr>
<th>Food item</th>
<th>Storage temperatures</th>
<th>Test conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour</td>
<td>25°C, 40°C</td>
<td>1, 2, 4, 7, 10, 20 d</td>
</tr>
<tr>
<td>Rice</td>
<td>20°C, 40°C</td>
<td>1, 2, 4, 7, 10 d</td>
</tr>
<tr>
<td>Milk powder milk</td>
<td>4°C, 30°C</td>
<td>1, 10, 30, 60, 90 d</td>
</tr>
<tr>
<td>Tea</td>
<td>20°C, 40°C</td>
<td>1, 2, 4, 7, 10 d, 16 d</td>
</tr>
<tr>
<td>Honey</td>
<td>20°C, 40°C</td>
<td>1, 2, 4, 7, 10 d</td>
</tr>
</tbody>
</table>

Table 1: Migration conditions for flour, rice, fat, powder milk, and honey.

Results and Discussion

Four rice, fat, powder milk and honey were selected by the FOODMIGROSURE project due to present different characteristics (Table 2) that represent the group of dry foodstuffs.

<table>
<thead>
<tr>
<th>Food item</th>
<th>Characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flour</td>
<td>High carbohydrate content, low fat content, rich in protein. High surface area.</td>
</tr>
<tr>
<td>Powder milk</td>
<td>Widely used also for baby food. Represents high surface area and fat content higher than 25%. Lactose is the main carbohydrate and it prevents an amorphous form.</td>
</tr>
<tr>
<td>Rice</td>
<td>High carbohydrate content, rich in proteins and low water content.</td>
</tr>
<tr>
<td>Tea</td>
<td>Representative of bakery products. Prevents porous structure and 4% fat.</td>
</tr>
<tr>
<td>Honey</td>
<td>Sufficient natural product with high carbohydrates content, low water content, no fat.</td>
</tr>
</tbody>
</table>

Table 2: Characteristics of dry foodstuffs concerning the selection of foods by the FOODMIGROSURE project.

Fig. 1: Migration of DPBD into milk at 20 and 40°C.
Fig. 2: Migration of DPBD into flour at 20 and 30°C.
Fig. 3: Migration of DPBD into rice at 20 and 40°C.
Fig. 4: Migration of DPBD into powder milk at 20 and 40°C.

> Results show migration is negligible in honey always below the quantification limit but not in the other foods. For similar time-temperature conditions, migration obtained had the following order:

milk > powder > rice > flour

- Flour has high surface area like milk powder, but its fat content is much lower. This makes it possible to conclude that fat content greatly contributes to the migration of DPBD in dry foods. Therefore, although the exact mechanism of migration is still unknown, data obtained in this work allow a better understanding of the behaviour of the migration.
- Flour and rice present similar fat content but rice has much lower surface area. Migration is dependent on the specific surface: therefore it presents higher levels in foods with high specific surface.

> Migration seems to be dependent on the fat content and on the specific surface and is not negligible in dry foods.

References

Acknowledgments
We wish to express our appreciation to EU Commission for the financial support of the FOODMIGROSURE project, to Foodstuffs Inspectorate of Spain for providing the materials and to the technical staff of the Laboratory of Food Science, University of Santiago de Compostela, for their help in the development of the work.

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KINETICS MIGRATION STUDIES OF DIPHENYL BUTADIENE FROM LDPE INTO MEAT PRODUCTS

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Introduction

One of the main concerns regarding safety of food packaging is the possible migration of chemical substances (plasticizers, additives, waxes) from plastics to foods. Due to the importance of this subject, the European Union (EU) is financing the project “FOODMIGROSURE”. One of the workpackages includes the study of the migration kinetics of selected substances from plastics to food simulants as well as the diffusion of these substances in the food products. Diphenyldibutadiene (DPBD) is an optical brightener, which has been selected by the project as model substance.

The aim of this work is to evaluate the influence of the fat content as well as the temperature of storage in the migration of DPBD into meat products and salmon.

Materials and methods

Migration tests using the sandwich method

1. Samples were accurately weighed in order to fill a glass vial of 25 ml capacity and 31 mm diameter and 65 mm height. Then they were put into contact with the plastic containing the DPBD (one side only). Then samples were stored in an aluminium foil.

2. Samples were packed under vacuum atmosphere with the aim of allowing a better contact between samples and meat.

3. Packaged samples were stored at different conditions (table 1). All analyses were conducted by duplicate.

<table>
<thead>
<tr>
<th>Substance</th>
<th>Storage temperature (°C)</th>
<th>Test conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pork neck and belly</td>
<td>36°C</td>
<td>1, 2, 4, 16, 30, 60</td>
</tr>
<tr>
<td>Pork muscle</td>
<td>36°C</td>
<td>1, 2, 4, 16, 30, 60</td>
</tr>
<tr>
<td>Different fat contents</td>
<td>23°C</td>
<td>1, 2, 4, 16, 30, 60</td>
</tr>
</tbody>
</table>

Extraction Procedure

Samples were accurately weighed in a 40 ml screw-cap centrifuge tube. 2 ml of ACN and 10 ml of hexane were added and immediately hand-shaken for 10 min. Tubes were centrifuged at 1336 g for 10 min and the hexane phase was removed. Extraction was repeated twice with 10 ml hexane. Collected hexane phases were evaporated to dryness in a rotary evaporator at 40°C. Then the solid residue was redissolved with 10 ml ACN 90% (v/v) and homogenised by sonication for 10 min. Finally the solution was filtered and an aliquot was transferred to an HPLC vial.

Chromatographic Conditions

- Column: ICN/Chromspher C18 5 µm particle size, 150 x 4 mm I.D.
- Column temperature: 30°C
- Mobile Phase: 50% acetonitrile, 50% water
- Injection: 50 µl
- Detection: 330 nm

Several types of meat have been used: chicken breast (10% fat), pork neck (10% fat) and lean pork meat (minimum fat content of 1%). The meat was cooked because fresh meat can hardly be standardized and different amounts of fat were added. Regarding to the meat, the different amounts of fat (10, 20, 30 and 60%) of pork fat were added and samples were homogenized, resulting in meat with higher (and more exactly defined) fat content. Also salmon (with a small fat level) was target of this study.

Results have shown that migration was lower in meat with low fat content like chicken (the maximum migration level found was about 15 ppm) after an exposure period of 20 days at 5°C and that migration increases as fat content does (the maximum migration level found for pork meat was about 120 ppm after an exposure period of 20 days at 5°C). However, the increase is not linear with the fat content.

References


Acknowledgments

This work was supported by the Cargill Company Ltd, USA.

For more information about this project visit the website: www.foodmigrosure.com
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TIME-TEMPERATURE STUDY OF THE KINETICS OF MIGRATION OF DPBD FROM PLASTICS IN TO CHOCOLATE, SPREAD CHOCOLATE AND MARGARINE

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Introduction

Packaging is an indispensable element in food manufacturing. The migration of components into foodstuffs is an important safety aspect of food packaging and other food contact materials. Plastic additives, frequently used to improve polymer properties and resist mechanical and chemical loads, can leach into the polymer matrix and can, therefore, move freely within the polymer matrix. Due to the increasing awareness of consumers in terms of health impacts, the importance of the migration of substances from food packaging materials to host-stimulated biological media has increased in recent years. Therefore, a great deal of research has been done on the migration of plastics into foods. The European Union has also accepted the migration modeling as an alternative to lab-testing in food contact materials (EU 2002/72/CE).

The aim of this work is to study the migration behavior of DPBD from a few plastic polyurethane (PU) films into chocolate, spread chocolate and margarine with different fat levels (50% and 80%) (fat content at different temperatures).

Materials and Methods

Migration tests

1. For margarine and chocolate spread, a glass wash of 0.9% (w/v) of diluent and 0.05% (w/v) of base was used. The samples were wrapped with an aluminum foil to protect them from light.

2. In order to allow a better contact between chocolate and plastic samples containing a DPBD with a single side exposure, samples were wrapped with an aluminum foil to protect them from light.

3. Samples were stored at different conditions (see above Table 1). All analyzes were conducted by duplicate.

Table 1: Migration tests conditions for the studied food items.

