Advanced Separation Techniques for Bioproducts

Achievements and Perspectives in UPB

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Outline

• Achievements
  • POSCCE Project *o-BIOGLYVAL ID 652*
  • High Vacuum Distillation – DSL 5 Plant
  • Molecular Distillation – KDL 5 Plant
  • Some Experimental results

• Perspectives
  • POC Project *ASPiRE P_37_449*
  • Supercritical CO₂ Fluid/Solid Extraction

• Conclusions
0. Introduction
Thank you for inviting me to this Exploratory Seminar DIASPORA 2016

University POLITEHNICA of Bucharest
Largest and oldest technical university in Romania
16 faculties in main engineering fields
25,000 students
2,000 academics
Faculty of Applied Chemistry and Material Science
Department of Chemical and Biochemical Engineering
Greetings from our team for you!
NEW POSSIBILITIES FOR SUSTAINABLE INTEGRATION OF BIODIESEL PRODUCTION. GLYCEROL AND ω-FATTY ACIDS VALORISATION  ω- BIOGLYVAL

Director : Dr. Jordi BONET-RUIZ – University of Barcelona
Period : November 2010 – September 2014

http://bioglyval.upb.ro
Laboratory for Innovative Products and Processes
Laboratory for Innovative Products and Processes

Molecular distillation KDL 5 plant
(Short Path Distillation)

High Vacuum Fractionation Plant
DSL 5
Omega-3 fatty acids are PolyUnsaturated Fatty Acids (PUFA) with a double bond (C=C) at the third carbon atom from the end of the carbon chain.

Target:
To obtain a product with a higher concentration of ω-3 fatty acids esters, using high vacuum distillation techniques.
2. Problem presentation

*Sigma Profile of ω-3 fatty acids produced in COSMOthermX*

Omega-3 fatty acids
CLASSIC DISTILLATION METHODS:
- batch distillation at ~ atmospheric pressure
- superheated stream distillation
- fractional distillation... and many other

THERMAL DEGRADATION OF PUFA:
deacarbonylation and polymerization,
low quality products

The solution:
HIGH VACUUM DISTILLATION!!
3. **PUFA esters synthesis from fish oil**

Fatty acids alkyl esters are easier to separate than fatty acids because they are more volatile.

**Transesterification reaction of fish oil with methanol/ethanol in alkaline-based catalysis**

\[
\begin{align*}
\text{fish oil} & \quad \text{methanol} \quad \text{KOH} \quad \text{FAME} \quad \text{glycerol} \\
H_2C - \text{OOOCR}_1 & \quad \quad + \quad 3\text{CH}_3\text{OH} & \quad \quad \rightarrow & \quad R_1\text{COOCH}_3 & \quad + \quad \text{H}_2\text{C} - \text{OH} \\
H_2C - \text{OOOCR}_2 & \quad \quad + \quad 3\text{CH}_3\text{OH} & \quad \quad \rightarrow & \quad R_2\text{COOCH}_3 & \quad + \quad \text{H}_2\text{C} - \text{OH} \\
H_2C - \text{OOOCR}_3 & \quad \quad + \quad 3\text{CH}_3\text{OH} & \quad \quad \rightarrow & \quad R_3\text{COOCH}_3 & \quad + \quad \text{H}_2\text{C} - \text{OH}
\end{align*}
\]

*FAME = Fatty Acids Methyl Esters

\[
\begin{align*}
\text{fish oil} & \quad \text{ethanol} \quad \text{KOH} \quad \text{FAEE} \quad \text{glycerol} \\
H_2C - \text{OOOCR}_1 & \quad \quad + \quad 3\text{C}_2\text{H}_5\text{OH} & \quad \quad \rightarrow & \quad R_1\text{COOC}_2\text{H}_5 & \quad + \quad \text{H}_2\text{C} - \text{OH} \\
H_2C - \text{OOOCR}_2 & \quad \quad + \quad 3\text{C}_2\text{H}_5\text{OH} & \quad \quad \rightarrow & \quad R_2\text{COOC}_2\text{H}_5 & \quad + \quad \text{H}_2\text{C} - \text{OH} \\
H_2C - \text{OOOCR}_3 & \quad \quad + \quad 3\text{C}_2\text{H}_5\text{OH} & \quad \quad \rightarrow & \quad R_3\text{COOC}_2\text{H}_5 & \quad + \quad \text{H}_2\text{C} - \text{OH}
\end{align*}
\]

