Novel Approaches in Industrial Research
Targeting Quality Attributes of Extruded Food Products

Tomas Davidek & Imre Blank
Nestle PTC Orbe
Extrusion with direct expansion
Intermeshing co-rotating twin-screw extruder

Flour Mix (~10% H₂O)
Water, Oil, ...

120-180 °C
100-150 bars
Steam

Moisture: 10%

~15-20%

Expansion

~10%

Average residence time: 20-30 seconds
Advantages of extrusion

The extruder performs several unit operations in one machine within seconds:

- Material conveying
- Ingredients mixing
- Heating, Cooking, Melting
- Shearing & compressing
- Expanding
- Texturizing & shaping

Operational benefits:

- High productivity
- Continuous processing system
- Low cost
  Lower labour, energy and floor space requirements
- Energy efficiency
  Lower moisture reduces quantity of energy required for cooking and drying

Flavour generation represents a challenge
Flavour is one of the major drivers of consumer preference

However, there are many other factors of similar importance

Colour, shape, texture, nutritive value, safety…

Louis-Leopold Boilly, Les Cinq Sens (The Five Senses), 1823.
To illustrate how different approaches help us to get a better insight on the role of ingredients and process parameters on desirable food quality attributes

- **Holistic approach based on experimental design**
  - Identification of key parameters driving different attributes
  - Identification of the link between different attributes

- **Targeted approaches based on labelling experiments**
  
  **CAMOLA experiments:** contribution of sugars
  
  ![CAMOLA reaction diagram](image)
  
  **Labelling studies with amino acids:** contribution of proline and ornithine
  
  ![Amino acid structure](image)
Holistic approach:
Flavour ⇔ Recipe/process parameters ⇔ Other key product attributes

Recipe parameters
- pH
- Ratio Rha/Lys
- Phosphate

Extrusion parameters
- Moisture levels
- Screw Speed
- Temperature
- Residence time
- Slurry vs. dry

Extrusion

Drying

Milling

Final product

<table>
<thead>
<tr>
<th>pH</th>
<th>6.4</th>
<th>7.7</th>
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<tbody>
<tr>
<td>Rha:Lys</td>
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<td>PO4 (mol/kg)</td>
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<td>Moisture (%)</td>
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<td>Screw speed (rpm)</td>
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<td>400</td>
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<td>Temp. (°C)</td>
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<td>Barrel length</td>
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<tr>
<td>Addition</td>
<td>Dry</td>
<td>Slurry</td>
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</table>

Product characterisation
- Texture
- Crispness
- Colour
- Flavour
- Acrylamide
- Starch degradation
- Granulometry
- Viscosity
- Sensory

Fractional factorial design:
- 32 instead of 576 trials
Modelling furaneol yield & rhamnose degradation by estimating
- all main effects and
- two-factor interactions
of 3 recipe & 5 process parameters
Correlation matrix
First indication about correlation degree between product properties

Furaneol is positively correlated with the degradation of rhamnose and colour development.

SME is negatively correlated with viscosity.
Conversion of rhamnose to furaneol can be modulated through changes of extrusion and/or recipe parameters.
## Furaneol vs Viscosity

<table>
<thead>
<tr>
<th>Furaneol ext / mol%</th>
<th>Furaneol dry / mol%</th>
<th>Visco5min / mPas</th>
<th>ViscoMax / mPas</th>
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</thead>
<tbody>
<tr>
<td>pH 6.4</td>
<td>pH 7.7</td>
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<td>Rha:Lys 3:0</td>
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<td>Phosphate 0.035 mol/kg</td>
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<td>17% H2O</td>
<td>20% H2O</td>
<td>300 1/min</td>
<td>120 °C-Long</td>
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<tr>
<td>23% H2O</td>
<td>5% H2O</td>
<td>400 1/min</td>
<td>120 °C</td>
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<td>150 °C</td>
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<td>500 1/min</td>
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<td>Dry Slurry</td>
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</table>

Texture = f(SME)
Furaneol vs Acrylamide
pH and amino acids most interesting mitigation options

- Furaneol ext / mol%
- Furaneol dry / mol%
- Acrylamide

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<th>120 °C-Long</th>
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<th>135 °C</th>
<th>150 °C</th>
<th>Dry</th>
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- **Holistic approach based on experimental design**
  - Identification of key parameters driving different attributes
  - Identification of the link between different attributes

- **Targeted approaches based on labelling experiments**
  - CAMOLA experiments: contribution of sugars
  - Labelling studies with amino acids: contribution of proline and ornithine
Targeted approach: Why labeling experiments upon extrusion?

