Sensory Testing of Food Contact Materials
- a science based practical approach

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Workshop on Sensory Testing of Food Contact Materials,
JRC EURL Ispra, 29th November 2011
Sensory Testing of Food Contact Materials

1. Legal situation
2. Physiological definitions
3. Sources and formation of taints
4. Standards for sensory testing of food contact materials
5. Basics of Sensory Testing of FCM
6. Training of a FCM sensory panel
7. Problems and needs
Legal Situation

– Framework Regulation 1935/2004 on food contact materials

  • Art. 3 no transfer of of constituents to food in quantities which could
    → endanger human health
    → bring about an unacceptable change in the composition of the food
    → bring about a deterioration in the organoleptic characteristics thereof
Legal Situation

– GMP Regulation (VO 2023/2006/EC)

• **Aim** (Art. 3):
  ‘... materials and articles are consistently produced and controlled to ensure conformity with the rules applicable to them and with the quality standards *appropriate to their intended use* by not .... *causing a deterioration in the organoleptic characteristics* thereof;

• **Tools:**
  → Systematic measures at any point of the production chain
  → **Communication** between the stages of the production chain about the intended use (food type, duration, temperature of contact etc.)
  → impose **complementary measures on all stages of production, transport and storage** to minimize taints
Situation

Are taints in FCM an Issue?

Yes!

Why are they seldom found in a final product?
Situation

- Consumer can act directly and will complain if the sensory properties of the food are deteriorated.
- Food companies do (normally) sensory tests on their final product (and hopefully FCM).
- FCM taints are masked/covered by the intense aroma of the filled food.
- FCM taints can be confused with the change of a food aroma during storage (→ best before date).
Situation

– Example

• Sensory testing has been done in ink producing industry far prior to the ITX crisis
• Responsibility for visible and 'smelling' transfer of substances has been well accepted by this industry while the so-called 'invisible' set off (ITX, benzophenone, ...) has not
Sensory Testing of Food Contact Materials

1. Legal situation
2. **Physiological definitions**
3. Sources and formation of taints
4. Standards for sensory testing of food contact materials
5. Basics of Sensory Testing of FCM
6. Training of a FCM sensory panel
7. Problems and needs
Physiological Definitions

- **Senses for the perception of conformity relevant sensory properties**
  - electromagnetic radiation:
    - Vision
  - chemical substances:
    - Gustation
    - Olfaction
    - 'Trigeminal factors'

[Image: aus http://www.g-netz.de]
Physiological Definitions

Perception in the mouth

– Gustation
  sensory cells located on the surface of the tongue as well as in the mucosa of the palate and areas of the throat
  • sweet
  • sour
  • bitter
  • salty
  • umami

– Trigeminal factors
  • astringent
  • burning
Physiological Definitions

- Olfaction
  - Distinction of thousands of odours
  - about 30 Mill. Receptor cells
  - 350 different olfactory receptors

- Odorant Perception
  a) orthonasally
  b) retronasally
Sensory Testing of Food Contact Materials

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# Taints

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Taints
Sources and Formation

– Plastic - Monomers:
  • Styrene in Polystyrene
    → Yoghurt cups
    → Foamed trays and cups
    → Transparent containers

Odour threshold (water) 0,01 mg/L
# Taints

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T.J. Simat, TU-Dresden, Workshop on Sensory Testing Ispra, 29th Nov. 2011 15
Taints
Sources and Formation

- Plastic:
  PET thermal degradation
    - Formation of acetaldehyde (blow moulding)

\[
\begin{align*}
\text{PET thermal degradation} & \\
\text{Formation of acetaldehyde (blow moulding)} & \\
\end{align*}
\]

\[
\text{Odour threshold (water): 0,02 mg/L}
\]

nach Ewender und Welle, Poster (2008)
Taints
Sources and Formation

- Plastic:
  PE/PP thermal degradation
  - Injection moulding

<table>
<thead>
<tr>
<th>Substance</th>
<th>Odour description</th>
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<tr>
<td>1-Octen-3-on</td>
<td>mushroom</td>
</tr>
<tr>
<td>Nonanal</td>
<td>rancid</td>
</tr>
<tr>
<td>Octanal</td>
<td>orange</td>
</tr>
<tr>
<td>2-Nonenval</td>
<td>paper, board</td>
</tr>
<tr>
<td>Hexanal</td>
<td>green, gras</td>
</tr>
<tr>
<td>8-Nonenal</td>
<td>plastic</td>
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Aus: Bravo et al. 1992, Sanders et al. 2005
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Taints
Sources and Formation

