FEICA® The European voice of the **adhesive** and **sealant industry**

FEICA WEBINAR

# FEICA testing project on hotmelts for food packaging

17 March 2021

15:00 - 16:00 Brussels CET

# Proceedings

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- 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

**Matthias Frischmann**

Food chemist and Head of Corporate Analytics, Henkel

**Martin Lommatzsch**

General Manager, Laboratory Lommatzsch & Säger

**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

- AFICAM
- Danish Coatings and Adhesives Association
- AICCS
- FCIO Chemische Industrie Berufsgruppe Bauklebstoffe
- aseFca
- PZPFK
- FKS Fachverband Klebstoff-Industrie Schweiz
- Federchimica AVISA Gruppo Adesivi e Sigillanti
- Gospodarska zbornica Slovenije
- BASA
- Industrieverband Klebstoffe e.V. German Adhesives Association – Experience Perfect Services
- deTic
- SVEFF Sveriges Färg och Lim Företagare
- Deutsche Bauchemie
- VLK vereniging lijmen en kitten

## 24 Direct Company Members

- 3M
- Dow
- novatech
- SAPICI
- ac marca Adhesives
- DuPont
- Selena Global Experience
- Aerosol Service
- H.B. Fuller
- Sika Building Trust
- ARaymond
- Henkel
- Soprema
- Avery Dennison
- Huntsman Enriching lives through innovation
- Bolton Adhesives
- Krimelte Member of Wolf Group
- Soudal
- Bostik smart adhesives
- Lohmann The Bonding Engineers
- TKK
- coim
- Mapei
- CPC Construction Products Group Europe

## 19 Affiliate Company Members

- AGC
- Imerys Carbonates
- Nynas
- alpha wax
- Kaneka
- Huntsman Enriching lives through innovation
- Arlanxeo Performance Elastomers
- Lanxess Energizing Chemistry
- Stepan
- BorsodChem Chemistry for generations
- Mitsui Chemicals Group
- synthomer
- covestro
- MOL Group
- PCC Rokita
- Eastman
- Momentive
- Ergon
- Emerald Kalama Chemical



FEICA

# FEICA Technical Working Group Paper and Packaging

BASA | IVK | DETIC | AFICAM | DFL

coim

Dow

H.B. Fuller

Bostik smart adhesives

Henkel

synthomer

EASTMAN

NYNAS

Ergon International, Inc.

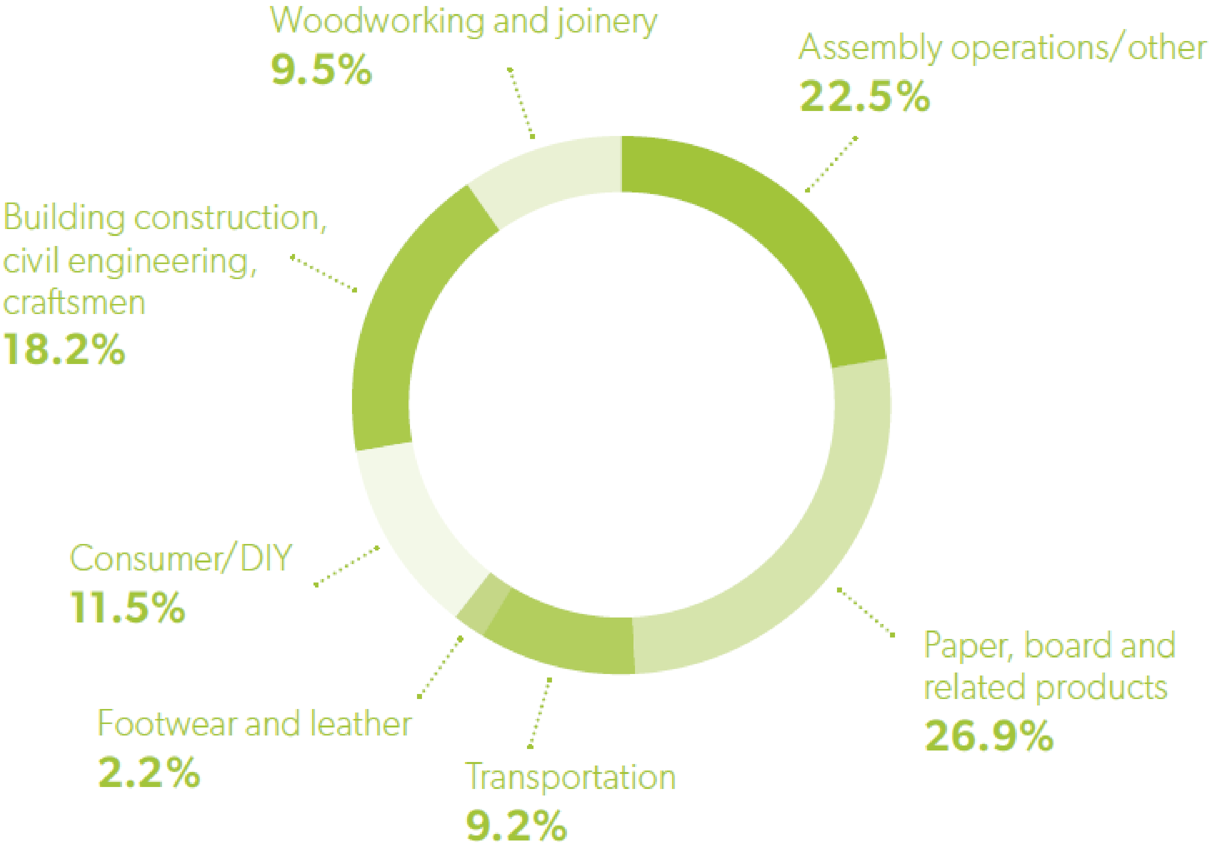
BorsodChem Chemistry for generations

IMERYS Carbonates

# Markets

## The European adhesive and sealant market 2020
## End-use sectors

**€17.1bn**




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 $dm^2$ 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 $dm^2$ which reflects a typical application.**

# Typical composition of a hotmelt

**Additives**

Colorants, Fragrances, Antioxidants etc

**Backbone polymer**

ethylene-vinyl acetate / EVA, rubber, metallocene Polyethylen / PE, poly-alpha-olefin (APAO), polyester, polyamide, etc.