Shear and non-shear systems behave differently

**Non shear system**
Wheat flour, Glc, Pro, Lys, Phosphate in water

- Freeze-drying
- Heating (135°C/17% or 20%)
- Analysis by GC-TOF-MS

**Extruded system**
Wheat flour, Glc, Pro, Lys, Phosphate dry mix

- Extruded at 135°C/150°C
- 17%/20%
- 300 rpm/400 rpm
- Analysis by GC-TOF-MS
Generation of roast-smelling compounds

2-Acetyl-1-pyrroline
\((\text{OT}_{\text{air}} \sim 0.02 \text{ ng/L}; \text{OT}_{\text{starch}} \sim 7.3 \text{ ng/kg})\)

2-Acetyl-1(or 3),4,5,6-tetrahydropyridine
\((\text{OT}_{\text{air}} \sim 0.06 \text{ ng/L}; \text{OT}_{\text{starch}} \sim 54 \text{ ng/kg})\)

2-Propionyl-1-pyrroline

2-Propionyl-1(or 3),4,5,6-tetrahydropyridine

(Hofmann et al., 1995)
(Rychlik; Schieberle & Grosch, 1998)
1-Pyrroline as key intermediate

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2-Propionyl-1(or 3),4,5,6-tetrahydropyridine

1-pyrroline

proline

(Hofmann et al., 1995)
(Rychlik; Schieberle & Grosch, 1998)
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2-Propionyl-1(or 3),4,5,6-tetrahydropyridine

(Hofmann et al., 1995)
(Rychlik; Schieberle & Grosch, 1998)
Dry blend:  
Rice flour  
Glyc (0.05 mol/kg)  
Pro (0.01 mol/kg)  
Orn (0.01 mol/kg)  
Phosphate  
CaCO₃

Slurry:  
Water  
[^12]C₆-glucose (0.1 mol/kg)  
or  
[^12]C₆-glucose (0.05 mol/kg)  
&  
[^13]C₆-glucose (0.05 mol/kg)

Clextral BC-21 twin screw extruder

Temperature: 135°C  
Moisure: 20%  
Screw speed: 400 rpm  
Throughput: 15 kg/h

Sample preparation:  
Ground sample (1g)  
anhydr. Na₂SO₄ (4g)  
Water (6 ml)

Analysis by  
SPME-GCxGC/TOFMS

• Extraction: SPME StableFlex DVB/CAR/PDMS; 2 cm  
• Column setup: DB-1701+DB-FFAP / or HP-5MS Inert  
• Modulation time: 6 s  
• Detection: LECO Pegasus® IV Time-of-Flight MS  
• Acquisition rate: 200 scans/s and 10 scans/s  
• Detector voltage: 1750 V
2-acetyl-1-Pyrroline
2-acetyl-1-Pyrroline
2-acetyl-1-Pyrroline

![Diagram of 2-acetyl-1-pyrroline](image)

- **Relative Intensity (%)**
  - M: 111
  - M+1: 112
  - M+2: 113
  - M+3: 114
  - M+4: 115
  - M+5: 116
  - M+6: 117

- **Chart**
  - 53% at M
  - 36% at M+1
  - 8% at M+2
  - 0% at M+3, M+4, M+5, M+6

**Formulas**

- ![1-pyrroline](image)
- ![Acetyl-1-pyrroline](image)
Acylation of pyrroline via C₂ sugar fragments

Formation of 2-AP from 1-pyrroline and acetylformoin (Rewicki et.al. 1993)

Formation of 2-AP from 1-pyrroline and hydrated 2-oxopropanal (Hofmann & Schieberle 1998)
Formation of 2-AP incorporating C₃ sugar fragment

Formation of 2-AP from 1-pyrroline and 2-oxopropanal (Hofmann & Schieberle 1998)
Formation of 2-AP incorporating C₃ sugar fragment

Hofmann & Schieberle 1998: [³¹⁵C₆]-glucose / proline; phosphate butter pH 7, dry heating - 160°C/10 min

