Optical methods to determine the gas atmosphere in various modified atmosphere packages: applications and correlation in meat spoilage

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Session 8: Food Packaging and Preservation

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Motivation and Objectives

Why non-destructive measurement systems?

- Food loss especially in Europe and North-America to 1/3 at the consumer → Expired best before date often causative
- Höll et al. stored poultry under high oxygen atmosphere in 2016
  - Invasive detection of $O_2$ and $CO_2$ of packaging headspace
  - Control of TVC (Total Viable Count)
- Critical Value of TVC for poultry defined at $10^7$ CFU/g → Reached at day 7 (10°C) or 9 (4°C)
  → Accompanied by a fast decrease of $p_{O_2}$ and increase of $p_{CO_2}$

**Idea:**
Gas development in the headspace of the packaging as a marker for microbiological spoilage

Measurement with non-destructive systems also allows wide application such as checks in the supermarket or process control in the food processing plant
Non-Destructive Measurement Devices

**Oxygen (O<sub>2</sub>)**
- Principle of O<sub>2</sub> measurement based on *dynamic fluorescence quenching*
- Integration of fluorescent material into packaging (lid-film) via heat-sealing
- Annular seal between films enables potentially food-compliant integration into packaging

**Carbon Dioxide (CO<sub>2</sub>)**
- Principle of CO<sub>2</sub> measurement based on *MIR (mid-infrared) absorption spectroscopy*
- $\lambda_1=4.26\mu m$, $\lambda_2=4.45\mu m$ & $\lambda_3=4.27\mu m$
- Laser beam is guided through a corner of the package at 45°
**Experimental Set-Up**

<table>
<thead>
<tr>
<th>Storage Time: 15 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>Empty</td>
</tr>
<tr>
<td>Filled (poultry)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Non-destructive headspace analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurement: Daily (exc. 3 (filled) and 3/5/6/12&amp;13 d (empty))</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Total Viable Count</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurement: 0/1/4/6/9/1/13/15 d</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sensory Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurement: 0/1/4/6/8/11/14 d (4°C) or 0/1/4/6/8 d (10°C)</td>
</tr>
</tbody>
</table>

- **80% O₂ / 20% CO₂** at 4°C
- **70% O₂ / 30% CO₂** at 10°C

**O₂**

**CO₂**

**TVC**

**Sensory Analysis**
**Results – Development of headspace gas concentration**

**Development of gas composition in empty trays**
- Hardly any change in the gas composition

**Significant change: empty vs. filled trays**
- Poultry stored at 4°C showed deviations of CO₂ from day 12-15 for 80% O₂/20% CO₂ MAP
- Poultry stored at 10°C showed deviations of O₂ and CO₂ for both gas compositions
- For O₂ values of 4°C stored samples, no significant difference between trays with and without product visible

**Cross-Over: empty vs. filled trays**
- Cross-Over (●) = gas value of filled tray intersects gas value of empty tray → indication for change of headspace atmosphere
- Appears 1 or 2 days before the first significant deviation
- For O₂ values of 4°C stored samples, only cross-over observed, where afterwards a decrease of O₂ of filled trays is indicated

![](chart.png)

-O <sub>2</sub> (●/●) and CO₂ (△/△) at different storage conditions over 15 days in trays with (●/△) and without (O/△) poultry

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1 Level of significance (T-test): * P ≤ 0.05 ** P ≤ 0.01 *** P ≤ 0.001
Results – Microbiological analysis

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Day 0</th>
<th>Day 15</th>
<th>Shelf life expired at</th>
</tr>
</thead>
<tbody>
<tr>
<td>80/20 4°C</td>
<td>1.36x10⁴ CFUg⁻¹</td>
<td>4.00x10⁹ CFUg⁻¹</td>
<td>Day 6</td>
</tr>
<tr>
<td>70/30 4°C</td>
<td>1.27x10⁴ CFUg⁻¹</td>
<td>4.29x10⁹ CFUg⁻¹</td>
<td>Day 7</td>
</tr>
<tr>
<td>80/20 10°C</td>
<td>1.36x10⁴ CFUg⁻¹</td>
<td>2.19x10¹⁰ CFUg⁻¹</td>
<td>Day 3</td>
</tr>
<tr>
<td>70/30 10°C</td>
<td>1.27x10⁴ CFUg⁻¹</td>
<td>4.47x10⁹ CFUg⁻¹</td>
<td>Day 4</td>
</tr>
</tbody>
</table>

**End of Shelf life**
Microbiologically spoiled (red line) when TVC=10⁷ CFU/g

**Shelf life vs. Gas development**
- All samples were microbiologically spoiled prior to headspace gas change
- In contradiction to the previous study (Höll et al. 2016)

→ End of Shelf life ≠ Gas change
→ Cross-Over is a useful spoilage indicator at 10°C
Results – Sensory Evaluation

**Sensory Evaluation**
Visual and orthonasal impression was evaluated on an analogue scale ranging from 0 to 100 (0 = not perceptible/fresh; 100 = strong perceptible/rotten).