**Wax**

paraffin, micro-crystalline wax, synthetic wax

**Resin**

ester resin, aliphatic & aromatic hydrocarbon resin

# Martin Lommatzsch

General Manager, Laboratory Lommatzsch & Säger

## Testing setup and results

# Testing setup - Hotmelt formulation

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

| | **Hydrocarbon resins** | **Waxes** | **Polyethylene** |
|---|---|---|---|
| Share | 30-40% | 25-35% | 35% |
| Hydrocarbon Type | Resin oligomers | n-Alkanes | Polyolefin oligomers |
| Concentration Saturated HC ≤C25 | 5.000 – 150.000 mg/kg | <100 – 20.000 mg/kg | 200 – 500 mg/kg |
| Concentration Aromatic HC ≤C25 | <100 – 50.000 mg/kg | <100 mg/kg | <10 mg/kg |
| GC peak pattern | Distinct humps | Sharp peaks | Sharp peaks<br>+ unresolved background |

# Testing setup - Hotmelt formulation

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

| | Hydrocarbon resins |
|---|---|
| Share | 30-40% |
| Hydrocarbon Type | Resin oligomers (ROSH & ROAH) |
| Concentration Saturated HC ≤C25 | 5.000 – 150.000 mg/kg |
| Concentration Aromatic HC ≤C25 | <100 – 50.000 mg/kg |
| GC peak pattern | Distinct humps |

- 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

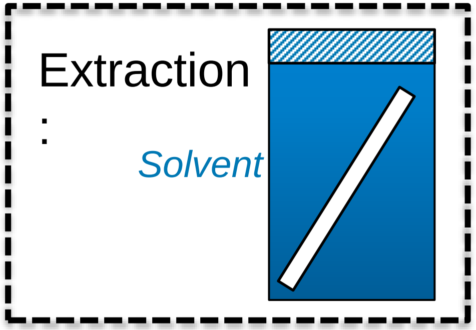
## *Test specimens (Std 0-13)*

| Hydrocarbon resins (30-40%) | Waxes (25-35%) | Polymer (35%) |
|---|---|---|
| Fully-hydrogenated C9 resin | Paraffin wax A<br>(Melting point: 54 – 70°C) | Polyethylene |
| Partially-hydrogenated C9 resin | Paraffin wax B<br>(Melting point: 70 – 90°C) | EVA |
| Fully-hydrogenated C5 resin | Synthetic wax<br>(Melting point: >90°C) | |
| Fully-hydrogenated DCPD resin | | |
| Partially-hydrogenated DCPD resin | | |

# Testing setup - Extraction of hotmelts

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

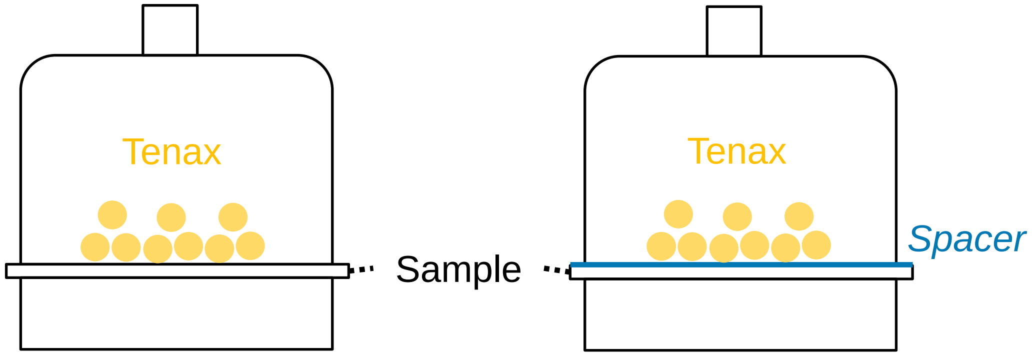
- **Extraction of adhesives, tackifier resins and waxes**
  - Most of them are soluble in n-hexane
  - 10-60 min ultrasonic bath