<table>
<thead>
<tr>
<th>Food Item</th>
<th>Storage temperature (°C)</th>
<th>Test conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Margarine</td>
<td>25</td>
<td>2.4 ± 10.20 ± 0.00</td>
</tr>
<tr>
<td>70</td>
<td>25</td>
<td>1.2 ± 10.20 ± 0.00</td>
</tr>
<tr>
<td>Chocolate</td>
<td>25</td>
<td>0.4 ± 10.20 ± 0.00</td>
</tr>
<tr>
<td>70</td>
<td>70</td>
<td>0.1 ± 4.50 ± 0.00</td>
</tr>
<tr>
<td>Spread chocolate</td>
<td>70</td>
<td>1.2 ± 4.50 ± 0.00</td>
</tr>
</tbody>
</table>

Results and Discussion

Diffusivity coefficients (D) were calculated using the Cahn equation (1973) and results are shown in Table 2.

Table 2: Diffusivity coefficients for margarine (50% and 80%) and spread chocolate.

<table>
<thead>
<tr>
<th>Food Item</th>
<th>Storage temperature (°C)</th>
<th>D (cm²/s)</th>
<th>α</th>
</tr>
</thead>
<tbody>
<tr>
<td>Margarine</td>
<td>5</td>
<td>5.10 ± 0.02</td>
<td>1.1</td>
</tr>
<tr>
<td>25</td>
<td>70</td>
<td>2.40 ± 0.02</td>
<td>3</td>
</tr>
<tr>
<td>70</td>
<td>70</td>
<td>1.20 ± 0.02</td>
<td>5</td>
</tr>
<tr>
<td>Margarine</td>
<td>5</td>
<td>5.00 ± 0.02</td>
<td>0.8</td>
</tr>
<tr>
<td>70</td>
<td>70</td>
<td>1.40 ± 0.02</td>
<td>2</td>
</tr>
<tr>
<td>Margarine</td>
<td>5</td>
<td>1.70 ± 0.02</td>
<td>100</td>
</tr>
</tbody>
</table>

References


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296
Migration Kinetics Studies of Diphenylbutadiene from LDPE into Cheeses with Different Fat Levels

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Introduction
Extrusion, and especially LDPE, are the films that present higher diffusion rates along polymers. Thus, it has been observed that perform in migration tests because they are representative and allow the worst case scenario.

The film of LDPE used in this work was a certified reference material (CRM) for specific migration testing, it is a LDPE film (thickness 185 μm) coated with 121 μg of fluorinated Oxy (1 and 4-hexyl-1H-1,3-bisoxadiazole (DPBD) and homogenous in terms of thickness, density and concentration of the reactant). In the present work, the migration of DPBD from LDPE is evaluated into cheeses with different fat and water levels (Goats cheese, soft cheese and cottage cheese).

Materials and Methods
Migration tests using the sandwich method

1. Glass cylinders of 0.1 cm of diameter and 9.8 mm of height were filled with samples which were accurately weighed, then they were placed inside with one of the sides of the film which is in contact with the LDPE. After that, samples were kept at an atmosphere of 50% RH.

2. Samples were packed under vacuum atmosphere with the aim of developing a better contact samaple/wax from which the silicone-containing with DPBD.

3. Packet samples were stored at 30°C. At the end of the experiment, all the samples were subjected to duplicate analysis.

Extraction Procedure [1-2]

- 10 g sample + 20 ml hexane
- Shake 2 min. Centrifuge at 3000 rpm 10 min.
- Remove extraction with 20 ml of hexane
- Evaporate hexane phases to dryness in a rotary evaporator
- Extract fat residue with 20 ml of dichloromethane, then shake 20 min in a Vortex. Repeat extraction with 20 ml of dichloromethane
- Evaporate solvent phases to 10 ml, filter and inject in the HPLC-UV

Chromatoographic Conditions
- Column: Xeromet C18 15 μm, 2.7 μm particle size, Column temperature 30°C
- Mobile Phase: 60% acetonitrile, 40% water
- Injection: 50 μl
- Detected: 254 nm

Results and Discussion
Table 1: Characteristics of the studied cheeses [3]

<table>
<thead>
<tr>
<th>Food Item</th>
<th>Volatile Protein</th>
<th>Carbohydrates</th>
<th>Fat</th>
<th>Water content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cheese</td>
<td>41.60%</td>
<td>26.84%</td>
<td>1.71%</td>
<td>27.44%</td>
</tr>
<tr>
<td>Soft cheese</td>
<td>53.68%</td>
<td>7.14%</td>
<td>1.57%</td>
<td>35.71%</td>
</tr>
<tr>
<td>Cottage cheese</td>
<td>78.96%</td>
<td>12.45%</td>
<td>2.63%</td>
<td>4.51%</td>
</tr>
</tbody>
</table>

Fig. 1: Migration of DPBD into cheeses

Newstand migration estimation is accepted by the EU regulation (Directive 2002/72/EC) as a valid and reliable method to check for the compliance with the existing regulations which aim to protect consumers.

Diffusion coefficients (D) were calculated using the Crank equation [4,5]

\[ D = \frac{2}{\pi} \left( \frac{k_0 - 1}{k_0 + 1} \right) \sqrt{\frac{t}{D}} \]

Where: \( k_0 = \frac{V_p}{V_p + V_w} \) and the performing coefficient \( K_P \) is defined as \( K_P = \frac{k_0 - 1}{k_0 + 1} \).

The lines represented in Fig. 1 correspond to the estimated migration. Cottage cheese presented a diffusion coefficient of \( 1.02 \times 10^{-7} \) cm²/s, while the other cheeses had a higher diffusion coefficient.

Goats cheese, with higher fat/water content and lower water content observed, showed the highest migration at 20 days. Otherwise, the cottage cheese with lower fat content and higher-water content presented a migration at 20 days higher than the soft cheese with higher fat content. It seems that the diffusion coefficient is not only dependent on fat and water content, but also on other physical/chemical properties of the food that determine the amount of migration and the migration of DPBD from LDPE into cheeses.

References
Kinetic migration studies from polymer films into foodstuffs

**Introduction**

FOODMIGROSURE project (EU Contract No. 'QLK1-CT2002-3390') aims to develop a physics-chemical migration model that describes mathematically the migration processes from polymers into foodstuffs. To achieve this aim a significant amount of experimental work was carried out measuring the kinetic migration of model substances from polymer films into a wide range of foodstuffs.

The films, containing 7 model migrants, were selected in Work Package 1 together with one partner assigned to each film. In Work Package 2 co-ordinated by Pira International, kinetic migration experiments were conducted using these films to obtain benchmark data needed to devise the migration model. The tests were conducted, where possible, using single site exposure techniques such as those pictured below. The test conditions given below in the table were selected as being most appropriate for the type of foodstuffs under test.

**Materials and methods**

### Foodstuffs

<table>
<thead>
<tr>
<th>Food source</th>
<th>Temp (°C)</th>
<th>Kinetic time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orange juice</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Apple juice</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Milk (3.8% fat)</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>UHT</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Tomato Ketchup</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Tomato Ketchup &amp; onion (6%)</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Potato chips</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Cereals</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Meat products</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Fish</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td>Vegetables</td>
<td>25</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>2, 4, 10, 25, 60 d</td>
</tr>
</tbody>
</table>

**Results**

Pira international were allocated Test Film 3, high density polyethylene containing 2-hydroxy-4-n octyl-phenyl-3-sulphonamido-2H-benzoxazolidone (CB1) and 2.5 2-benzoxazacyclo-hexene (UOB).

Test methods were developed using high performance liquid chromatography with UV detection to determine the two analytes together in the range of foodstuffs. See chromatogram below.

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**Acknowledgments**

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298
STATE OF THE ART OF ANALYTICAL STRATEGIES TO EVALUATE TRANSPORT & PARTITION PROCESSES OF SELECTED MIGRANTS IN FOODSTUFFS


1. State of the Art

Analytical strategies to evaluate the transport and partition processes of selected migrants in foodstuffs are crucial for food safety and quality control. The focus of this work is to present an overview of the state of the art in analytical strategies for the evaluation of transport and partition processes of selected migrants in foodstuffs.