Extruded system: [³¹⁵C₆]-glucose / [³¹²C₆]-glucose / proline / ornithine; Phosphate pH 7.2, Low moisture - 135°C/30 sec 20% moisture

Model rice system: [³¹⁵C₆]-glucose (0.3M) / proline (0.1M); Phosphate pH 5.5 and 8.2, Low moisture - 135°C/20 min 20% moisture

Acidic ~3 : 1 (23%)  
Alcaline ~1 : 1 (46%)
2-propionyl-1-Pyrroline

1-pyrroline
2-propionyl-1-Pyrroline

CAMOLA

1-pyrroline
2-propionyl-1-Pyrroline

<table>
<thead>
<tr>
<th>relative Intensity (%)</th>
<th>M</th>
<th>M+1</th>
<th>M+2</th>
<th>M+3</th>
<th>M+4</th>
<th>M+5</th>
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1-pyrroline

2-propionyl-1-pyrroline

M 125   M+1 126   M+2 127   M+3 128   M+4 129   M+5 130   M+6 1131
Formation of 2-PP incorporating C$_3$ sugar fragment

Formation of 2-PP from 1-pyrroline and hydrated 2-oxobutanal (Hofmann & Schieberle 1998)
2-Acetyl-1(or 3),4,5,6-tetrahydropyridine
2-Propionyl-1(or 3),4,5,6-tetrahydropyridine

1-pyrroline
2-Acetyl-1(or 3),4,5,6-tetrahydropyrididine
2-Propionyl-1(or 3),4,5,6-tetrahydropyrididine
2-Acetyl-1(or 3),4,5,6-tetrahydropyridine
2-Propionyl-1(or 3),4,5,6-tetrahydropyridine
Formation of 2-ATHP and 2-PTHP

Formation of 2-ATHP from proline and acetylformoin (Rewicki et.al. 1993)

Formation of 2-ATHP and 2-PTHP from 1-pyrroline and sugar fragments (1-hydroxy-2-propanone or 1-hydroxy-2-butanone (Hofmann & Schieberle 1998)
Summary - CAMOLA

\[
\begin{align*}
\text{1-pyrroline} & \quad \text{methylglyoxal} \quad \text{Acetylformoin} \\
& \quad \text{hydroxyacetone} \quad \text{acetylformoin} \\
2\text{-oxobutanal} & \quad \text{1-hydroxy-2-butanal} \\
& \quad \text{2-oxobutanol}
\end{align*}
\]
To illustrate how different approaches help us to get a better insight on the role of ingredients and process parameters on desirable food quality attributes

• Holistic approach based on experimental design
  • Identification of key parameters driving different attributes
  • Identification of the link between different attributes

• Targeted approaches based on labelling experiments

CAMOLA experiments: contribution of sugars

Labelling studies with amino acids: contribution of proline and ornithine
**Experimental setup**

**Contribution of glucose - CAMOLA**

- **Dry blend:** Rice flour, Gly (0.05 mol/kg), Pro (0.01 mol/kg), Orn (0.01 mol/kg), Phosphate, CaCO₃

- **Slurry:**
  - [¹²C₆]-glucose (0.1 mol/kg) or [¹²C₆]-glucose (0.05 mol/kg) & [¹³C₆]-glucose (0.05 mol/kg)

**Eurolab 16 (Thermo Fisher Scientific) twin screw extruder**

- **Throughput:** 15 kg/h
- **Temperature:** 135°C
- **Moisur.:** 20%
- **Screw speed:** 400 rpm

**Contribution of amino acids**

- **Dry blend:** Rice flour, glucose (0.1 mol/kg), Gly (0.05 mol/kg), Phosphate, CaCO₃

- **Slurry:**
  - Pro (0.01 mol/kg) or Pro + Orn (0.01 mol/kg each) or [U-¹³C₅, ¹⁵N]-Pro (0.01 mol/kg) or [U-¹³C₅, ¹⁵N]-Pro + Orn (0.01 mol/kg each)

**BC-21 (Clextral) twin screw extruder**

- **Throughput:** 1.2 kg/h
- **Temperature:** 135°C
- **Moisur.:** 20%
- **Screw speed:** 400 rpm
## Contribution of Pro and Orn - Literature data