# 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:



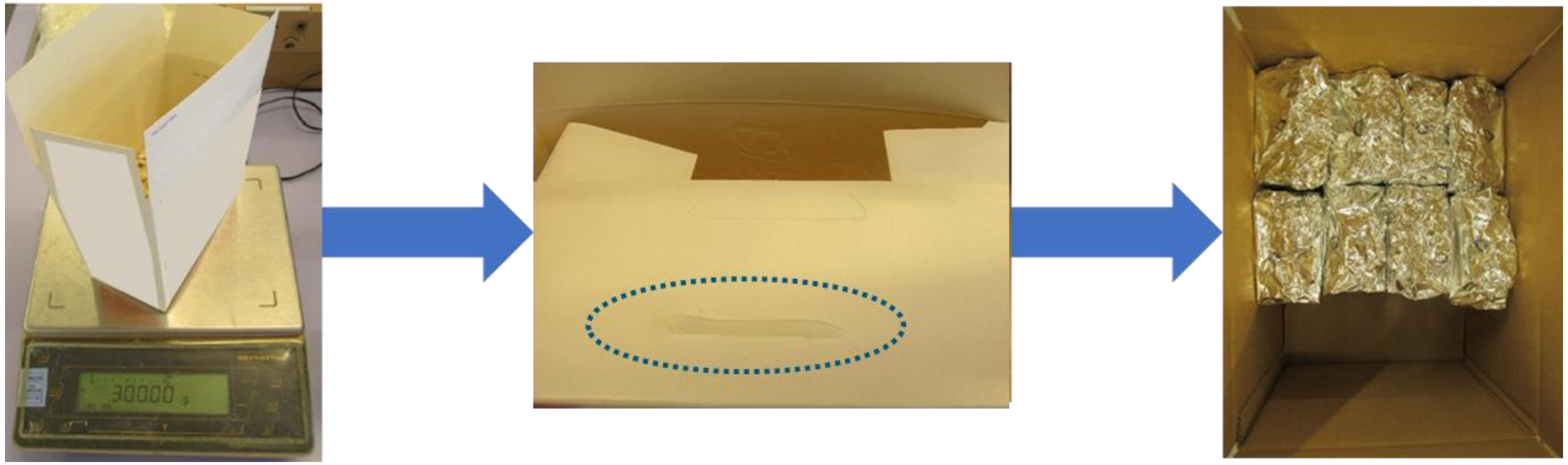

Reference: gassner-glastechnik.de

- Conditions:
  - **10d at 40˚C**

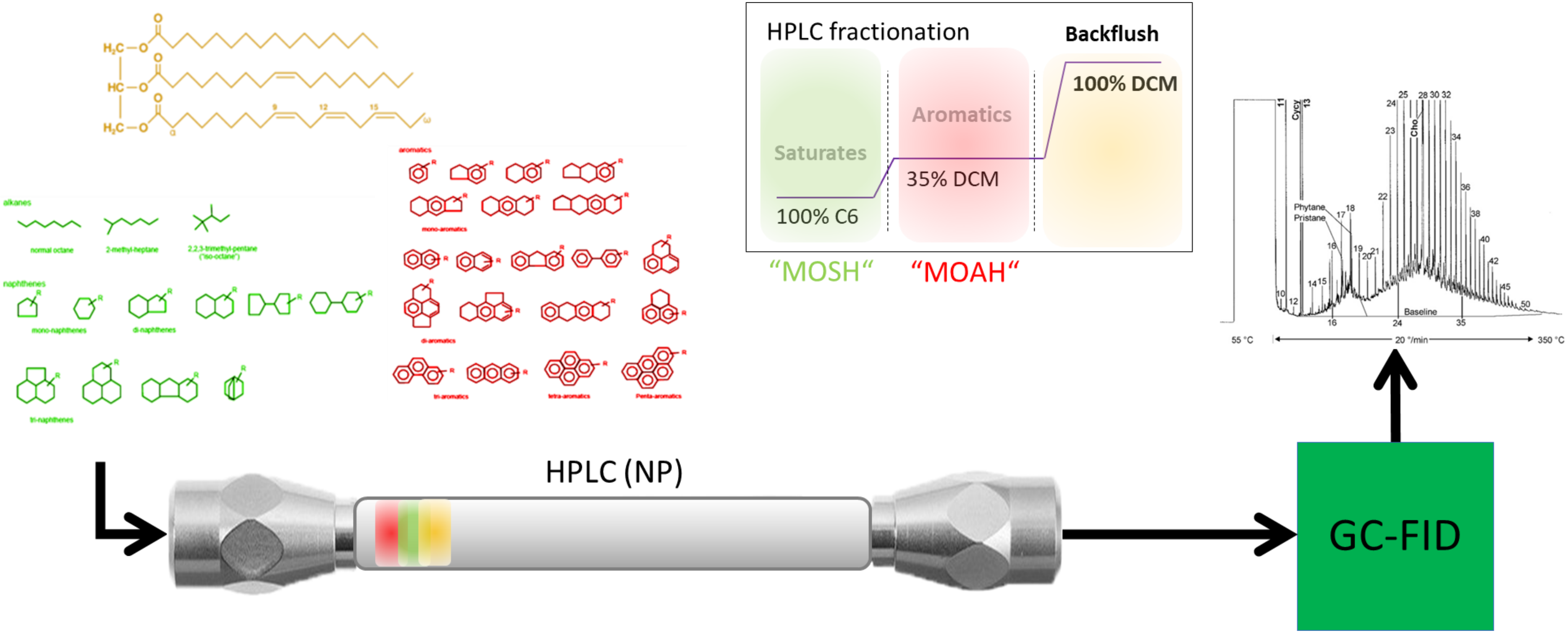
- Extraction of Tenax with n-hexane

# 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

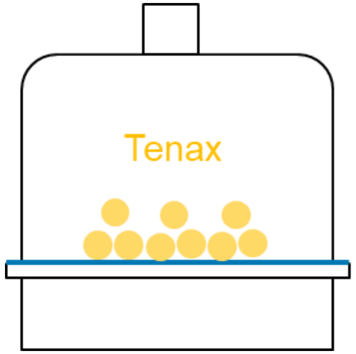