*FAEE = Fatty Acids Ethyl Esters*
4. Gas chromatography analysis

FAME FAEE → Gas-chromatography analysis → GCXGC - Mass Spectrometer Detector → FAME/FAEE mixture distribution
### Example of FAEE concentrate obtained from molecular distillation plant KDL5

<table>
<thead>
<tr>
<th>Component</th>
<th>Mass fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethyl stearate (C18:0)</td>
<td>0.0302</td>
</tr>
<tr>
<td>Ethyl oleate (C18:1)</td>
<td>0.0123</td>
</tr>
<tr>
<td>Ethyl linoleate (C18:2)</td>
<td>0.0165</td>
</tr>
<tr>
<td>Ethyl linolenate (C18:3)</td>
<td>0.0242</td>
</tr>
<tr>
<td>Ethyl octadecatetrananoate (C18:4)</td>
<td>0.0598</td>
</tr>
<tr>
<td>Ethyl eicosenoate (C20:1)</td>
<td>0.0359</td>
</tr>
<tr>
<td>Ethyl eicosatetrananoate (C20:4)</td>
<td>0.0193</td>
</tr>
<tr>
<td><strong>Ethyl eicosapentanoate (C20:5-EPA)</strong></td>
<td><strong>0.3980</strong></td>
</tr>
<tr>
<td><strong>Ethyl docosapentanoate (C22:5)</strong></td>
<td><strong>0.0817</strong></td>
</tr>
<tr>
<td><strong>Ethyl docosahexanoate (C22:6-DHA)</strong></td>
<td><strong>0.3219</strong></td>
</tr>
</tbody>
</table>
5. Evaluation of critical and physical - chemical properties of PUFA esters mixture

- Critical properties
- Normal boiling point
- Critical temperature
- Critical pressure
- Critical volume
- Critical density
- Vapor pressure
- Heat capacity
- Thermal conductivity
- Density
- Thermal diffusivity
- Enthalpy of vaporization

Modeling, simulation and optimization of high vacuum distillation: high vacuum fractionation and molecular distillation
Molecular distillation (short path distillation) is a very low pressure distillation technique such as the distance between the heating surface and the cooling surface is smaller than the mean free path of the light molecules, but larger of that of heavy molecules.

The **mean free path** is the distance that a molecule travels between two successive collisions.

\[ \lambda = \frac{k_B \cdot T}{\sqrt{2\pi d^2 \rho}} \]

- \( k_B \) = Boltzmann constant \( 1.3810^{-23} \) [m\(^2\) kg s\(^{-2}\) K\(^{-1}\)]
- \( d \) = diameter of the molecules (m)
- \( P \) = system pressure (\( 10^{-6} \) bar = 0.1 Pa)
- \( T \) = system temperature

\( \lambda_{\text{light}} \) and \( \lambda_{\text{heavy}} \) refer to the mean free path for light and heavy molecules, respectively.

For FAME/FAEE – LOW VAPOR PRESSURE SPECIAL DISTILLATION TECHNIQUES – MOLECULAR DISTILLATION, a distance of 2-3 cm is often used to ensure the effectiveness of the process.
ALA (C18:3) – Light molecule
DHA (C22:6) – Heavy molecule

ALA diameter of molecule = 8Å
\( \lambda = 3.81 \text{ cm} \)
(at \( T = 100^\circ \text{C}, \ p = 10^{-6} \text{ bar} = 0.1 \text{ Pa} \))

DHA diameter of molecule = 10Å
\( \lambda = 1.66 \text{ cm} \)
(at \( T = 100^\circ \text{C}, \ p = 10^{-6} \text{ bar} = 0.1 \text{ Pa} \))
Features Wiped Film Evaporator

- Residence time of a few seconds with a narrow spread, an important feature for heat sensitive products
- Required evaporation is achieved in a single pass, avoiding product recirculation and possible degradation
- Scale formation on the heat transfer surface is reduced due to the intense agitation of the liquid film
- Excellent turn down capability
- Low product holdup, ideal for hazardous applications
- Operating pressure as low as 1 mbar and operating temperature up to 400 °C
- Special designs for clean room applications
6. **Principle of high vacuum distillation**

High vacuum distillation is a special technique of purifying a mixture, by lowering the pressure and so the components boiling points.