### Chemical and physical information of the model migrants

<table>
<thead>
<tr>
<th>MIGRANTS</th>
<th>CAS Number</th>
<th>Chemical Name</th>
<th>MW</th>
<th>PI (C)</th>
<th>βP (C)</th>
<th>Solubility</th>
<th>Application</th>
<th>Structure</th>
<th>Spectral Data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Styrene</td>
<td>104-68-3</td>
<td>Ethylene oxide</td>
<td>106</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>BPA</td>
<td>80-54-7</td>
<td>Bisphenol A</td>
<td>190</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Caffeine</td>
<td>58-08-2</td>
<td>Caffeine</td>
<td>194</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>DPP</td>
<td>110-15-6</td>
<td>Dioxin 123-126-3</td>
<td>197</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Linuron</td>
<td>495-50-4</td>
<td>Linuron</td>
<td>197</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Tefluthrin</td>
<td>67-61-9</td>
<td>Tefluthrin</td>
<td>197</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Atrazine</td>
<td>1605-75-6</td>
<td>Atrazine</td>
<td>197</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>BHT</td>
<td>132-04-4</td>
<td>Butylated Hydroxytoluene</td>
<td>197</td>
<td>0.3</td>
<td>0.03</td>
<td>1.6</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
</tbody>
</table>

### Guidelines suggested to prepare analytical procedures for food migrant interactions

1. Sample Preparation
   - Extraction methods: Solid-Phase Extraction (SPE), Liquid-Liquid Extraction (LLE), etc.
   - Sample cleanup: Solid-Phase Microextraction (SPME), SPE, etc.
   - Sample concentration: Evaporation, solvent extraction, etc.

2. Analysis Procedure
   - Determination methods: GC-MS, LC-MS, HPLC-MS/MS, etc.
   - Detection methods: Electron Capture Detection (ECD), MS detection, etc.
   - Quantification methods: Standard addition, isotope dilution, etc.

### Experimental Procedure

1. Apparatus and Conditions
   - GC-MS: Use a Tiger 1100 GC/MS system with a J&W capillary column.
   - LC-MS: Use a Waters Acquity UPLC system with a Xevo G2 Q-TOF mass spectrometer.

2. Sample Preparation
   - Extraction using SPE: Use a Discover Plus SPE cartridge containing silica.
   - Sample cleanup: Use an Agilent 1200 series preparative LC system.
   - Sample concentration: Use a vacuum concentrator.

3. Analysis Procedure
   - Determination using GC-MS: Use a J&W capillary column, with a flame ionization detector.
   - Detection using LC-MS: Use a Xevo G2 Q-TOF mass spectrometer, with a electrospray ionization source.

For more information about this project visit the website: www.foodmigrosure.com

Acknowledgements

For more information, please contact the authors at info@foodmigrosure.com
Concentration profile studies of model migrants into/within foodstuffs - experimental approach

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Introduction

The essential aim within workpackage 3c from project "FOODMIGROSURE" is to measure dependent concentration profiles of model migrants in the food allowing the migration direction from the skin into the inner food compartment. To achieve this aim a meaningful set of experimental work was required and different experimental methods and test systems have been developed and/or improved.

For the investigation and study of the concentration profiles a suitable migrant release from film system was required. According to the requirements, it was necessary to develop and improve methods for the repetitive preparation of films with a homogeneous migrant concentration.

<table>
<thead>
<tr>
<th>Foodstuffs/Model</th>
<th>Method</th>
<th>Temperature (°C)</th>
<th>Time (days)</th>
<th>Exposure Type</th>
<th>Slicing method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cheese/Gouda</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
<tr>
<td>Soft cheese</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>metal box</td>
<td>saturated vials</td>
</tr>
<tr>
<td>Cottage cheese</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>metal box</td>
<td>saturated vials</td>
</tr>
<tr>
<td>Cheese spread</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
<tr>
<td>Mayonaise</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
<tr>
<td>Minced pork meat</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
<tr>
<td>Minced pork meat (10%)</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
<tr>
<td>Minced pork meat (20%)</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
<tr>
<td>Minced pork meat (40%)</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
<tr>
<td>Butter spread</td>
<td>Filter paper sandwich</td>
<td>33/5/40°C</td>
<td>30/60/120</td>
<td>ring cell</td>
<td>cutting machine</td>
</tr>
</tbody>
</table>

During the nalysis, it was developed the test design as well. For the concentration profiles studies different migrants were improved or developed concerning the food properties. The metal box cell was mainly used for the experiments carried out with food items with a soft consistency and for the rest of the foodstuffs analysed was utilised the ring cell.

One of the most important and difficult procedure after the migration experiments was the food slicing. Each food item required a particular slicing method. Regarding this aspect, it was necessary to improve and develop methods for thin and repetitive slicing of the food, in some cases was required to freeze the food before slicing to facilitate the cut.

Results

Different experimental procedures and methods were developed to permit the measurement of the concentration profiles in the selected foodstuffs. Results examples are demonstrated on poster - "Concentration profile studies of model migrants into/within foodstuffs - example of results".

For more information about this project visit the website: www.foodmigrosure.com
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Acknowledgments

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BHT, TRICLOSAN AND DPBD DIFFUSION BEHAVIOUR FROM
PLASTIC INTO AND WITHIN CHEESES

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Introduction
During recent years considerable legislative attention has been focused on food packaging materials, especially on plastics. In view of the few available data found in the bibliography concerning migration from plastics into real food matrices, the European Commission has decided to fund the FOODMIGROSURE project. This project aims to provide a more versatile and rapid tool to estimate exposure from FCM, to increase the knowledge regarding diffusion and partition mechanisms of compounds in foodstuffs. To establish a scientific basis for further amendments of food packaging EU Directives (90/220/EEC and 2002/72/EC), and to make migration modeling transparent to the consumer.

Diffused compounds (BHT, triclosan and butylated hydroxytoluene (BHT)) were chosen to carry out the present study. Their migration level into and within cheeses was investigated after being in close contact with the low density polyethylene (LDPE) container material. Two kinds of cheese were tested: Gouda and soft cheese. Moreover, the diffusion coefficients (D) and the influence of fat content and of the storage temperature were discussed.

Materials and methods
Partition tests
1. Cheeses were processed in the same way.
2. One of the surfaces of a ring (3 mm thickness and 0.43 cm² internal surface) was put in contact with a LDPE contaminated plastic.
3. Five rings were overlapped and filled with the foodstuff.
4. Samples were wrapped with aluminum foil and put into a plastic bag which was vacuum sealed with the aim of allowing a better contact sample/contaminated plastice and were stored at 9°C and 25°C. All analyses were conducted by duplicate.
5. Layers (corresponding to rings) were removed carefully. Samples were processed as for the migration tests.

Extraction Procedure [1-3]
Sample: 3 x 30 ml terrine
Shake 10 min + centrifugation
Evaporate to near dryness under a rotary evaporator
Extract residue 2 x 20 ml methanol
Repeat phases are taken to dryness in a rotary evaporator
Pre-dissolve in 10 ml methanol, filter and store

Chromatographic Conditions
Column: LKB silica 10 x 0.4 cm I.D., 5 m particle size
Column temperature: 30°C
Mobile Phase: 65% acetonitrile
Injection: 60 µl
Detection: LKB 3491 UV/Vis
Triscram: 290 nm
BHT: 202 nm

Results and Discussion
Diffusion profiles
Fig. 1: Concentration levels for DPBD into Gouda and soft cheese.
Fig. 2: Concentration levels for triclosan into Gouda and soft cheese.
Fig. 3: Concentration levels for BHT into Gouda and soft cheese.

- All migration has been accumulated in amounts higher than 85% in the 5 mm of Gouda cheese in contact with the plastic film. This is also true for DPBD and triclosan in soft cheese, whereas BHT has a higher penetration level (more than 25% has been detected in a distance higher than 5 mm of depth).

- Diffusion coefficients were calculated according to the Fickian equation (1):

\[ \frac{Q}{Q_s} = 1 - \sqrt{\pi \cdot \frac{D \cdot t}{4}} \]

Where:
- \( Q_s \) : total quantity of product which has diffused in the thickness \( e \) during time \( t \);  
- \( Q \) : total quantity of product which has diffused in the stack; \( D \): Diffusion coefficient; \( t \): diffusion time;

- For Gouda cheese there was no significant difference among the two storage temperature studies and the modelled results evaluated, being the diffusion coefficient around 0.8 x 10^-4 cm²/s. For soft cheese diffusion coefficients increased with the temperature for all migrants.

