The influence of a different fatty acid profile on the crystallization of milk fat as measured by pNMR, DSC and XRD

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Milk fat (1)

- Importance: 25% of total fat intake (Belgium)

- Major fatty acid composition:

<table>
<thead>
<tr>
<th>Milk fat</th>
<th>%</th>
<th>Milk fat</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C10:0</td>
<td>2.99</td>
<td>C18:1c9</td>
<td>20.1</td>
</tr>
<tr>
<td>C12:0</td>
<td>4.03</td>
<td>C18:2c9c12</td>
<td>1.19</td>
</tr>
<tr>
<td>C14:0</td>
<td>9.30</td>
<td>C18:3c9c12c15</td>
<td>0.271</td>
</tr>
<tr>
<td>C16:0</td>
<td>27.7</td>
<td>C18:1t10-11</td>
<td>1.83</td>
</tr>
<tr>
<td>C18:0</td>
<td>8.94</td>
<td>C18:2c9t11</td>
<td>0.675</td>
</tr>
</tbody>
</table>


Changing fatty acid composition by changing the feed composition
Milk fat (2)

- Enriching milk fat with unsaturated fatty acids
  - More sensitive to oxidation
  - Changed physical properties of dairy products
  - Interfering in production process
Milk fat (2)

- Enriching milk fat with unsaturated fatty acids:
  - More sensitive to oxidation
  - Changed physical properties of dairy products
    - Cold-spreadability of butter (e.g. Baer et al. (2001). J. Dairy Sci., 84:345-353)
    - Softer texture of butter, yoghurt, ice cream and cheese (e.g. Chen et al. (2001). J. Agr. Food Chem., 52:3422-3428)
Milk fat (2)

- Enriching milk fat with unsaturated fatty acids
  - More sensitive to oxidation
  - Changed physical properties of dairy products
  - Interfering in production process
    - Decreased viscosity of ice cream mix (Gonzalez et al. (2003). J. Dairy Sci. 86:70-77)
Milk fat (2)

- Enriching milk fat with unsaturated fatty acids
  - More sensitive to oxidation
  - Changed physical properties of dairy products
  - Interfering in production process

- Aim of the experiment:
  - Investigating the influence of a more unsaturated fatty acid composition on the crystallization behaviour of milk fat
Setup of the experiment (1)

- Production of milk with a different fatty acid profile

<table>
<thead>
<tr>
<th>Fatty acids</th>
<th>Control (g/100 g FAME)</th>
<th>Linseed (g/100 g FAME)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FEED SFA</td>
<td>32.59</td>
<td>10.66</td>
</tr>
<tr>
<td>MUFA</td>
<td>39.28</td>
<td>17.63</td>
</tr>
<tr>
<td>PUFA</td>
<td>28.08</td>
<td>71.71</td>
</tr>
<tr>
<td>n-6</td>
<td>24.48</td>
<td>18.99</td>
</tr>
<tr>
<td>n-3</td>
<td>3.6</td>
<td>52.71</td>
</tr>
<tr>
<td>n-6/n-3</td>
<td>6.80</td>
<td>0.36</td>
</tr>
<tr>
<td>P/S</td>
<td>2.07</td>
<td>8.38</td>
</tr>
</tbody>
</table>

- Isolation of milk fat with BDI method

<table>
<thead>
<tr>
<th>Fatty acids</th>
<th>C milk (g/100 g FAME)</th>
<th>U milk (g/100 g FAME)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SFA</td>
<td>71.8</td>
<td>61.3 \downarrow</td>
</tr>
<tr>
<td>MUFA</td>
<td>25.3</td>
<td>33.4 \uparrow</td>
</tr>
<tr>
<td>PUFA</td>
<td>2.92</td>
<td>5.28 \uparrow</td>
</tr>
<tr>
<td>LA</td>
<td>1.30</td>
<td>1.67</td>
</tr>
<tr>
<td>LNA</td>
<td>0.38</td>
<td>1.08</td>
</tr>
<tr>
<td>Total CLA</td>
<td>0.71</td>
<td>1.86</td>
</tr>
<tr>
<td>n-6/n-3</td>
<td>2.98</td>
<td>1.55 \downarrow</td>
</tr>
</tbody>
</table>
Crystallization behaviour

- SFC profile (pNMR)
- Melting profile (DSC)
- Isothermal crystallization behaviour
  - Mechanisms: - one-step crystallization: 22°C
    - two-step crystallization: 5°C
  - Techniques: - pNMR
    - XRD
SFC profile

≠ SFC: 13.5%: - storage temperature of butter, cream
- ripening temperature of ice cream

≠ SFC: 4.3%: consumption temperature

Solid fat content (%)

Temperature (°C)

Control milk fat
Unsaturated milk fat

ILVO – Technology and Food
http://www.ilvo.vlaanderen.be/
Temperature profile:

- Isothermal: 80°C (15min)
- -0.5°C/min until -50°C
- Isothermal - 50°C (5min)
- +5°C/min until 80°C

= melting curve
DSC: melting profile

<table>
<thead>
<tr>
<th></th>
<th>C AMF (%)</th>
<th>U AMF (%)</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>HMT</td>
<td>40.39</td>
<td>25.86</td>
<td>0.002</td>
</tr>
<tr>
<td>MMT</td>
<td>31.42</td>
<td>26.74</td>
<td>0.057</td>
</tr>
<tr>
<td>LMT</td>
<td>28.19</td>
<td>47.38</td>
<td>&lt;0.001</td>
</tr>
</tbody>
</table>

Temperature (°C):
-40 -20 0 20 40 60 80

Heat flow (W/g):
-1.4 -1.2 -1.0 -0.8 -0.6 -0.4 -0.2 0.0 0.2 0.4 0.6 0.8 1.0 1.2

-10°C < MMT < 20°C

< 10°C: LMT

> 20°C: HMT
Isothermal pNMR (22°C)

One-step crystallisation

\( \text{SFC}_{\text{max}} = 14.7\% \)

\( \text{SFC}_{\text{max}} = 8.72\% \)
Isothermal pNMR (5°C)

Two-step crystallisation

SFC_{int} = 28.2%

SFC_{int} = 42.3%

SFC_{max} = 57.2%

SFC_{max} = 45.3%
Wide Angle X-Ray Diffraction

- Information about lateral chain packaging

Hexagonal $\alpha$
Orthorhombic $\beta'$
Triclinic $\beta$
WAXD Control AMF (22°C)

\[ \beta' \]
\[(4.25 + 3.82 \text{ Å})\]

\[ \alpha \]
\[(4.12 \text{ Å})\]

WAXD Unsaturated AMF (22°C)

\[ \beta' \]
\[(4.28 + 3.83 \text{ Å})\]

WAXD Control AMF (5°C)

\[ \beta' \]
\[(4.25 + 3.82 \text{ Å})\]

\[ \alpha \]
\[(4.12 \text{ Å})\]

WAXD Unsaturated AMF (5°C)

\[ \alpha \]
\[(4.12 \text{ Å})\]

\[ \beta' \]
\[(3.82 \text{ Å})\]
Conclusion

- Crystallization behaviour characterized with:
  - pNMR
  - DSC
  - XRD

- Knowledge will be used for the production of dairy products with a more unsaturated fatty acid composition
This work is the result of a cooperation of:

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