# Testing setup – Comparison of approaches

Extraction test

Content of migratable substances

Migration test



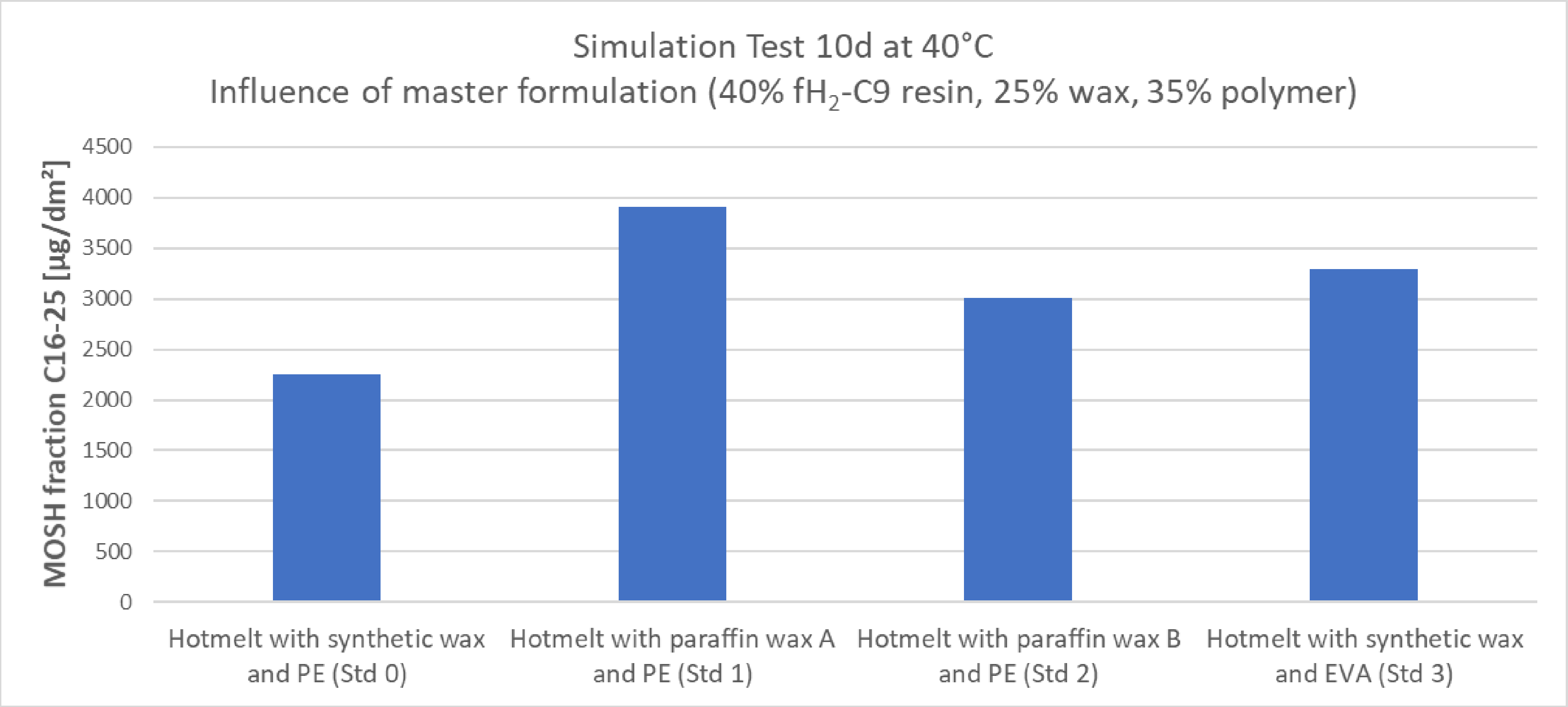

Simulated migration

Storage test

Real migration

*Estimation approach?*