Acknowledgement
This work is supported by EU contact no. QLRT-2002-2290 FOODMIGROSURE. Authors are grateful to the "Ramón y Cajal" Program financed by the Ministry of Education of Spain. The authors are grateful to Mr. Patricia Cerro Borto and Mr. Gonzalo Herrera Vidal for their excellent technical assistance.

References
Introduction

The increasing use of plastics in food packaging has required more research regarding the interactions between foods and packaging materials. Migration is one of the more important processes that can occur as a result of these interactions, and the key parameters needed for the determination of contaminants in foods are the migration coefficient (K) of the transfer of the plastic material and the partition coefficient (Kp) of the migrant between the plastic and the food simulants. These parameters are influenced by several factors such as temperature, pH, chemical structure and molecular size of the migrant, fat content and structure of the food item (density, degree of crystallinity, orientation).

Due to the scarce amount of information about migration phenomena, this research work has determined the diffusion coefficient (D) of DPBD and triclosan into pork meat with different fat contents by measuring the extent to which these migrants penetrated pork meat. Pork meat was mixed with fat in order to obtain meat with 5 different amounts of fat meat with 0, 10, 20, 30, and 50% plus of pork raw fat. Then, it was placed in direct contact with plastic highly contaminated with DPBD and triclosan.

Materials and methods

Partition tests

1. Pork meat was mixed with fat in order to obtain meat with 5 different amounts of fat meat with 0, 10, 20, 30, and 50% plus of pork raw fat.

2. One of the surfaces of a ring (3 cm thickness and 0.43 cm internal surface) was put in contact with a LDPE contaminated plastic.

3. Five rings were overlapped and filled with the meat.

4. Samples were wrapped with an aluminum foil and put into a plastic bag which was vacuum sealed with the aim of allowing a better contact between the meat and the contaminated plastic, and were stored at 20°C for 5 days. All analyses were conducted by duplicate.

5. Layers (corresponding to rings) were removed carefully. Samples were processed as for the migration tests.

Extraction Procedure

Samples were accurately weighed in a 40 ml screw-cap centrifuge tube (1.2) ml of ACN and 10 ml of hexane were added and immediately hand-shaken for 10 min. Tubes were centrifuged at 1200 g for 10 min and the hexane phase was removed. Extraction was repeated twice with 10 ml hexane. Collected hexane phases were evaporated to dryness in a rotary evaporator at 40°C. Then the solid residue was re-dissolved with 10 ml ACN 99% (v/v) and homogenized by ultrasonic for 10 min. Finally, the solution was filtered and an aliquot was transferred to an HPLC vial.

Chromatographic Conditions

Column: Crowsil C18 15 x 4 mm I.D., 5 µm particle size
Column temperature: 32°C
Mobile phase: 50:50 acetonitrile:0.1% water
Injection: 50 µl
Detection: 330 nm (DPBD) and 260 nm (triclosan)

Results and Discussion

Partition profiles

Fig. 1: Graph plots for DPBD into pork meat with different fat contents at 25°C.

Fig. 2: Graph plots for triclosan into pork meat with different fat contents at 25°C.

The layers in close contact with the LDPE film have presented DPBD and triclosan levels that increased with the fat content. The studied migrants have been found in a considerable amount (9 mm of depth). The total penetration levels across the fourth and fifth layers (counting from the plastic film) were lower than 17% for DPBD and 13% for triclosan in all the samples assayed.

Diffusion coefficients

Diffusion coefficients were calculated according to the Kedem equation (14), which assumes that there is a continuous release of migrants from the source.

Diffusion coefficients are shown in tables 1 and 2. They correspond to the diffusion coefficients in pork meat with different amounts of added fat.

Table 1: Diffusion coefficients of DPBD and triclosan at 25°C.

<table>
<thead>
<tr>
<th>Migrant</th>
<th>Raw pork fat content</th>
<th>Diffusion coefficient (mm/µg)</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPBD</td>
<td>0%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>20%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>30%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>50%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Triclosan</td>
<td>0%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10%</td>
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</tr>
<tr>
<td></td>
<td>50%</td>
<td>0.000</td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Diffusion coefficients of DPBD and triclosan at 25°C.

<table>
<thead>
<tr>
<th>Migrant</th>
<th>Raw pork fat content</th>
<th>Diffusion coefficient (mm/µg)</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPBD</td>
<td>0%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5%</td>
<td>0.000</td>
<td></td>
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<tr>
<td></td>
<td>10%</td>
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<td>20%</td>
<td>0.000</td>
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<tr>
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<td>30%</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>50%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Triclosan</td>
<td>0%</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5%</td>
<td>0.000</td>
<td></td>
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<tr>
<td></td>
<td>10%</td>
<td>0.000</td>
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<td></td>
<td>50%</td>
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</tr>
</tbody>
</table>

Acknowledgments

The authors would like to thank Caja Madrid and Rioja for financial support. We also acknowledge the helpful comments of reviewers.

References

TRANSPORT AND PARTITIONING PROCESSES OF DPBD AND TRICLOSAN INTO AND WITHIN FLOUR AND RICE

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2 Fraunhofer Institut fur Verfahrenstechnik und Verpackung, Gipf-Hohenaustr. 35, D-93054, Germany
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Introduction

The determination of a specific migrant into a food simulant or food system is often a complex task due to technical problems, chemical degradation or volatilization or the reliability of an appropriate analytical method. Therefore, in order to minimize the number of experimental tests and to check the compliance of a plastic food contact material with the existing legislation, the European Union has recently accepted the theoretical migration estimation as an alternative to lab testing which allows ensuring food safety (EU2002/72/EC).

The aim of this work is to study the transport and partitioning processes of DPBD and triclosan into and within flour and rice at different conditions. Diffusion coefficients were calculated according to the Model equation (1989) `[1/2]`

\[
Q = A - \sqrt{\frac{1}{2}} \int \left[ \frac{1}{E} \right] \left[ \frac{K}{E} \right] \left[ \frac{c}{E} \right] dE
\]

Materials and methods

Partition tests

1. Flour and Rice were processed in the same way. (both the representative of dry food with different particle size).

2. One of the surfaces of a ring (3 mm thickness and 0.4 cm internal surface) was in contact with a 1.186 g/m² contaminated plastic.

3. Five rings were overlapped and filled with the foodstuff.

4. Samples were wrapped with aluminium foil and put into a plastic bag which was vacuum sealed with the aim of allowing a better contact sample-contaminated plastic and were stored at 25°C, 40°C, and 70°C. All analyses were conducted by duplicate.

5. Layers (corresponding to rings) were removed carefully. Samples were processed as for the migration tests.

Extraction Procedure (1-4)

Sample (rice or flour) was homogenized in a mixer (20 ml)

Chromatographic Conditions

- Column: Kromasil C18 (15 μm, 4.6 cm 250 mm x 4.6 mm, E 5 μm particle size, 35°C)
- Injection: 5 μl
- Gradient: 2 mins at 300 mm line, 260 nm

Results and Discussion

Diffusion profiles

- According to our results, migration of substances included in plastic to dry foods is not negligible. At 25°C and 40°C, DPBD has been detected at the third layer of the plastic film, while triclosan was only detected up to the second layer (9 mm of depth).
- There are many factors involved in the migration phenomenon; nevertheless, in this specific case the molecular weight (MW: 298 g·mol⁻¹ for DPBD and 209 g·mol⁻¹ for triclosan) is probably the most important and it can be needed with the solubility power. *MW* = *X* penetration power
- Migration levels found for flour were greater than for rice, for all studied conditions and in both model migrants. The most probable explanation is the higher particle size of the rice grains in comparison with wheat flour particles.

