FEICA WEBINAR

FEICA testing project on hotmelts for food packaging

17 March 2021
15:00 - 16:00 Brussels CET
Proceedings

- Please be advised that this webinar will be recorded for internal use only. By joining, you are consenting to the recording.

- We ask participants to turn off their camera to avoid system performance issues.

- Note that you will be muted upon entry. During the Q&A session following the presentations, you will be able to unmute or use the chat box to ask questions.

- In case we don’t have sufficient time during the Q&A session to address your question, please feel free to send your question to info@feica.eu.

- The presentation slides will be sent to all webinar registrants.
Speakers/Moderators

Jana Cohrs
Executive Director Regulatory Affairs, FEICA

Martin Lommatzsch
General Manager, Laboratory Lommatzsch & Säger

Matthias Frischmann
Food chemist and Head of Corporate Analytics, Henkel

Alexandra Ross
Product Regulatory Specialist EIMEA, H.B. Fuller
Agenda

15.00  Introduction to the testing project - Jana Cohrs (Executive Director Regulatory Affairs, FEICA)

15:10  Testing setup and results - Martin Lommatzsch (General Manager, Laboratory Lommatzsch & Säger)

15:30  Conclusion and Interpretation - Matthias Frischmann (Food chemist and Head of Corporate Analytics, Henkel)

15:45  Q&A -panel - Martin Lommatzsch, Matthias Frischmann and Alexandra Ross (Product Regulatory Specialist EIMEA, H.B. Fuller)

16.00  Close of the webinar, Jana Cohrs
Jana Cohrs
Executive Director Regulatory Affairs, FEICA

Introduction to the testing project
FEICA - Association of the European Adhesive & Sealant Industry

15 National Associations representing 16 countries +800 members

24 Direct Company Members

19 Affiliate Company Members
FEICA Technical Working Group Paper and Packaging

BASA
IVK
DETC
AFICAM
DFL
coim
Dow
H.B. Fuller
Bostik
Henkel
synthomer
EASTMAN
NYNAS
Ergon International, Inc.
BorsodChem
IMERYS Carbonates
Markets

The European adhesive and sealant market 2020
End-use sectors

€17.1bn

- Assembly operations/other: 22.5%
- Building construction, civil engineering, craftsmen: 18.2%
- Consumer/DoItYourself: 11.5%
- Footwear and leather: 2.2%
- Transportation: 9.2%
- Woodworking and joinery: 9.5%
- Paper, board and related products: 26.9%

Data source: Smithers
Mineral Oil Hydrocarbons (MOH) in Food

Some Regulations and legal approaches to MOH

- EUROPEAN UNION – no Regulation yet
  Joint Research Centre Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials

- GERMANY – Draft Regulation
  Obligation to use a functional barrier if food contact materials are made of recycled paper

- AUSTRIA & SWITZERLAND
  Regulation has been implemented: mandatory barrier if recycled paper is intended for food packaging
FEICA testing project on hotmelts for food packaging

Objectives of the project

- Explore the contribution of hotmelts to MOSH / MOAH fractions measured in food, despite no mineral oil being used in their formulation
- Demonstrate the safety of typical hotmelts intended for food packaging *
- Assist customers to avoid unsuitable testing set-ups and incorrect interpretation

* The hotmelts represented in this project are typical for the food packaging market. For guidance on choosing the right raw material please consult the FEICA guidance related to the food contact status of adhesives and mineral oil hydrocarbons: https://www.feica.eu/our-priorities/food-contact.
FEICA testing project on hotmelts for food packaging

What you can expect today

- Explanation on sample preparation and test set-up.
- Explanation on interpreting the peaks resulting from the tests.
  - Extraction: Severe overestimation of migration
  - Migration simulation: Predicts real migration with safety margin
- A rough correlation between extraction, simulation and testing on real foodstuff
Hotmelts in food packaging

- EU regulation 10/2011 assumes that 1 kg of food is wrapped in 6 dm² of packaging.
- However, adhesives are applied only on a small surface area of this packaging.
- In this project, 300g oat flakes were packed into a cardboard carton.
- The application area of the hotmelt between the overlapping cardboard layers of the carton was roughly 0.2 dm² which reflects a typical application.
Typical composition of a hotmelt

**Backbone polymer**
- ethylene-vinyl acetate (EVA), rubber, metalloocene Polyethylene (PE), poly-alpha-olefin (APAO), polyester, polyamide, etc.