# Results – Migration simulation



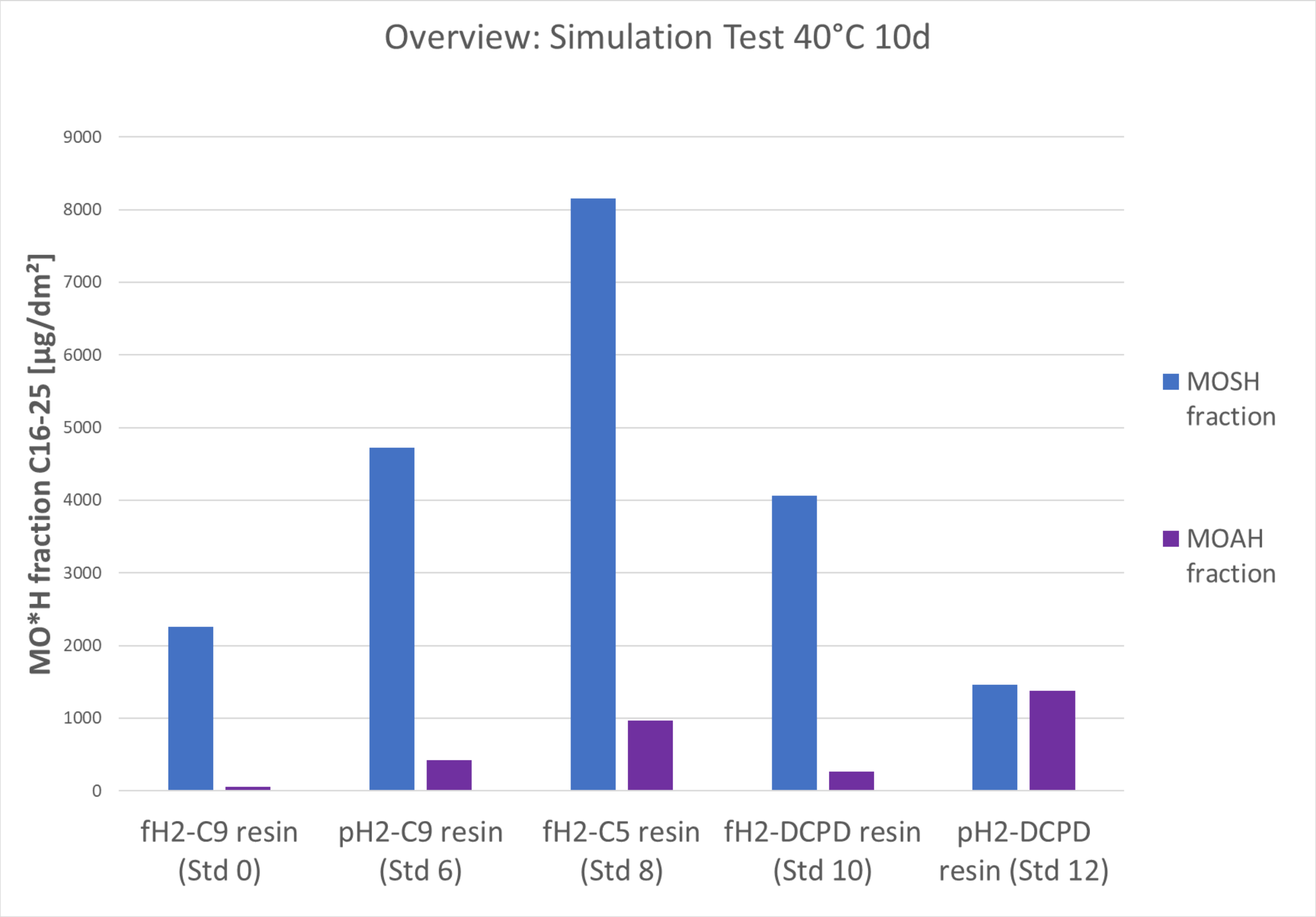

Hotmelt matrix effect

- 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





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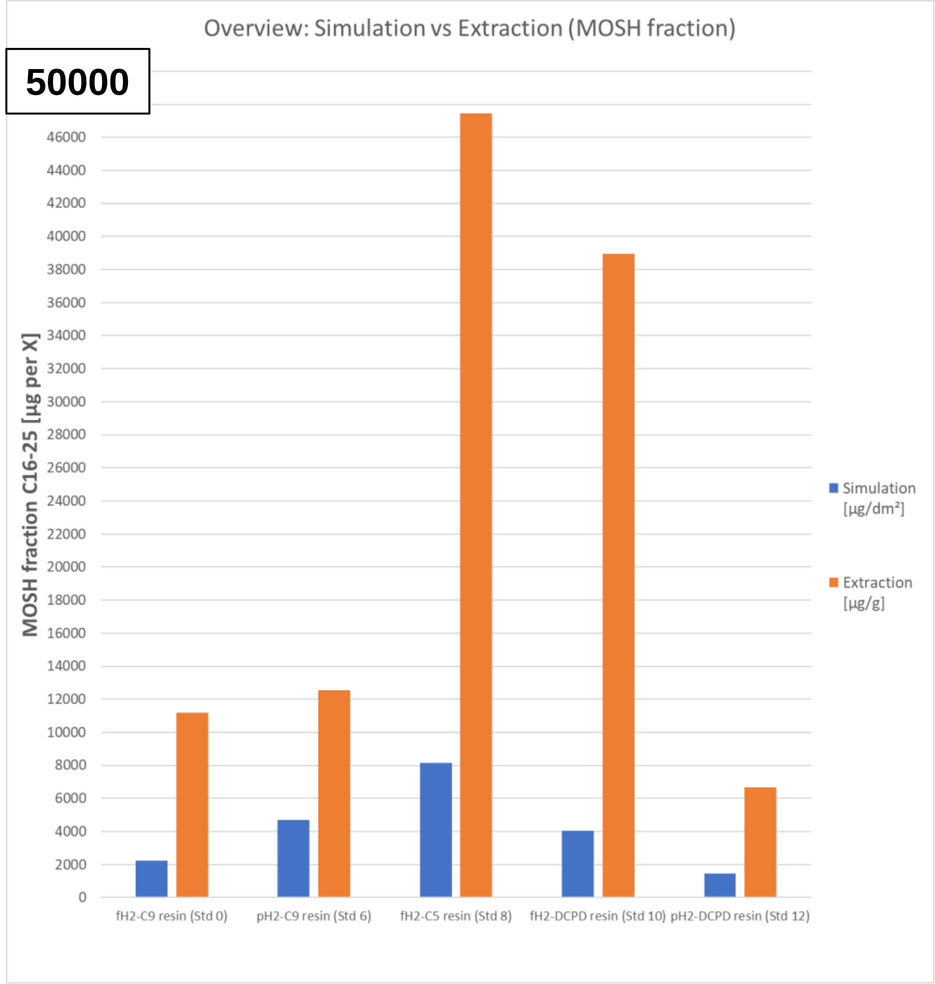
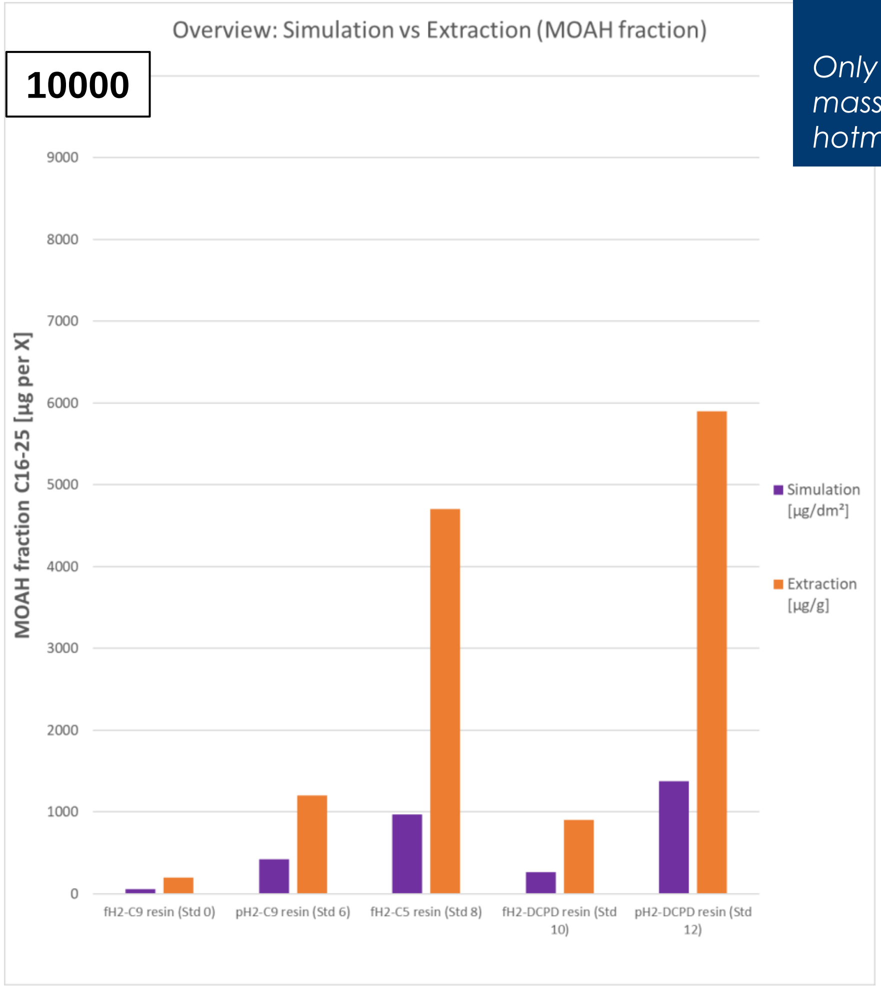
# Results – Migration simulation and extraction