Diffusion coefficients

<table>
<thead>
<tr>
<th>Foodstuff</th>
<th>Migrant</th>
<th>Limit of detection (LC) (μg·g⁻¹)</th>
<th>Limit of quantification (LQ) (μg·g⁻¹)</th>
<th>Calculation of diffusion coefficients (log k)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Foodstuff</td>
<td>Migrant</td>
<td>Limit of detection (LC) (μg·g⁻¹)</td>
<td>Limit of quantification (LQ) (μg·g⁻¹)</td>
<td>Calculation of diffusion coefficients (log k)</td>
</tr>
<tr>
<td>Flour</td>
<td>DPBD</td>
<td>2.9391</td>
<td>6.3283</td>
<td>3.5821</td>
</tr>
<tr>
<td>Rice</td>
<td>DPBD</td>
<td>2.9391</td>
<td>6.3283</td>
<td>3.5821</td>
</tr>
<tr>
<td>Flour</td>
<td>Triclosan</td>
<td>2.9391</td>
<td>6.3283</td>
<td>3.5821</td>
</tr>
<tr>
<td>Rice</td>
<td>Triclosan</td>
<td>2.9391</td>
<td>6.3283</td>
<td>3.5821</td>
</tr>
</tbody>
</table>

References


Acknowledgments

The following is reprinted with permission from Food & Agriculture Organization of the United Nations, Rome, Italy.
Concentration profile studies of model migrants into/within foodstuffs - example of results

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Introduction
The essential aim within our project FOODMIGROSURE is to measure dependent concentration profiles of model migrants in the food along the migration direction from the source into the inner food compartment. Scientific investigations concerning the migration potential and behaviour of food packaging materials have demonstrated that diffusion in and migration from food contact materials are irreversible physical and, in principle, mathematically describable processes. Besides the contribution of the plastic packaging material itself, the expected key parameters controlling the extent of migration into foods are the diffusion processes and partitioning effects within and within-like food.

Overview of experimental procedures for the volatile migrant models (stylene, trimethylamine)
The experiments carried out for the studies of the concentration profiles into or within foodstuffs were carried out with a LDIPE closed foil. For the experiments realized with pork mince meat was prerinse added lard and the meat was homogenised in order to obtain meat with different fat contents. The food was put in contact with the spiked foil inside the migration cell at the established experimental conditions (23 ± 3°C/10 days and 5°C/10 days). Regarding the food properties, for some items it was necessary to freeze the samples after the migration to facilitate the cut. The slices were weighed, homogenised and then each slice a replica was prepared. 1g of contaminated lard was directly weighed into the headspace vial and 20 µl ethanol were added. The vial was closed and analysed by HS-GC.

1-Volatiles Temperature Effect

Fig. 1: Concentration profile of styrene into collage cheese at 5°C and 23°C

Fig. 2: Concentration profile of octane into soft cheese at 5°C and 23°C

2 - Volatiles Fat Content Effect

Fig. 3: Concentration profile for trimethylamine in minced pork meat with different content of lard added after 5 days at 23°C. The free plane thickness is approximately 1mm

3 - Non Volatiles Behaviour in Different Foodstuffs

Experimental procedure for the non volatile migrant model (ATBC)
The experimental procedure for the concentration profiles carried out with ATBC was similar to the one carried out with the non volatile substances. The significant difference was the need to extract the samples with acetone/dichloromethane (1:1) after the migration period. The samples were measured by GC-MS.

Conclusions
After carrying out several experiments with various model migrants and at different temperatures the results have showed that the migrant concentration is increasing with the temperature increase. In figure 1 and 2 it is shown that migrant concentration measured at 23°C are higher than at 5°C. Experiments carried out with minced pork meat have shown that migration is lower in meat with less fat content. The migrant concentration is increasing with the addition of lard as represented in figure 3. Results provided from the experiments executed with the non volatile migrant (ATBC) (figure 4) do demonstrates that for the different foodstuffs analysed the migration processes occur mainly at the contact superficial area. One reason for that could be the food consistency. In soft foodstuffs like chocolate spread measured values are higher. Also the molecule size of ATBC can influence and difficult the penetration into the inner food compartments.

For more information about this project visit the website: www.foodmigrosure.com

Acknowledgments
The Foodmigrosure project is supported by the European Community under the 6th Framework Programme (QLK1-CT2002-2390). Contact person: Roland Franz roland.franz@fraunhofer.de

ja butter / iaf food nutrition and health
Transport and partitioning processes of model migrant Chimassorb 81 into and within food

Introduction
FOCOMIGROSURE project (EU Contract No. QLK1-CT2002-2390) aims to develop a physico-chemical migration model that describes mathematically the migration processes from polymers into foodstuffs. To achieve this aim a significant amount of experimental work has been completed measuring the diffusion of model migrant CB1 through a selection of foodstuffs.

Materials and methods
The experiments in this work package were carried out using the Test Cell II (TCLC Peru) remaining CB1 as shown in WP1. Some experiments were conducted in duplicates using the apparatus shown above, which assure 0.5 d.m. of the test film is the foodstuff. The large bulge at the top of the construction was lighter to ensure sealing of the cell against the test film. The three small bolts on the cell were tightened to apply a light pressure ensuring good contact of the test film with the food. The food was exposed to the test film for the specified time given in the table.

<table>
<thead>
<tr>
<th>Product Type</th>
<th>Temperature</th>
<th>Times</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filter</td>
<td>40°C</td>
<td>10 days</td>
<td>Single sided cell</td>
</tr>
<tr>
<td>Milk powder</td>
<td>40°C</td>
<td>10 days</td>
<td>Single sided cell</td>
</tr>
<tr>
<td>Cooking oil</td>
<td>5°C</td>
<td>10 days</td>
<td>Pre-sliced food</td>
</tr>
<tr>
<td>Bacon</td>
<td>5°C</td>
<td>10 days</td>
<td>Pre-sliced food</td>
</tr>
<tr>
<td>Sesame seeds</td>
<td>5°C</td>
<td>10 days</td>
<td>Pre-sliced food</td>
</tr>
<tr>
<td>Cheese Milar Cheddar</td>
<td>20°C</td>
<td>20 days</td>
<td>Pre-sliced food</td>
</tr>
<tr>
<td>Cheese Milar Cheddar</td>
<td>24°C</td>
<td>20 days</td>
<td>Pre-sliced food</td>
</tr>
<tr>
<td>Gouda</td>
<td>20°C</td>
<td>10 days</td>
<td>Pre-sliced food</td>
</tr>
<tr>
<td>Gouda</td>
<td>20°C</td>
<td>10 days</td>
<td>Pre-sliced food</td>
</tr>
<tr>
<td>Philadelpia</td>
<td>5°C</td>
<td>20 days</td>
<td>Single sided cell</td>
</tr>
<tr>
<td>Margarine</td>
<td>5°C</td>
<td>30 days</td>
<td>Boiled in water</td>
</tr>
<tr>
<td>Mayonnaise</td>
<td>20°C</td>
<td>20 days</td>
<td>Single sided cell</td>
</tr>
<tr>
<td>Mayonnaise</td>
<td>5°C</td>
<td>30 days</td>
<td>Single sided cell</td>
</tr>
<tr>
<td>Smoked salmon</td>
<td>5°C</td>
<td>10 days</td>
<td>Pre-sliced food</td>
</tr>
</tbody>
</table>

Examples of results
The test data were submitted in a standardized format to partner C0 for exploitation and derivation of the key parameters D. and Kc.

Diffusion of CB1 in Nutella at two different temperatures

Diffusion of CB1 in mayonnaise at two different temperatures

Acknowledgments
The FOCOMIGROSURE project was supported by the European Commission within the IST program (project no. QLK1-CT2002-2390). The authors thank all collaborators and partners for their contributions.
WP 5: Development, verification & validation of the migration model
Introduction

Aim of work package 5 was to establish on the basis of the experimental data with kinetic migration experiments and concentration profile measurements from work package 3 an advanced migration model for the migration of low molecular weight compounds from plastic materials into foodstuff by adapting the validated diffusion model that exists for food simulants.

In order to accelerate the evaluation of the time dependent (kinetic) migration experiments an "inhouse fitting tool" (software) for the determination of the diffusion coefficient \( D_0 \) in the food (F) and the partition coefficient \( K_{PF} \) between polymer (P) and food was developed by FABES.

Evaluation of time dependent migration experiments: "Fitting tool" by FABES

The results of the time dependent migration investigations of work package 3 as well as additional input data required for their evaluation (e.g. density and thickness of polymer) were summarised in standardised experimental data sheets.