**Wax**
- paraffin, micro-crystalline wax, synthetic wax

**Resin**
- ester resin, aliphatic & aromatic hydrocarbon resin

**Additives**
- Colorants, Fragrances, Antioxidants etc.
Martin Lommatzsch
General Manager, Laboratory Lommatzsch & Säger

Testing setup and results
**Testing setup - Hotmelt formulation**

**Overview ingredients according to Lommatzsch et al. (2015)**

<table>
<thead>
<tr>
<th></th>
<th>Hydrocarbon resins</th>
<th>Waxes</th>
<th>Polyethylene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Share</td>
<td>30-40%</td>
<td>25-35%</td>
<td>35%</td>
</tr>
<tr>
<td>Hydrocarbon Type</td>
<td>Resin oligomers</td>
<td>n-Alkanes</td>
<td>Polyolefin oligomers</td>
</tr>
<tr>
<td>Concentration</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Saturated HC ≤C25</td>
<td>5.000 – 150.000 mg/kg</td>
<td>&lt;100 – 20.000 mg/kg</td>
<td>200 – 500 mg/kg</td>
</tr>
<tr>
<td>Concentration</td>
<td>&lt;100 – 50.000 mg/kg</td>
<td>&lt;100 mg/kg</td>
<td>&lt;10 mg/kg</td>
</tr>
<tr>
<td>Aromatic HC ≤C25</td>
<td>Distinct humps</td>
<td>Sharp peaks</td>
<td>Sharp peaks + unresolved background</td>
</tr>
<tr>
<td>GC peak pattern</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
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### Overview ingredients according to Lommatzsch et al. (2015)

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<tr>
<td>Hydrocarbon Type</td>
<td></td>
</tr>
<tr>
<td>Resin oligomers (ROSH &amp; ROAH)</td>
<td></td>
</tr>
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<td>GC peak pattern</td>
<td>Distinct humps</td>
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- Hydrocarbon resins are the main source of hydrocarbon able to migrate via gaseous phase (≤C25)
- Resin oligomers show distinct humps in GC, which can be misinterpreted as MOSH/MOAH
- Other sources of hydrocarbons: Low-melting paraffinic waxes
  Additives based on mineral oils
## Testing setup - Hotmelt formulation

### Test specimens (Std 0-13)

<table>
<thead>
<tr>
<th>Hydrocarbon resins (30-40%)</th>
<th>Waxes (25-35%)</th>
<th>Polymer (35%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fully-hydrogenated C9 resin</td>
<td>Paraffin wax A (Melting point: 54 – 70°C)</td>
<td>Polyethylene</td>
</tr>
<tr>
<td>Partially-hydrogenated C9 resin</td>
<td>Paraffin wax B (Melting point: 70 – 90°C)</td>
<td>EVA</td>
</tr>
<tr>
<td>Fully-hydrogenated C5 resin</td>
<td>Synthetic wax (Melting point: &gt;90°C)</td>
<td></td>
</tr>
<tr>
<td>Fully-hydrogenated DCPD resin</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Partially-hydrogenated DCPD resin</td>
<td></td>
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</tr>
</tbody>
</table>
Testing setup - Extraction of hotmelts

- **Extraction of polymers (granulates/films)**
  - Solvent: n-Hexane
  - Conditions: 24h at 60°C

- **Extraction of adhesives, tackifier resins and waxes**
  - Most of them are soluble in n-hexane
  - 10-60 min ultrasonic bath
Testing setup - **Migration simulation**

- **Migration via gaseous phase**
  - According to EU 10/2011 or EN 14338:2003
  - Simulant: MPPO (Tenax 60/80 mesh)

- **Migration cell:**

- **Conditions:**
  - 10d at 40°C

- Extraction of Tenax with n-hexane
Testing setup - Storage test

- Indirect contact
- 8 dm² virgin fibre folding box closed with 0.3 g of hotmelt (0.2 dm²)
- 300g oat flakes
  - Blank check of food and cardboard
- Storage for 12 months at ambient temperature
Testing setup – Analysis of hydrocarbons

HPLC fractionation
- Saturates
  - 100% C6
- Aromatics
  - 35% DCM

Backflush
- 100% DCM

“MOSH”
“MOAH”

HPLC (NP) → GC-FID
Testing setup – Comparison of approaches

- Extraction test
- Migration test
- Storage test

Content of migratable substances → Simulated migration → Real migration

Estimation approach?
**Results – Migration simulation**

**Hotmelt matrix effect**
- Migration slightly increases with different waxes and polymer (factor 1.4 – 1.7)
- Hotmelt formulation of choice:
  - Synthetic wax (25%), polyethylene (35%), hydrocarbon resin (40%)
Results – Migration simulation

Formulation of choice:
- 25% synthetic wax
- 35% polyethylene
- 40% hydrocarbon resin
Results – Migration simulation and extraction

Units
Simulation: µg/dm²
Extraction: µg/g (ppm)

Only comparable for a mass/area ration of 1g/dm² hotmelt
Results – **Migration simulation and extraction**

Migration simulation

Extraction

Different order of magnitude (Zoom factor)