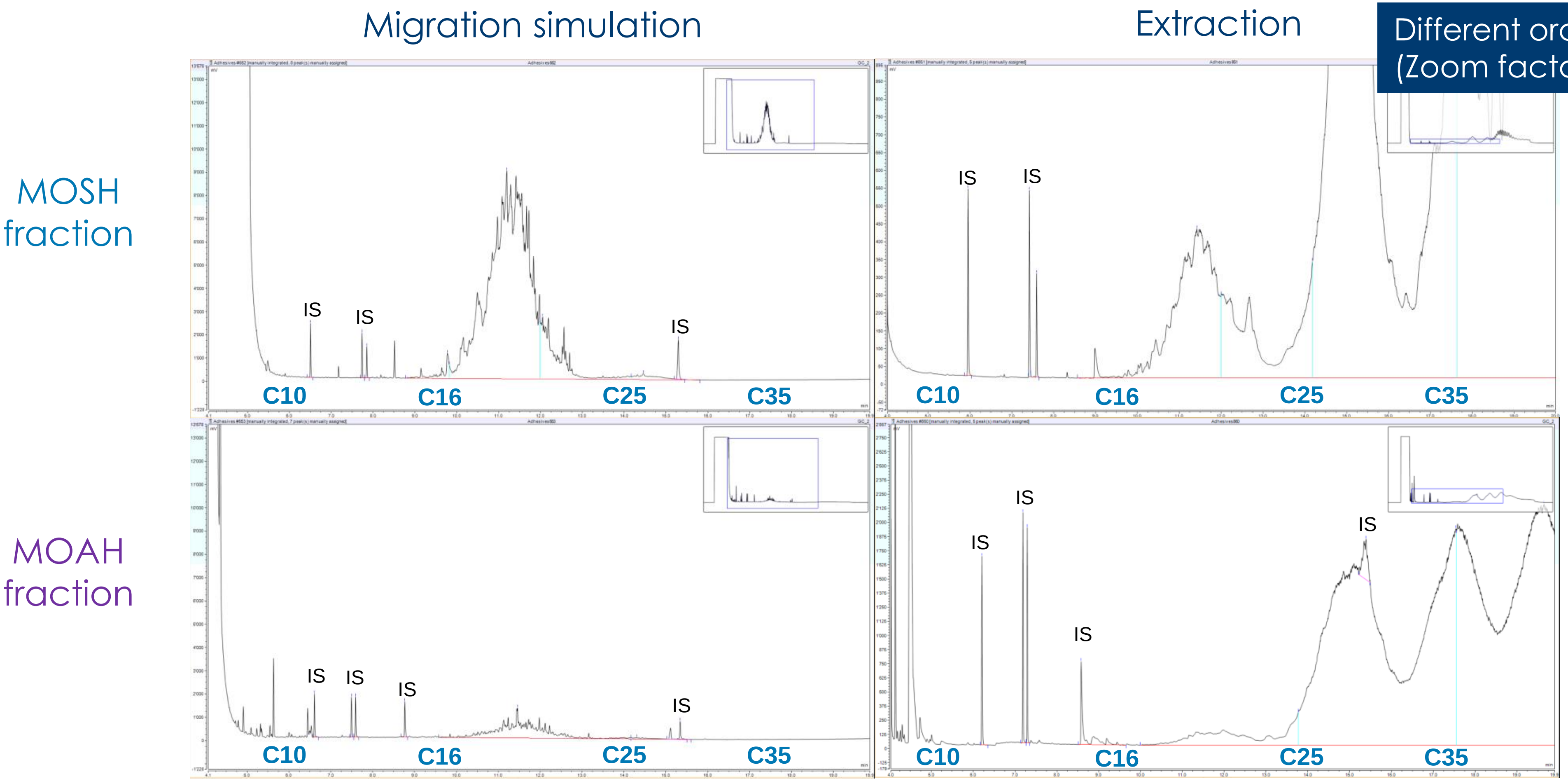




Units
Simulation: µg/dm²
Extraction: µg/g (ppm)

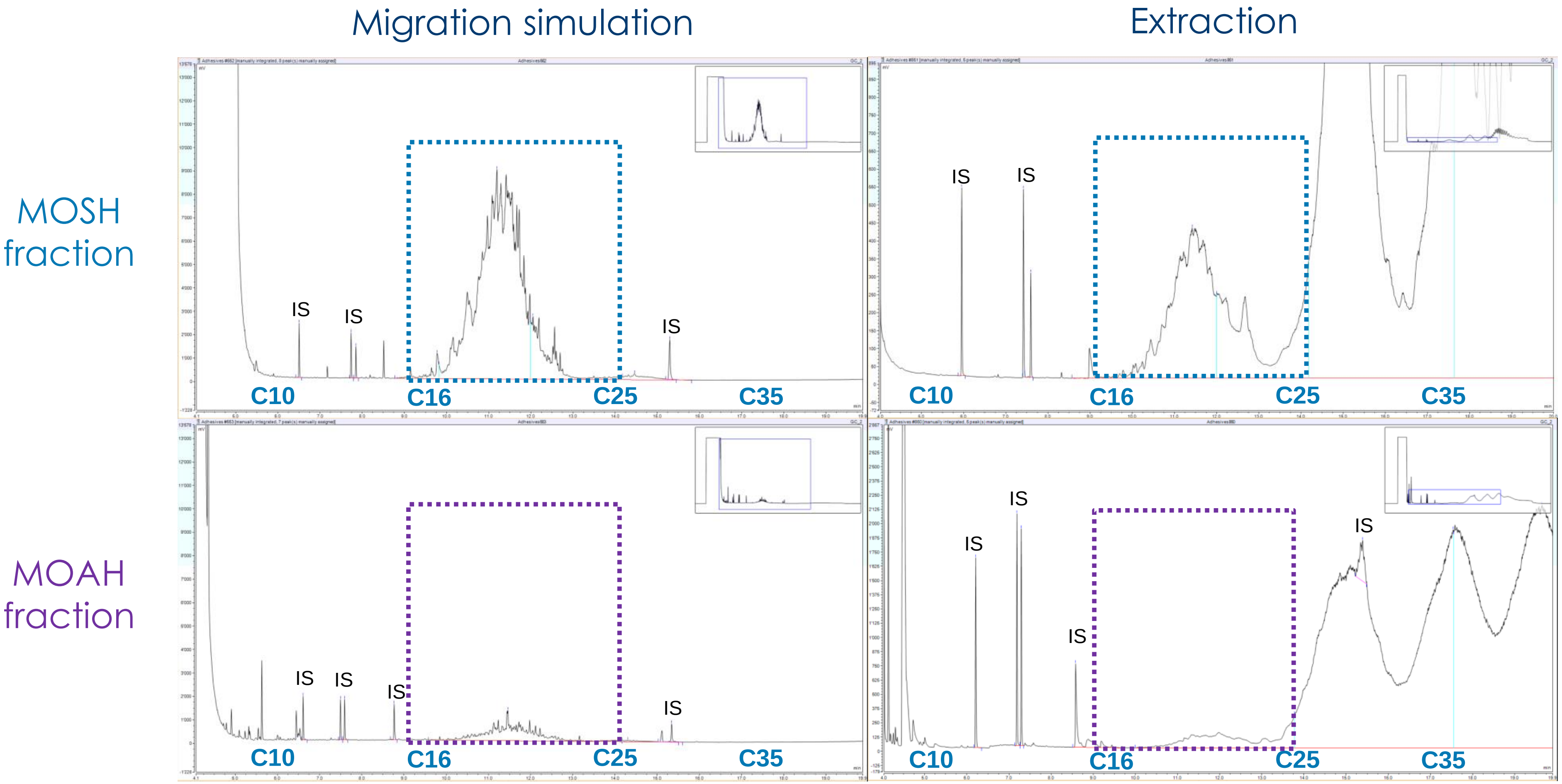
*Only comparable for a mass/area ration of 1g/dm² hotmelt*