Experimental data sheet

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>1.23</td>
</tr>
<tr>
<td>Thickness</td>
<td>0.5cm</td>
</tr>
</tbody>
</table>

These data are imported into the inputarea of the "fitting tool" and a trend line (see figure below white line) in between the experimental data points is calculated according to the Gaussian method of compensation:

\[ y = ax + bx^n + cx^{-n} \]

Input mask of "fitting tool"

On the basis of these input data three types of calculation tools are available:

1) Manual fit

The computed migration curve can be manually adapted to the trend line by variations of the apparent diffusion coefficient \( D_0 \) and the partition coefficient \( K_{PF} \) (visual fit).

2) Upper fit

Initial values at the beginning of the Upper fit are a small value of the partition coefficient \( K_{PF} \) (\( \approx 1 \)) and a high value of the diffusion coefficient \( D_0 \) (\( \approx 10^{-10} \) cm²/s). The fitting procedure starts with the stepwise increase of \( K_{PF} \) followed by a stepwise alternating increase or decrease of the apparent diffusion coefficient \( D_0 \) and partition coefficient \( K_{PF} \). The calculated curve is adapted to the trend line by the distance between calculated curve and trend line is minimized. The calculated curve (see figure above yellow line) must be located above the trend line.

3) Lower fit

The calculation of the Lower fit starts with a small value of the partition coefficient \( K_{PF} \) (\( \approx 0,1 \)) and a small value for the diffusion coefficient \( D_0 \) (\( \approx 10^{-12} \) cm²/s) followed by the increase of \( D_0 \) and the alternating increase or decrease of the apparent diffusion constant \( D_0 \) and partition coefficient \( K_{PF} \). The lower migration curve is adapted to the trend line in consecutive calculation steps until a defined minimum distance (1.00 of the highest experimental points) between calculated curve and trend line is reached. The calculated curve (see figure above green line) must be located below the trend line.

The Lower fit might result in smaller \( D_0 \) and \( K_{PF} \) values compared to the Upper fit.

Reliability of data

Depending on the chosen time and temperature conditions of the migration experiment the packaging/food system reaches the equilibrium value or not. Based on this test more or less defined \( D_0/K_{PF}/D_0 \) systems were observed.

1) Defined \( D_0/K_{PF}/D_0 \)-System

Upper and Lower fit with the same result: Ítsporo cheese, \( D_0 = 3.4 \times 10^{-7}, K_{PF} = 49 \)

- measurement in equilibrium
- high amount of substance migrated so far
- reliable data
- 1 possibility of curve progression

2) Undefined \( D_0/K_{PF}/D_0 \)-System

Upper fit: Ítsporo cheese, \( D_0 = 7.0 \times 10^{-9}, K_{PF} = 6.7 \)

- measurement is not yet balanced
- small amount of substance migrated so far
- unreliable data
- > 1 possibility of curve progression

The following figure shows concentration profiles for a pair of variants of an undefined \( D_0/K_{PF}/D_0 \)-System.

Undefined \( D_0/K_{PF}/D_0 \)-System expressed as concentration profile

This figure classifies that in case of an undefined \( D_0/K_{PF}/D_0 \)-System a corresponding concentration profile is necessary to determine the correct \( D_0 \) and \( K_{PF} \) value.

For more information about this project visit the website:

www.foodmigrosure.com
Contact person: retinal2@tigex�esxhux.de
Introduction

The aim of work package 5 was to establish the basis of the experimental data set (kinetic migration experiments and concentration profile measurements) from work package 4 as an advanced migration model for the migration of low molecular weight components from plastic materials into foodstuff by adapting the validated diffusion model that exists for food simulants.

After the determination of the diffusion coefficients $D_i$ of the migrants in the food $D_i$ and the partition coefficients $K_{hi}$ between polymer $P$ and food the diffusion and partition behaviour of the contaminants was analysed regarding eleven different food categories.

### Determination of the diffusion coefficients $D_i$ and partition coefficients $K_{hi}$

**Time dependent migration experiment:**
- 3076 in Gouda cheese, 50°C/C
- $D_i = 7.4 \times 8.1 \times 290$

**Concentration profile:**
- Strain in Gouda cheese, 50°C/C
- $D_i = 3.2 \times 8.1 \times 1.6$

### Summary of $D_i$ and $K_{hi}$ values in Gouda cheese

<table>
<thead>
<tr>
<th>Sample</th>
<th>$D_i$ (10⁻⁹m²/s)</th>
<th>$K_{hi}$</th>
<th>$S_i$</th>
<th>$T$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cheese</td>
<td>122</td>
<td>0.12</td>
<td>0.12</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>122</td>
<td>0.12</td>
<td>0.12</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>122</td>
<td>0.12</td>
<td>0.12</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>122</td>
<td>0.12</td>
<td>0.12</td>
<td>40</td>
</tr>
</tbody>
</table>

### Statistical evaluation of partition coefficients $K_{hi}$

For statistical evaluation the partition coefficients $K_{hi}$ were logarithmised and plotted against the log octanolwater partition coefficient $K_{ow}$ (see figure below) or against the molecular weight (dry body). For each foodstuff the regression line is between the data points as well as the 95% confidence level was calculated (see figure below).

**Log $K_{ow}$ vs. Log $K_{hi}$ - diagram for cheese products at 20-25°C**

For each food category the "Upper-Level" safety limit of Log $K_{hi}$ was calculated (see figure below).

### Calculation of the "Upper-Level" safety limit for cheese products at 20-25°C

For each food category the "Upper-Level" safety limit of Log $K_{hi}$ was calculated (see figure below).

### Statistical evaluation of diffusion coefficients $D_i$

The activation energy of the apparent diffusion process in foods was determined by use of the Arrhenius type equation:

$$D = D_0 e^{E_a/R T}$$

The diffusion coefficients of each migrant were logarithmised and plotted against $1/T$.

**Ln $D_i$ vs. 1/T - diagram for gouda cheese**

From this an average activation energy was derived and applied for the calculation of the food specific constant $A_i$.

### Food specific constants $A_i$ for cheese products

$$A_i = \ln D_0 + \frac{E_a}{RT}$$

For more information about this project visit the website: [www.foodmigrosure.com](http://www.foodmigrosure.com)

Acknowledgments

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**Work package 5: Migration model**

**R. Brandsch**, P. Mecenas, A. Zülich, O.-G. Piringer, V. Tosa

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2. National Institute for R&D of Isotopic and Molecular Technology, 3400 Cluj Napoca, Romania

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**Introduction**

The aim of work package 5 was to establish on the basis of the experimental data set (kinetic migration experiments and concentration profile measurements) from work package 3 an advanced migration model for the migration of low molecular weight components from plastic materials into foodstuffs by adapting the validated diffusion model that exists for food stuffs.

**Partitioning and Diffusion Processes**

The migration process from a polymeric material $P$ in contact with food $F$ (parallel arrangement) is characterized by the diffusion and partition coefficient of the migrants.

- **Diffusion coefficient $D_p$**
  - Arrhenius equation
  - $D = D_0 \cdot e^{-E/A}$

- **Partition coefficient $K_{PF}$**

**Numeric algorithms**

- One of the layers might be food.

**Discretisation times $t$**

**Discretisation distance $x$**

**Calculation with Migrant Exp.**

- $c_x = \frac{c_0}{1 + x K_p}$

**D/K-Model for polymer in contact with simulant $S$**

- Homogeneous or heterogeneous state
- Well-mixed liquid (diffusion of polymer much faster than in simulants)
- No boundary resistance
- No sorption of polymer
- Prior to migration in case of ingested food items products remain constant over time

**D/K/D-Model for polymer in contact with food $F$**

- The transfer of migrants from food contact materials into foodstuffs is influenced by the diffusion in the packaging material $D_p$ as well as the diffusion in the foodstuff $D_f$.