– Plastic:
  PE/PP thermal degradation of clarifiers
  • Degradation of bis(p-Methylbenzylidene)sorbitol (BMBS) to p-Methyl benzaldehyde during moulding of PP

\[
\text{BMBS} + \text{H}_2\text{O} \rightarrow \text{4-Methyl benzaldehyde}
\]

Aus: Roth et al. 2000

4-Methyl benzaldehyde

Odour Threshold (water): 0.13 µg/L
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Taints
Sources and Formation

- Plastic:
  PVC: degradation of additives:
  plasticizer, stabilisator (HCl scavenger)
  - Epoxidized Soy Bean Oil (ESBO)
  - oxidation of fatty acids yielding short chain aldehydes
    → musty, rancid

\[
\text{R: z.B. Linolsäure} \quad \text{R: epoxidierte Linolsäure}
\]

\[
\text{Triacylglycerol}
\]
Taints
Sources and Formation

- Plastic:
  degradation of additives: lubricants
    • Polyolefin screw caps are treated with e.g. erucamide
    • plastic-like off-flavor in mineral water 'sunlight flavor' (Strube et al. 2009)
    • Odorants:
      saturated and mono or di-unsaturated carbonyl compounds
    • Formation
      → Light (VIS/UV-) irradiation

\[
\begin{align*}
&\text{H}_2\text{N} \\
&\text{O} \\
&\text{Erucamid}
\end{align*}
\]
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<td>contaminants</td>
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<td>starting mat.</td>
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<td>Storage environment</td>
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<td>- reaction</td>
<td>Sealing (heat, ultrasonic)</td>
<td>- Odorous goods</td>
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<td>Curing (UV, Temp.)</td>
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<td>Corona treatment</td>
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Taints
Sources and Formation

– Printing ink: solvent

- Ethyl acetate
  \[
  \begin{array}{c}
  \text{Odour threshold (water):} \\
  1 \text{ mg/L}
  \end{array}
  \]

- Toluene
  \[
  \begin{array}{c}
  \text{Odour threshold (water):} \\
  4 \text{ mg/L}
  \end{array}
  \]

- Ethoxy propanol
  \[
  \begin{array}{c}
  \text{Odour threshold (water):} \\
  160 \text{ mg/L}
  \end{array}
  \]

- Methyl ethyl ketone
  \[
  \begin{array}{c}
  \text{Odour threshold (water):} \\
  75 \text{ mg/L}
  \end{array}
  \]
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Taints
Sources and Formation

– adhesives:

reaction product of a monomer and an impurity in a solvent:
2-ethyl-5,5′-dimethyl-1,3-dioxane (2-EDD) (Schweitzer et al. 1999)
• Neopentyl glycol is a frequently used monomer (e.g. for polyester)
• Propionaldehyde is an impurity in propylene glycol

```
OH  OH
\   \ \\
O   O
Neopentylglykol  Propionaldehyde  2-ethyl-5,5′-dimethyl-1,3-dioxane
```

Odour threshold (water): 0,01 µg/ L
artificial fruity, blueberry
# Taints

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Taints
Sources and Formation

- Wax coating for inner pouches contamination with aromatic hydrocarbons

Kellogg Company has launched legal action against a Canadian firm over allegations its tainted packaging cost the food giant millions of dollars and forced it to issue a nationwide recall of breakfast cereals.

The US-based firm this week filed a lawsuit against FPC Flexible Packaging Corp (FPC) in the wake of the country-wide recall in June 2010 alleging the Ontario outfit was responsible for the supply of tainted inner linings that sickened dozens of consumers.

The incident, which attracted global headlines, also saw the company change it packaging formulation for the affected products, Kellog revealed.

Damaged reputation

In papers file in the US District Court for the Western District of Michigan, Kellogg is demanding FPC pay it damages resulting from the purchase of what it claims to be “defective liners” bought from the Scarborough company.