MOSH fraction

MOAH fraction
Results – Migration simulation and extraction

Migration simulation

Extraction

MOSH fraction

MOAH fraction
Results – Storage test

Order Magnitude (blank adjusted):

Saturated resin oligomers (MOSH fraction): 

\[ \approx 1 \text{ – } 3 \text{ mg/kg} \] for the tested hotmelt types

Aromatic/unsaturated resin oligomers (MOAH fraction):

\[ < 0.2 \text{ mg/kg} \] for the tested hotmelt types
Results – Storage test

Blank oat flakes

MOSH fraction

MOAH fraction

12 months

Saturated resin oligomers: ≈ 2 mg/kg

Std 6

Aromatic resin oligomers: ≤ 0.2 mg/kg
Results – Comparison

- Good correlation between calculated migration (simulation) and real migration for the tested hotmelt types
- Limiting factor: Contact area of the hotmelt adhesive (0.2 dm²)
Matthias Frischmann
Food chemist and Head of Corporate Analytics, Henkel

Conclusion and Interpretation
Conclusion - Aim of the study

- Hotmelt raw materials (mainly resins) contribute to MOSH and MOAH fractions, although not being mineral oil.

- Standard application investigated with model hotmelts:
  - Oat flakes stored for 12 months: real migration compared with migration simulation and extraction.
  - Influence on MOSH/MOAH analysis investigated by use of different polymers, resins and waxes.

- Standard MOSH/MOAH analysis performed:
  - LC-GC-FID method separates all hydrocarbons into MOSH fraction and MOAH fraction.
**Conclusion - Hotmelt preparation**

- Hotmelt raw material variation
  - Basic formulation: 35 % (w/w) polymer, 40 % (w/w) resin, 25 % (w/w) wax
  - Five resin types compared at 40 and 30 % (w/w) in formulation, combined with 25 and 35 % (w/w) of synthetic wax respectively
  - Two polymer types compared at 35 % (w/w) in basic formulation
  - Three wax types compared at 25 % (w/w) in basic formulation
Conclusion - Extraction test on hotmelt samples

- Direct extraction with n-hexane
  - Severe overestimation of migration (up to 50 times)
  - Raw materials of hotmelt formulations are partly dissolved when being exposed to organic solvents
  - Chemical properties of raw materials explain their dissolving (rather than their ability to migrate)
  - Extraction is no meaningful approach to estimate migration!
Conclusion - *Migration simulation of hotmelt films*

- **MPPO (Tenax® 60/80 mesh) used as a food simulant**
  - Ten days of migration at 40 °C (MPPO and hotmelt film) simulates real oat flakes storage in cardboard box (12 months at room temperature)
  - **Predicts real migration** with safety margin (up to factor 2.1)
  - Effectively integrates volatility of migrating compounds and matrix effects
  - **Preferred way to simulate real storage!**
Conclusion - Real migration: oat flakes storage

- Oat flakes stored in cardboard boxes (12 months at RT)
  - Hotmelt raw materials (resins and waxes) contribute to MOH fractions, although not being mineral oil:
    - MOSH fraction slightly increases over time
    - MOAH fraction at or below limit of detection

- Variation of resins investigated
  - Leads to variation in oat flakes migration results
  - Varies in same order of magnitude (< 50% from the mean)

- All model hotmelts are safe* for use in the tested application!
  * would meet BfR draft criteria concerning MOAH, if interpreted as mineral oil
Conclusion - Summary

- **Safe use of hotmelts** in cardboard packaging demonstrated
  - Storage of oat flakes for 12 months at room temperature
  - Impact of various resins and waxes on contribution to MOSH/MOAH fractions investigated

- **Calculation model** developed
  - Based on MPPO migration simulation (Tenax®, 40 °C, 10 days)
  - Predicts real migration (storage of dry foodstuff for 12 months at room temperature), based on indirect contact area with hotmelt
Conclusion - **Summary**

- Typical hotmelt raw materials are polymers, resins and waxes

  ➜ Raw materials **partly contribute to MOSH/MOAH fractions** in LC-GC-FID analysis

  ➜ However, raw materials are not mineral oil!

  ➜ **Further analytical techniques required and available (e.g. GCxGC-TOF-MS)** to distinguish hydrocarbons from real mineral oil vs. hydrocarbons from hotmelt raw materials
Q&A Panel

- Please use the chat box or raise your hand if you have a question
- Once the moderators call you out, please unmute yourself
- Lower your hand and mute yourself once your question has been answered
- Questions in the chat box will be covered as we go along

Martin Lommatzsch
General Manager, Laboratory Lommatzsch & Säger

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Food chemist and Head of Corporate Analytics, Henkel

Alexandra Ross
Product Regulatory Specialist EIMEA, H.B. Fuller
THANK YOU

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info@feica.eu