**Migration model for partition and diffusion processes in foods (20-25°C)**

- **“Upper-Level” Safety Limit for $K_{PF}$-values**
- **“Upper-Level” Safety Limit for $A_q$-values**

**Liquid foods**

**Processed vegetables and fruits**

**Dry foods**

**Milk products**

**Meat products**

**Grease products**

**Margarine/Mayonnaise**

**Chocolate products**

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For more information about this project visit the website:
www.foodmigrosure.com

Contact person: reinald.brauchle@izp-izki.de
WP 6: Demonstration of representativeness, workability, applicability & validation of the migration model for exposure estimations
Introduction

A migration translation tool was to correlate food simulants migration data with foodstuff migration data generated in work package 3. A migration translation tool is generated for the correlation of any food simulant migration data obtainable from various available migration data bases with food migration at any conditions of interest.

D/K/D-Model: Migration Model for Food (MMF)

- Migration in polymer P and in food F follows the law of diffusion
- Physical-chemical parameters D and K describe the migration process in the food (C₀) in the polymer (Cₕ₀) and the partitioning process between polymer and food (Kₓ₀)

Migration translation tool (MTT)

The food simulant migration data to be translated into food migration values need to be given "quality" in terms of information related to the food contact material FCM as well as the testing device and procedure used for determination of the experimental migration values into the food simulant. Based on the quality of information available to mimic the migration experiment into the food simulant the following cases can be identified:

Case A: Initial concentration of migrant in FCM c₀ and diffusion coefficient of migrant in FCM D₀ and partition coefficient between FCM and food simulant Kₓ₀ are known
- substitute Kₓ₀ with Kₓ₀ and calculate migration into food with MMF
- derive unknown value from migration data with food simulant by applying migration model for food simulants MMS
- substitute Kₓ₀ with Kₓ₀ and calculate migration into food with MMF

Case B: one of c₀, D₀, Kₓ₀ unknown
- make reasonable assumptions concerning one of the unknown values
- derive second unknown value from migration data with food simulant by applying migration model for food simulants MMS
- substitute Kₓ₀ with Kₓ₀ and calculate migration into food with MMF

Case C: Two of c₀, D₀, Kₓ₀ unknown
- make reasonable assumptions concerning one of the unknown values
- derive second unknown value from migration data with food simulant by applying migration model for food simulants MMS
- substitute Kₓ₀ with Kₓ₀ and calculate migration into food with MMF

Example for Case A

Time-dependent migration of Casperlactose from Nylon (PA6) into water (liquid) at 60°C: 
- Dₓ₀ = 5.40 x 10⁻¹⁰ m²/s, Kₓ₀ = 0.5
- c₀ = 500 mg/kg
  - Migration calculation (water) 40°C: 
  - Dₓ₀ = 5.40 x 10⁻¹⁰ m²/s, Kₓ₀ = 0.5
  - c₀ = 500 mg/kg

Example for Case C

One point measurement resulting in case C: Dₓ₀ and Kₓ₀ are unknown, c₀ is known

Migration data of Tricosan from HDPE film into 3% acetic acid:

Experimental migration mₓ₀ = 2.35 mg/dm²

→ Reasonable assumptions for Dₓ₀

From statistical evaluation of the polymer specific constants Aₓ for HDPE (determined in prior migration investigations) an upper limit Aₓ of 14.5 and a mean value Aₓ of 11.20 for HDPE can be derived (see figure below)

- Calculation of mₓ₀ (2.35) of Tricosan in HDPE by applying MMS (see above or mean Aₓ = value 11.20 (Dₓ₀ = 1.07 x e⁻¹⁰ cm²/s)

→ Reasonable assumptions for Kₓ₀

For the same FCM (c₀ = 1100 mg/kg) one point measurements with the fatty food simulant HB 307 at 40°C are available (Pₓ₀ of 0.37 mg/dm²). Due to good suitability of Tricosan in HB 307 a partition coefficient of Kₓ₀ = 1.5 can be reasonably assumed.
- Calculation of Dₓ₀ = 4.8 x e⁻¹⁰ cm²/s by applying MMS on the basis of Kₓ₀ = 1.5

⇒ Dₓ₀, for Tricosan in HDPE (both assumptions) ≈ 1 x e⁻¹⁰ cm²/s

Kₓ₀, for Tricosan HDPE/HB 307 = 1 and HDPE/3% acetic acid = 36

→ Substitute Kₓ₀ with Kₓ

For gouda cheese a Tricosan TRCₜ diffusion coefficient of 1.6 x e⁻⁸ cm²/s at 40°C and a partition coefficient between polyethylene and food of Kₓ = 3.6 at 40°C was determined from experimental data of WB 305.

Calculation of Tricosan migration after 10 days at 40°C into gouda cheese by use of MMT, Pₓ₀ = 0.37 mg/dm²

Calculation of Reduction Factor Z for Tricosan into gouda cheese: 
- From migration calculation: 
  - 0.5 x 3.6 x 10⁻¹⁰ m²/s, Kₓ₀ = 0.37 mg/dm²

According to directive 80/777/EEC: Reduction factor for HDPE migration at 40°C: 36

317

Acknowledgements:

For more information about this project visit the websites:

www.foodmicrosure.com

Contact person: roelia@kbb.kaub.de
WP 7: Investigation of Consumer attitude towards modeling
Consumer perception investigation on migration modelling:
Large scale questionnaire polling approach

By: The JRC Contact Materials/Migration Processes Group
European Commission DG Joint Research Centre, Institute for Health and Consumer Protection. Physical and Chemical Exposure Unit, Ispra

Introduction
The workpackage 7 of the project foodmigration was to investigate the perception of the consumer to predictive modeling to simulate migration. This posed a number of challenges since packaging is not perceived as a source of risks in relation and consequently the issue has never been considered in any former EU risk perception or communication projects, and neither did the most recent Eurobarometer polling large scale study on risk perception performed by the European Food Safety Authority (EFSA) published in February 2006.

We therefore considered for one of the approaches directed at the citizen to take advantage of an Open Day at the JRC on May 13 2009 which welcomed about 3000 in total to conduct a large scale citizen perception study.

Our goal therefore became three-fold, because the topic of food packaging safety is not perceived by the public, we had a part of risk education and communication. Because it was the open day, we had a goal of exhibiting in simple terms the topic of relevance and the means for doing it like the JRC instances. And because of the highly localized nature of the foodmigration project we had to test people on their perception of mathematic modeling as a mean to simulate and predict migration from food contact materials.

Experimental
We developed a questionnaire based on the one used by EFSA and questions raised in the EU TRUST project.

Because the topic of packaging is quite unknown to the citizen, we prepared visual stimuli with an art piece made of packages (Figure 1), and posters (Figure 2). Visitors were then given a tour programme (Figure 3) that explained our goal within the project in addition. The tour was of a total of 30 min and about 5 min to compile the questionnaire (Figure 4). To quickly and entertainingly illustrate our work on safety of food packaging, we developed a short movie (Figure 5) to educate the public on the general risk issues and tests associated with packaging. The resulting video (12 min) was shown followed by a visit of 3 of the laboratories for visual impact where the same scientists were welcoming visitors and answering questions. The questionnaire was given upon exiting and completed questionnaires were rewarded with a small gadget.

A test trial was run on consumer associations with 35 representatives of the Lombardia region.

The experiment was then conducted on citizens at full scale during the JRC Open Day which was highly publicized regionally (Figure 5) in total. The event also involved the presence of the consumer association representative. Questionnaires and comments were collected for 700 units which represented about 1400 visitors to the food contact activities.

Acknowledgments
The Foodmigration is supported by the European Commission Research Framework Programme VI. The authors would like to thank the JRC’s JAC platform for their support and the participants.

For more information about this project visit the website:
www.foodmigration.com
Contact person: Mariam Danel@jrc.ispra.it

Contact person: Roman Bauschke@de
Consumer perception investigation on migration modelling:
Large scale questionnaire polling approach
By: The JRC Contact Materials/Migration Processes Group
European Commission DG Joint Research Centre, Institute for Health and Consumer Protection, Physical and Chemical Exposure Unit, Ispra

For more information about this project visit the website:
www.foodmigrosure.com
Contact person: oliver.foerster@jrc.ec.europa.eu

Acknowledgments
The authors and this workshop would like to thank the participants for their contribution and enthusiasm.

Yard and Entrance
Capacity was 40 visitors per 15 min. For combination 1 lane

Compilation of questionnaires. Representatives of consumer authorities with their patients or forms to used other audiences were present and involved.