# Results – Migration simulation and extraction

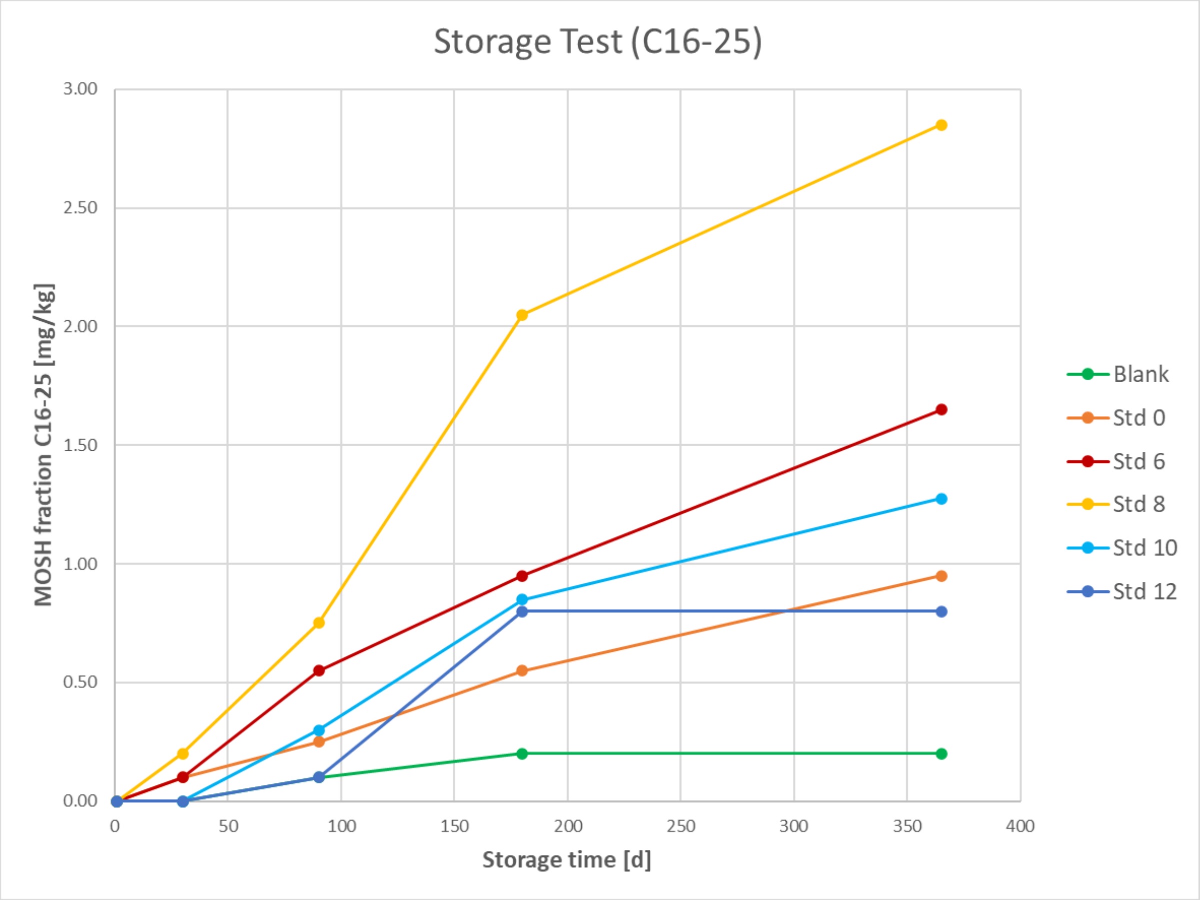
Different order of magnitude (Zoom factor)



# Results – Migration simulation and extraction



# Results – Storage test




| Sample | MOSH fraction C16-25 [mg/kg] | | | | MOAH fraction C16-25 [mg/kg] | | | |
|---|---|---|---|---|---|---|---|---|
| | 30 d | 90 d | 180 d | 365 d | 30 d | 90 d | 180 d | 365 d[1] |
| Blank | < 0.20 | < 0.20 | 0.20 | 0.20 | < 0.20 | < 0.20 | < 0.20 | < 0.10 |
| Std 0 *(fH2-C9)* | < 0.20 | 0.25 | 0.55 | 0.95 | < 0.20 | < 0.20 | < 0.20 | < 0.10 |
| Std 6 *(pH2-C9)* | < 0.20 | 0.55 | 0.95 | 1.65 | < 0.20 | < 0.20 | < 0.20 | 0.20 |
| Std 8 *(fH2-C5)* | 0.20 | 0.75 | 2.05 | 2.85 | < 0.20 | < 0.20 | < 0.20 | 0.22 |
| Std 10 *(fH2-DCPD)* | < 0.20 | 0.30 | 0.85 | 1.30 | < 0.20 | < 0.20 | < 0.20 | 0.15 |
| Std 12 *(pH2-DCPD)* | < 0.20 | < 0.20 | 0.80 | 0.80 | < 0.20 | < 0.20 | < 0.20 | 0.27 |

## Order Magnitude (blank adjusted):

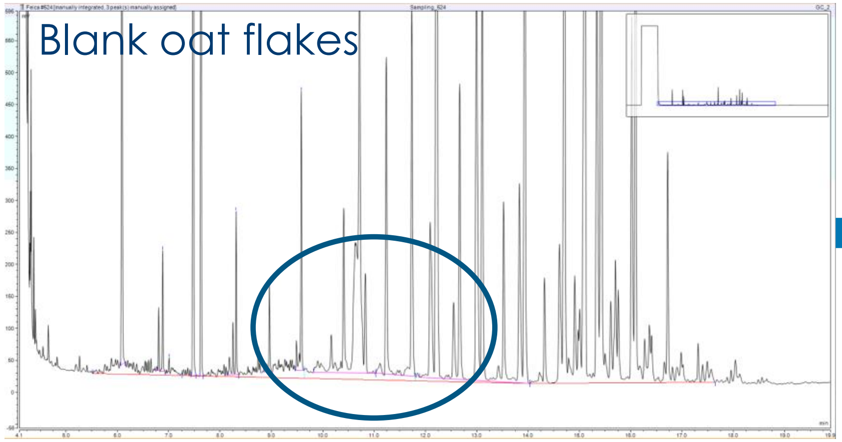
Saturated resin oligomers (MOSH fraction):
≈ **1 – 3 mg/kg** for the tested hotmelt types

Aromatic/unsaturated resin oligomers (MOAH fraction):
**< 0.2 mg/kg** for the tested hotmelt types