**50 Scores Limit**
For the evaluation, a sample was defined as no longer acceptable, when the average value of the orthonasal or visual impression was ≥ 50.

**Visual Impression**
- 4°C stored samples „fresh“ until day 11
- 10°C stored samples „fresh“ until day 4 (80/20) or day 6 (70/30)

**Orthonasal Impression**
- 4°C stored samples „fresh“ until day 8 (80/20) or day 11 (70/30)
- 10°C stored samples „fresh“ until day 4 (80/20) or day 6 (70/30)

**Sensory Spoilage ≠ Microbiological Spoilage**

Visual (▲) and orthonasal (●) impression at different storage conditions over 8 or 15 days. Red line marks TVC > 10^7 CFUg⁻¹
Purple area marks the defined sensory limit of 50 scores.
# Correlation of the results - Discussion

<table>
<thead>
<tr>
<th></th>
<th>4°C</th>
<th>10°C</th>
<th></th>
<th>4°C</th>
<th>10°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>80/20</td>
<td>70/30</td>
<td>80/20</td>
<td>70/30</td>
<td>------</td>
</tr>
<tr>
<td>cross-over $O_2$</td>
<td>12</td>
<td>13</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>$P \geq 0.05$ $O_2$</td>
<td>-</td>
<td>-</td>
<td>6</td>
<td>7</td>
<td>------</td>
</tr>
<tr>
<td>cross-over $CO_2$</td>
<td>10</td>
<td>-</td>
<td>4</td>
<td>4</td>
<td>------</td>
</tr>
<tr>
<td>$P \geq 0.05$ $CO_2$</td>
<td>12</td>
<td>-</td>
<td>5</td>
<td>6</td>
<td>------</td>
</tr>
<tr>
<td>microbiologically spoiled</td>
<td>6</td>
<td>7</td>
<td>3</td>
<td>4</td>
<td>------</td>
</tr>
<tr>
<td>olfactory spoiled</td>
<td>11</td>
<td>14</td>
<td>6</td>
<td>8</td>
<td>------</td>
</tr>
</tbody>
</table>

$P \geq 0.05$ stands for the day of the first calculated statistical significance between empty and filled trays after reaching the cross-over, microbiologically spoiled indicates a TVC of $10^7$ CFU/g and olfactory spoiled stands for a classification of the sensory panel above the limit of 50 scores.

### Possible Correlations:
- **Yellow**: Cross-Over & Microbiologically Spoilage
- **Orange**: Cross-Over & Olfactory Spoilage
- **Green**: Headspace gas change & Olfactory Spoilage

### $O_2$/$CO_2$ & Microbiologically Spoilage
- Significant change of headspace gas composition not correlatable with microbiological limit of $10^7$ CFU/g
- Cross-Over (esp. $CO_2$) for 10°C samples useful

→ **Cross-Over for detection of irregular storage conditions (e.g. cold chain interruption)**

- Spoilage under regular storage not detectable with $O_2$/$CO_2$ change

### $O_2$/$CO_2$ & Sensory Spoilage
- Cross-over of 4°C stored samples well in common with olfactory spoilage
- Headspace change of 10°C stored samples correlateable with sensory spoilage

→ **$O_2$/$CO_2$ change = Sensory spoilage**
Conclusion and Outlook

- No individual shelf-life prediction for regular stored, high-oxygen packed poultry possible via measurement of the headspace gas atmosphere (O₂/CO₂)

- Other gas compositions and/or foods (e.g. red muscle beef) must be tested

- Volatile Organic Compounds as marker to be discussed

**Applicability:**

- Both measurement systems useful for non-destructive detection of O₂ and CO₂

- Control of cold-chain interruption/irregular storage conditions possible

- Further applications to be tested: process control for MAP production lines or detection of leakages in packages
  → Research regarding measuring precision for both systems is work in progress
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References