The action of the study

INGRESS
ENTRANCE
Consumer perception investigation on migration modelling: Large scale questionnaire polling approach

By: The JRC Contact Materials/Migration Processes Group
European Commission DG Joint Research Centre, Institute for Health and Consumer Protection, Physical and Chemical Exposure Unit, Ispra

Results

The questionnaire then gave a choice of a number of reasons commonly used regarding the relevance of the use of such computer simulations. People had to rank each reason as a function of personal perceived importance.

The answers highlighted that consumers felt that migration modelling was most relevant as a value added tool to point out more quickly worst cases and as support to science.

This attitude echoed also consumer associations and focus groups for which Q&A or focus sessions showed they felt having no source for information and being open to exchanging more in the media and lack of communication from official national sources. Therefore a source of positive feeling from migration modeling seemed to be the help to faster science communication and consumer (unbiased) reassurance.

Responses to the questionnaire

In terms of importance for internalization of the packaging when shopping for food, people consider safety the most important, followed by resistance and convenience as important. Shape and color were not considered important.

On the question of food packaging safety evolution over the last 10 years, the answer was that safety was considered by 100% of the people better or much better than 10 years ago.

On the question of a case of a problem associated with packaging and whom who one trusts for information, the answer showed that people would like to receive relevant safety related information primarily from scientists, followed by public authorities and newspaper/TV, followed by consumer associations and last by food producers or supermarkets.

On the question of an opinion on the use of modelling as helping tool to investigate safety food packaging, almost 90% people were either favourable or strongly favourable to modelling when they understood what it meant: only 3% were against.

Some comments showed that people inherently trusted the simulations because they assumed or knew that scientists would have tested the model compared with experimental data. Ranking "predictably favourable" seemed to indicate a lack of certainty on the relation between experimental data and modelling.

The data also reflected comments obtained by focus groups, which was completely different approach with no prior risk education.

People in the overwhelming majority both for this questionnaire approach and for the focus group approach felt much reassured regarding the safety of packaging simply from the fact that they did not previously know that such research and controls existed. Most spontaneous written comments were to have this type of research much more visible at the level of both consumer associations and consumers themselves. People were also extremely enthusiastic and grateful for experiencing an entertaining science about producers and made enthusiastic comments on the humanizing and added-value the initiative represented for the consumer’s understanding of science.

Conclusions

The responses were echoing quite interestingly many answers also contained in the focus group, although a completely different methodology. There is a fundamental trust from the public in the scientists to distinguish and understand safety issues. The consumer wants sincerely to be approached and informed by scientists for this reason and is also ready to favor new approaches such as migration modelling if it can be an additional tool for better consumer protection. However, the consumer needs to be sure that at the root for use are experimental data which demonstrate the applicability of the model.

For more information about this project visit the website: www.foodmicrosure.com
Contact person: Roland Rasen@ec.europa.eu

Acknowledgments
The contributions have been received from several of the consumers and national experts. The information is given in the table below.
Snapshots of the event

The EU project researchers and conference speakers
Opening by Dr. D. Kotzias, head of the Physical and Chemical Exposure Unit, on behalf of the IHCP Director Mrs. E. Anklam.

Introduction to the project by R. Franz, project co-ordinator

Introduction to the project by D. Bennink, project RTD Officer

Presentation of WP1, choice of substances, by I. Copper

Presentation of WP2, choice of foodstuffs, by I. Steiner

Presentation of WP3a, method of analysis for migrants, by P. Paseiro

Presentation of WP3b, kinetics of migrants in foods, by R. Franz

Presentation of WP4-5, Diffusion and Partition by R. Bandsch

Presentation of WP7, Consumer attitudes, by C. Simoneau

Presentation of relations to legislation on Food Contact, by A. Schaefer
Presentation of comparison to FDA research, by T. Begley

Presentation of exposure assessment for the consumer, by P. Oldring

Presentation of implications of the project for the food industry, A. Mandanis, Nestlé

Presentation of implications of the project for the National Reference Laboratories, X. Trier Thorsager, FDIR

Presentation of implications of the project for the chemical industry, C. Gueris, CEFIC

Presentation of implications of the project for the converters’ industry, G. Tillieux, PlasticsEurope

Presentation of implications of the heritage of other EU project used in the consumer attitude WP, L. Pellizoni, TRUST project.

Presentation of future EU projects in the frame of FP7, D. Bennink, DG RTD
The room

The posters and poster break

Questions and answers
The conference Dinner
*The event was sponsored by Partner 08 Nestlé*

The conference Lunch
*The event was sponsored by Partner 09 CEFIC*
Snapshots of the dinner.....
The JRC conference team who made it possible
The project research team

The project team and conference speakers
The conference was organised by the Joint Research Centre for the European Commission and its Community Reference Laboratory for Food Contact Materials.

Information:

Catherine Simoneau catherine.simoneau@jrc.it
Satisfaction survey

**CHOICE OF LOCATION**

- Poor: 11.3%
- Average: 88.7%
- Good: 0.0%
- Very good: 0.0%

**EVENT ORGANIZATION**

- Poor: 19.6%
- Average: 79.4%
- Good: 0.0%
- Very good: 0.0%
- No answer: 0.0%
INFORMATION PROVIDED ABOUT THE EVENT

- Poor: 32.0%
- Average: 3.1%
- Good: 13.4%
- Very good: 51.5%
- No answer: 0%

MATERIALS PROVIDED WITH THE EVENT

- Poor: 44.3%
- Average: 4.1%
- Good: 7.2%
- Very good: 44.3%
- No answer: 0%
HOW USEFUL ARE THE RESULTS, PRESENTATIONS AND POSTERS FOR YOUR FIELD?

METHODS OF ANALYSES

- Little useful: 35.1%
- Somewhat useful: 4.1%
- Useful: 15.5%
- Very useful: 45.4%
- No answer: 4.1%

HOW USEFUL ARE THE RESULTS, PRESENTATIONS AND POSTERS FOR YOUR FIELD?

MIGRATION MODELLING

- Little useful: 37.1%
- Somewhat useful: 1.0%
- Useful: 7.2%
- Very useful: 54.6%
- No answer: 7.2%
HOW USEFUL ARE THE RESULTS, PRESENTATIONS AND POSTERS FOR YOUR FIELD?

**MIGRATION PHENOMENA**

- Little useful: 46.4%
- Somewhat useful: 4.1%
- Useful: 18.6%
- Very useful: 51.5%
- No answer: 28.9%

**RISK COMMUNICATION**

- Little useful: 28.9%
- Somewhat useful: 1.0%
- Useful: 18.6%
- Very useful: 51.5%
- No answer: 46.4%
ARE YOU

- **Gov. NRL**: 27.8%
- **Industry / Prof. Assoc.**: 4.1%
- **Contract Lab / Inst. / Academia**: 26.8%
- **Other**: 1.0%
- **No answer**: 40.2%
Abstract
The closing conference of the EU project Foodmigrosure project gathered 140 participants from both the EU as well as Norway, Switzerland, US, Japan, Thailand etc. The objective of the project was to develop a normative predictive tool to assess safety of food contact materials. The scientific conference exhibited the results of comprehensive sets of migration data for a wide range of substances and large sets of various foodstuffs. The data obtained allowed to validate a migration models on real foods both for compliance as worst case or refined for exposure assessment. The results also showed aspects of food chemistry that can influence migration, and initiated a new area of public risk perception specific to food contact materials safety. These systematic kinetics studies have a direct impact on Directive 85/572/EC on correspondence factors of foodstuffs and food simulants; The results obtained also show an impact on 2002/72/EC on allowing the use of mathematical modelling, a the data is used to validate the current diffusion model for further applications; in addition The project shows an impact on risk assessment from systematic kinetics of migration related to real foods in different conditions for exposure assessment (EFSA), as well on standardisation (CEN) from the development of methods in foodstuffs. The conference was organised both around keynote speakers of the different workpackages as well as in day external speakers from different branches of stakeholders exemplifying the added value of the project for their field. The participants were evenly distributed between industrial stakeholders (40%), National Reference Laboratories/Government (30%), and institutions, associations, academia etc (30%).
The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.