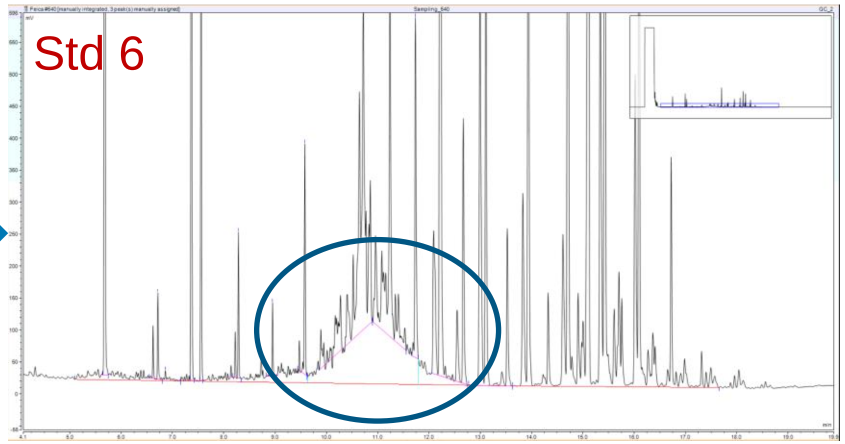
# Results – Storage test

Saturated resin oligomers: ≈ 2 mg/kg

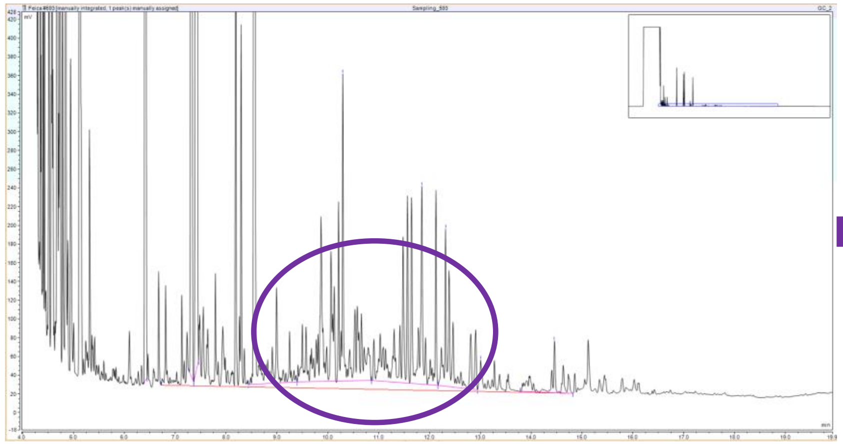
MOSH fraction



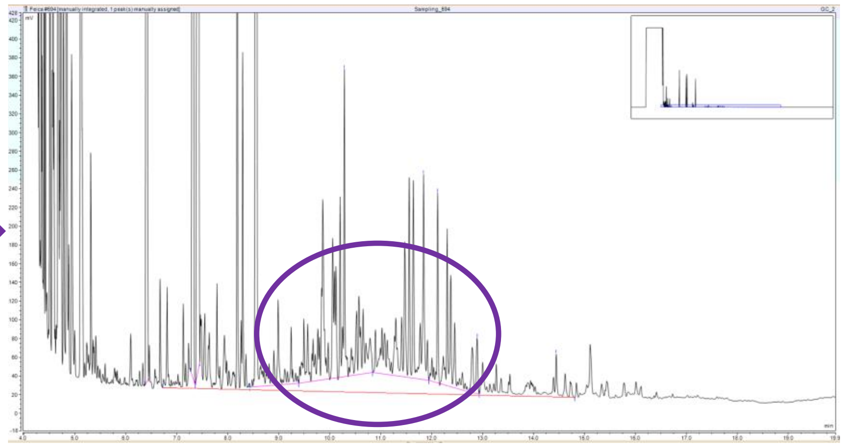

12 months




MOAH fraction



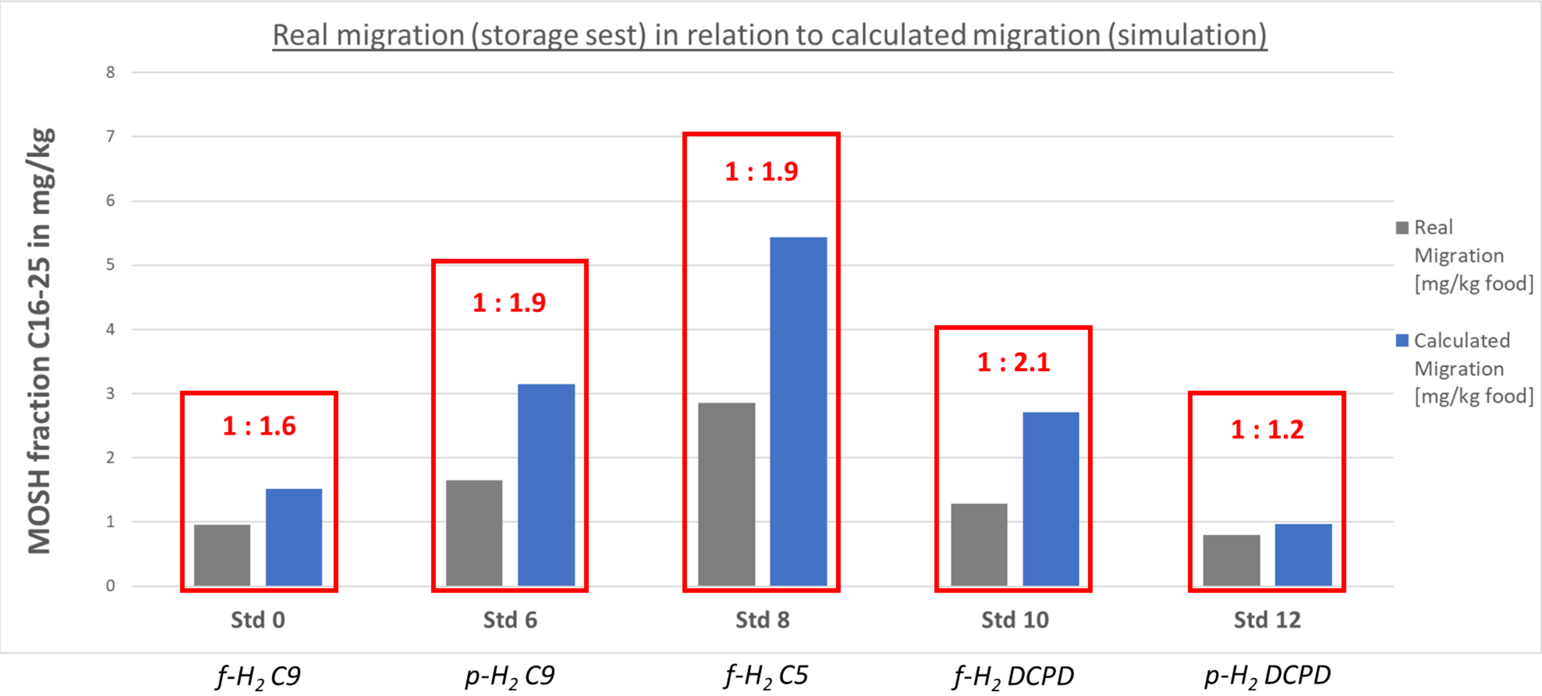
12 months



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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Other interests or questions ?

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