Kellogg claims that elevated levels of hydrocarbons in the liners caused “offensive, off-characteristics that caused symptoms including nausea and diarrhoea”.

It said the subsequent “costly recall“ of an estimated 28 million boxes of four types of cereal and the destruction of millions more in its warehouses not only hit its annual profits but also injured its reputation and damaged customer goodwill.

# Taints

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Taints
Sources and Formation

– Recycled paper and board:
(Czerny und Büttner, 2009)

Verbindung | Geruchscharakteristik
---|---
Vanillin | Vanilla
E-Non-2-enal | Fatty, board
γ-Nonalacton | coco
2-Methoxyphenol | Smoky, vanilla
δ-Decalacton | coco
3-Propylphenol | Leather, phenolic
p-Anisaldehyde | fatty, plastic
Z-Non-2-enal | fatty, board
tr-4,5-Epoxy-E-dec-2-enal | metallic

Packaging for fresh milk

http://www.verpackungs-shop.net/images/auto_01.jpg
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Taints
Sources and Formation

– Contaminating taints during transport and storage: halophenols and haloanisols
  • Pentachlorophenol: - frequently applied fungicide for wood
  • Tribromophenole: - flame retardant
    - fungicide for leather, textiles etc.
  • Chloro- und Bromoanisole are formed from the corresponding phenols by **microbial Methylation**

![Chemical Structures]

2,4,6-Tribromophenol
Odour threshold (water): 0,1 µg/L

2,4,6-Tribromoanisol
Odour threshold (water): 0,00002 µg/L
Taints
Sources and Formation

- Halophenols and -anisols
  • Odour type:
    → Bromo- und Chlorophenols: desinfectant, medicinal
    → Chloro- und Bromoanisols: musty, mouldy
  • Source of contamination:
    → pallet
    → Wooden boards
Visible set-off

– Comparison Food - Simulant (LUA Dresden 2011)

Simulation with test paper, DIN EN 646 salvia simulant pH 8,2 10 min, 1 kg

Serviette

egg, 10 min, 220g
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Standards for FCM Testing

FCM from paper and board in indirect contact (transfer via gas phase)

- Test for odour

FCM in direct & indirect contact with food

- Test for odour

- Test for transfer of taints

- Test for aroma transfer

Training of a sensory panel

- CEN/TR 15645 1 bis 3 (2007)
Standards for FCM Testing

- Example
  'Robinson Test'

- Test Conditions
  - volume 1L at 75% r.H.
  - 6 sdm paper or board
  - inkubation 48 h at 23°C
  - 20g ground milk chocolate

  • accelerated test:
    - humidity
    - ground food
    - surface-to-volume-ratio
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Basics of Sensory Testing of FCM

– Odour Test
  - fill FCM sample into a empty glass test container, wait 24h at RT → release of taint?

  • Not suitable
    → for some materials (e.g. polyamide kitchen utensils)
    → taints are transferred at higher temperatures
Basics of Sensory Testing of FCM

- Test with simulating food
  - Dry food for gas transfer
  - Liquid and viscous for direct contact

- Mandatory since compliance has to be proven for food (see Art. 3 1935/2004)
Basics of Sensory Testing of FCM

- Test with simulating food

Only smelling at the simulating food (orthonasally) or 'tasting' it (retronasal perception)?
Basics of Sensory Testing of FCM

- generally smelling and 'tasting'
  - within dry food smelling is often more sensitive
  - within liquid food 'tasting' is often more sensitive
Basics of Sensory Testing of FCM

- Test with simulating food

What is the influence of the food matrix? (e.g. compared with water)
Basics of Sensory Testing of FCM

- Odour threshold in food is much higher than in water
Basics of Sensory Testing of FCM

- Odour threshold in liquid food is much higher than in water:
  water > apple juice ≈ 0.2% acetic acid >> yoghurt
Basics of Sensory Testing of FCM

- Odour threshold depends on the type of dry food (tested for benzaldehyde)

  chocolate ≈ butter cookie > coconut flakes >> glucose ≈ lactose
Basics of Sensory Testing of FCM

- Test with simulating food

Do all humans have a comparable odour sensitivity?

