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

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# **Motivation and Objectives**



# 100 80 60 40 20 0 2 4 6 8 10 12 14 100 12

### 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
  - $\circ$  Invasive detection of O<sub>2</sub> and CO<sub>2</sub> of packaging headspace
  - Control of TVC (Total Viable Count)
- Critical Value of TVC for poultry defined at 10<sup>7</sup> CFU/g
  - $\rightarrow$  Reached at day 7 (10°C) or 9 (4°C)
  - $\rightarrow$  Accompanied by a fast decrease of  $p_{O2}$  and increase of  $p_{CO2}$

# ldea:

# 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



Oxygen at  $4^{\circ}C(\bullet)$  and  $10^{\circ}C(\bullet)$  and  $CO_2$  at  $4^{\circ}C(\bullet)$  and  $10^{\circ}C(\bullet)$ [2].

# **Non-Destructive Measurement Devices**

# Oxygen (O<sub>2</sub>)

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



Used Sensors for the integration (left) [3] and schematic representation of the sealed-in sensor material into the lid film of a plastic tray (right)

# Carbon Dioxide (CO<sub>2</sub>)

- Principle of CO<sub>2</sub> measurement based on *MIR (mid-infrared) absorption* spectroscopy
- ο  $λ_1$ =4.26μm,  $λ_2$ =4.45μm &  $λ_3$ =4.27μm
- $\circ~$  Laser beam is guided through a corner of the package at 45°





# **Experimental Set-Up**





Non-destructive headspace analysis Measurement: Daily (exc. 3 (filled) and 3/5/6/12&13 d (empty))

<u>Total Viable Count</u> Measurement: 0/1/4/6/9/1/13/15 d

<u>Sensory Analysis</u> Measurement: 0/1/4/6/8/11/14 d (4°C) or 0/1/4/6/8 d (10°C)





# **Results – Development of headspace gas concentration**



 $O_2(\bullet \land \bigcirc)$  and  $O_2(\bullet \land \bigcirc)$  at different storage conditions over 15 days in trays with ( $\bullet \land \land$ ) and without ( $O \land \bigcirc$ ) poultry

# Development of gas composition in empty trays

• Hardly any change in the gas composition

### Significant<sup>1</sup> change: empty vs. filled trays

- Poultry stored at 4°C showed deviations of CO<sub>2</sub> from day 12-15 for 80% O<sub>2</sub>/ 20% CO<sub>2</sub> MAP
- Poultry stored at 10°C showed deviations of  $O_2$  and  $CO_2$  for both gas compositions
- For O<sub>2</sub> 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 O<sub>2</sub> values of 4°C stored samples, only cross-over observed, where afterwards a decrease of O<sub>2</sub> of filled trays is indicated

Level of significance (T-test): \* P ≤ 0.05 \*\* P ≤ 0.01 \*\*\* P ≤ 0.001

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# **Results – Microbiological analysis**

	Day 0	Day 15	Shelf life expired at
80/20 4°C	1.36x10 <sup>4</sup> CFUg <sup>-1</sup>	4.00x10 <sup>9</sup> CFUg <sup>-1</sup>	Day 6
70/30 4°C	1.27x10 <sup>4</sup> CFUg <sup>-1</sup>	4.29x10 <sup>9</sup> CFUg <sup>-1</sup>	Day 7
80/20 10°C	1.36x10 <sup>4</sup> CFUg <sup>-1</sup>	2.19x10 <sup>10</sup> CFUg <sup>-1</sup>	Day 3
70/30 10°C	1.27x10 <sup>4</sup> CFUg <sup>-1</sup>	4.47x10 <sup>9</sup> CFUg <sup>-1</sup>	Day 4



End of Shelf life

Microbiologically spoiled (red line) when TVC=10<sup>7</sup> 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)

### $\rightarrow$ End of Shelf life $\neq$ 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  $\geq$  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 ( $\blacktriangle$ ) and orthonasal ( $\bullet$ ) impression at different storage conditions over 8 or 15 days. Red line marks TVC > 10<sup>7</sup> CFUg<sup>-1</sup> Purple area marks the defined sensory limit of 50 scores.

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	4°C		10°C	
	80/20	70/30	80/20	70/30
cross-over O <sub>2</sub>	12	13	5	5
P≥0.05 O <sub>2</sub>	-	-	6	7
cross-over CO <sub>2</sub>	10	-	4	4
P≥0.05 CO <sub>2</sub>	12	-	5	6
microbiologically spoiled	6	7	3	4
olfactory spoiled	11	14	6	8

 $P \ge 0.05$  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  $10^7 \text{ CFUg}^{-1}$  and olfactory spoiled stands for a classification of the sensory panel above the limit of 50 scores.

### **Possible Correlations:**

- <u>Yellow</u>: Cross-Over & Microbiologically Spoilage
- **Orange**: Cross-Over & Olfactory Spoilage
- o **<u>Green</u>**: Headspace gas change & Olfactory Spoilage

### O<sub>2</sub>/CO<sub>2</sub> & Microbiologically Spoilage

- Significant change of headspace gas composition not correlatable with microbiological limit of 10<sup>7</sup> CFU/g
- $\circ~$  Cross-Over (esp. CO\_2) for 10°C samples useful

# $\rightarrow$ Cross-Over for detection of irregular storage conditions (e.g. cold chain interruption)

 $\circ~$  Spoilage under regular storage not detectable with O\_2/CO\_2 change

### O<sub>2</sub>/CO<sub>2</sub> & 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

### $\rightarrow O_2/CO_2$ 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 (O<sub>2</sub>/CO<sub>2</sub>)
- 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 O<sub>2</sub> and CO<sub>2</sub>
- 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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