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<th>Amino acid</th>
<th>Heating Method</th>
<th>Carbonyl</th>
<th>Reference</th>
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<td>Glc &lt; 0.3 µg</td>
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<td>Roasting 150°C/ 10 mn</td>
<td>Frc 15 µg</td>
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<td>Baking</td>
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<td></td>
<td>Extrusion 165°C; 18%</td>
<td>MeGlx 0.1 - 27 µg/kg</td>
<td>2.1 -4900 µg/kg</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Glc 0.3 - 22 µg/kg</td>
<td>1.7 -1200 µg/kg</td>
</tr>
<tr>
<td></td>
<td>Reflux 2h</td>
<td>MeGlx 43 µg</td>
<td>&lt; 0.3 µg</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Frc 53 µg</td>
<td>&lt; 0.3 µg</td>
</tr>
<tr>
<td></td>
<td>Foam</td>
<td>Wheat dough</td>
<td>9.4 µg</td>
</tr>
<tr>
<td></td>
<td>Extrusion 165°C; 18%</td>
<td>MeGlx 1.5 - 120 µg/kg</td>
<td>n.d. -120 µg/kg</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Glc 3 - 290 µg/kg</td>
<td>n.d. -100 µg/kg</td>
</tr>
</tbody>
</table>

- **MeGlx**: Methylglyoxal
- **FrC**: Formic acid
- **Glc**: Glucose
- **n.d.** : Not determined
Contribution of Pro alone

Glc/Pro*

Proline significantly enhances generation of all three compounds.
Contribution of Pro & Orn

Proline significantly enhances generation of all three compounds

Ornithine contributes to generation of all three compounds namely of 2-AP
Relative concentration generated from Pro & Orn as compared to matrix

- Orn is much better precursor of 2-AP than Pro
- Orn slightly inhibits generation of 2-AP from Pro

- Efficiency of Orn to generate 2-ATHP is comparable to Pro
- Orn slightly promotes generation of 2-ATHP from Pro

- Orn is better precursor of 2-PP than Pro
- Orn slightly promotes generation of 2-PP from Pro
Only 1-Pyrroline as key intermediate?

Glc/Pro*/Orn

0% 25% 50% 75% 100%

Matrix Pro Orn

2-AP 2-AP 2-AP

8 4 2

2-ATHP 2-ATHP 2-ATHP

88 56 32

2-PP 2-PP 2-PP

43 43 43

Only 1-Pyrroline as key intermediate?
Only 1-Pyrroline as key intermediate?

Glc/Pro*/Orn

<table>
<thead>
<tr>
<th></th>
<th>2-AP</th>
<th>2-ATHP</th>
<th>2-PP</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>8</td>
<td>4</td>
<td>2</td>
</tr>
<tr>
<td>25%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>50%</td>
<td></td>
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</tr>
<tr>
<td>75%</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>100%</td>
<td>88</td>
<td>43</td>
<td>66</td>
</tr>
</tbody>
</table>

Matrix  Pro  Orn

H₂N-ornithine → H₂N-4-aminobutanal → 0.8 1-pyrroline → proline

2-AP: 2-aminopyridine
2-ATHP: 2-aminothiazole-5-carboxylic acid
2-PP: 2-aminopropionitrile

Matrix  Pro  Orn
Conclusions

Combination of different approaches is necessary to modulate flavour generation upon food processing and to optimize it with respect to other food product attributes (colour, texture, …)

**Holistic approaches**
- based on experimental design and a global product characterisation allow rapid optimisation:
  - Identification of key parameters driving different product attributes
  - Identification of links between attributes

**Targeted approaches**
- based on labelling experiments allow:
  - understanding of formation pathways during food processing
  - identification/verification of key precursors and intermediates
Thanks to…

- Daniel Festring  PTC Orbe  Flavour analysis, processing
- Thierry Dufosse  PTC Orbe  Flavour analysis
- Hélène Chanvrier  PTC Orbe  Structure, texture analysis
- Werner Pfaller  PTC Orbe  Extrusion expert
- Andreas Rytz  NRC Lausanne  Experimental design
- Silke Illmann  Univ. Karlsruhe  Diploma work (Prof. H. Schuchmann)
- Lara S. Kirsch  TU Munich  Master thesis (Prof. T. Hofmann)

… and to you for your kind attention!

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