No!
Basics of Sensory Testing of FCM

- **Individual odour threshold** varies depending on the substance
  - Odour thresholds of 1,3-Dimethylnaphthaline and p-methyl benzaldehyde

\[
F = \frac{\text{thr}_{\text{most insensitive assessor}}}{\text{thr}_{\text{most sensitive assessor}}}
\]

- p-MBA: \( F = 63 \)
- 1,3-DMN: \( F = 7.5 \)
Basics of Sensory Testing of FCM

- Repeatability of odor thresholds
  - Odour thresholds for styrene of 16 panellists (n=7)

  
  • Smallest median, assessor 13: 0.6 µg/L
  • Largest median, assessor 16: 60.0 µg/L

[Graph showing odour thresholds for styrene]
Sensory Testing of Food Contact Materials

1. Legal situation
2. Physiological definitions
3. Sources and formation of taints
4. Standards for sensory testing of food contact materials
5. Basics of Sensory Testing of FCM
6. **Training of a FCM sensory panel**
7. Problems and needs
Training of a sensory panel

- Description and Recognition of FCM Taints
  - FCM taint set
    → Sniffing sticks
    → Odour solutions
Training of a sensory panel

- **In preparation:**
  Wheel of FCM Taints with sniffing sticks
Training of a sensory panel

Training of detection of taints in simulating foods

- Duo-/Triangle Tests
- Liquid foods:
  - Styrene in water (0.010 mg/L)
  - Styrene in apple juice (0.040 mg/L)
  - Styrene in 0.2% acetic acid (0.080 mg/L)
  - Hexanal in milk (3.5% fat) (0.020 mg/L)
  - 4-MBA in milk (3.5% fat) (0.031 mg/L)
  - Styrene in yoghurt (3.5% fat) (0.32 mg/L)
- Solid foods
  - Non-2-enal in chocolate
  - Ethylacrylate in butter cookie
  - Acetophenone in toast
Training of a sensory panel

- Training of the ordinal scale
  - Hexanal in aqueous solution

<table>
<thead>
<tr>
<th>Intensity</th>
<th>Hexanal (mg/L)</th>
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<tr>
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Sensory Testing of Food Contact Materials

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Problems

- No harmonized standardisation except for gas phase transfer form paper and board (EN 1230)
- German Standard DIN 10955 for sensory testing of FCM
  - is not harmonized
  - is a guide but leaves questions to testing conditions on specific materials open

• In consequence:
  → uncertainty for testing labs
  → problems may occur in export to other EU member states
  → non co-ordinated working groups on this topic
  → BfR working group to be established
  → Eurolab/DG Sens working group
Problems

– No appointment on the legal interpretation of sensory results:
  • BfR Germany: ordinal grade '3' is non compliant to Art. 3
  • Some producers/fillers set the limit to 2,5
  • Mineral water industry only accepts '≤ 2'

– 'natural materials' with 'natural' aroma
  • Wood
  • Bamboo

  → no wooden spoon will pass the sensory test after 2h 100°C
Needs

- European standard or guideline (analogous to the guideline for FCM by the ERL/NRL) for
  - Testing procedures and test conditions for FCM (time, temperature, simulating food, contact (surface to volume))
  - Legal interpretation of sensory results
  - Working group / networking groups to elaborate and maintain the guideline / standard
- Organisation of training / retraining
- Organisation of round robin tests
Conclusions

- Sensory inertness of FCM is a demand of the framework regulation 1935/2004, it must be achieved by complementary measures at all stages of the production chain (GMP 2023/2006/EC)
- Sources for taints are manyfold (often NIAS)
- Formation can even depend on storage conditions
- European Harmonisation of FCM testing is mandatory since this situation may lead to trade barriers
- Training of panellists and organisation of proficiency testings
Thanks

– Members of my group
  • Elke Striebing
  • Maria Dreger
  • Lydia Richter

For your attention
Legal Situation

- Good Manufacturing Practice - GMP (VO 2023/2006/EC)
  
  • Examples for complementary measures on all stages of production, transport and storage
    → Testing starting materials
      + solvents
      + Adhesives, waxes
      + Printing inks
    → Supervise process parameters
      + Injection molding, blow molding, extrusion, corona treatment
    → Final product, products from the market