- Thin film distillation (p>10^{-2} mbar i.e. ~1 Pa) and fractionation
- Molecular distillation (p<10^{-3} mbar, i.e. ~0.1Pa)

1. Feed
2. Distillate
3. Residue
4. Heating
5. Cooling
6. Vacuum
High Vacuum Fractionation

The mixture to be separated is dispersed uniformly on the walls of the evaporation unit by means of the rollers attached to the wiper basket.

• Vapours of more volatile compounds are evacuated in the exterior packed column and then condensed
• Heavy product falls down in evaporator space

Fractionation column with Montz structured packing Type A3 -500M – for low pressure drop

Distillate
Bottoms product
Montz A3-500 Structured packing performance

F- Factor = $w_g \rho^{0.5} [Pa^{0.5}]$

$w_g = \text{superficial vapor (gas) velocity, [m/s], } \rho = \text{density, [kg/m}^3\text{]}$
F- Factor = $w_g \cdot \rho^{0.5} \text{ [Pa}^{0.5}]$

$W_g =$ superficial vapor (gas) velocity, $[\text{m/s}]$, $\rho =$ density, $[\text{kg/m}^3]$
7. PUFA esters separation scheme

MeOH EtOH

Fish oil

Transesterification

FAME FAEE

High vacuum fractionation

Distillate product

High vacuum fractionation

Bottom product

High vacuum fractionation

Distillate product

Bottom product

Molecular distillation

Light product

Heavy product
High vacuum fractionation plant DSL5

- External condenser
- Rectification column
- Product feed
- Evaporation unit
- Vacuum pump
Molecular distillation plant KDL5

- **Product feed**
- **Molecular distillation unit**
- **Gases trap**
- **Vacuummeter**
- **Diffusion pump**
- **Light fractions collector**
- **Heavy fractions collector**
- **Rotary Vacuum pump**
8. Experimental results – FAME/FAEE esters synthesis

**Reaction conditions:**
Methanol/Ethanol:fish oil molar ratio 6:1
KOH 1% based on oil mass
Reaction time 2h
Temperature: 75°C (MeOH) 90°C (EtOH)

**Legend**
1-4 neck flask  
2- funnel  
3- thermometer  
4- mechanical stirrer  
5- condenser  
6- electric heating

**Legend**
1- separating funnel;  
2- Erlenmeyer flask

**Liquid-liquid separation**

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Transesterification reaction

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Experimental setup with reaction conditions explained.
Extraction with methanolic solution

Vacuum dehydration

Second tranesterification step with 30\% MeOH and 30\% KOH

Liquid-liquid separation

Vacuum dehydration
Experimental results – PUFA esters chromatographic analysis

### GC-MS analysis

#### PUFA methyl esters

<table>
<thead>
<tr>
<th>Ester</th>
<th>C14:0</th>
<th>C16:0</th>
<th>C16:1</th>
<th>C16:4</th>
<th>C18:3</th>
<th>C18:2</th>
<th>C18:1</th>
<th>C18:0</th>
<th>C20:5</th>
<th>C20:4</th>
<th>C20:1</th>
<th>C22:6</th>
<th>C22:5</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>7.11</td>
<td>17.38</td>
<td>14.42</td>
<td>1.12</td>
<td>5.04</td>
<td>2.51</td>
<td>11.47</td>
<td>3.99</td>
<td>16.11</td>
<td>1.9</td>
<td>1.14</td>
<td>15.98</td>
<td>2.98</td>
</tr>
</tbody>
</table>

#### PUFA ethyl esters

<table>
<thead>
<tr>
<th>Ester</th>
<th>C14:0</th>
<th>C16:1</th>
<th>C16:0</th>
<th>C16:4</th>
<th>C18:3</th>
<th>C18:2</th>
<th>C18:1</th>
<th>C18:0</th>
<th>C20:5</th>
<th>C20:4</th>
<th>C20:1</th>
<th>C22:6</th>
<th>C22:5</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>6.75</td>
<td>12.14</td>
<td>17.22</td>
<td>3.4</td>
<td>1.2</td>
<td>7.68</td>
<td>3.84</td>
<td>3.85</td>
<td>15</td>
<td>1.63</td>
<td>1.16</td>
<td>14.26</td>
<td>2.96</td>
</tr>
</tbody>
</table>

### GC-MS analysis

#### PUFA methyl esters

- **FAME**
- **FAEE**

#### PUFA ethyl esters
Experimental results –

FAME/FAEE mixtures high vacuum separation

DSL5 - Thin Film Distillation Plant
KDL5 - Molecular Distillation Plant
**Separation conditions:**

- **Feed type:**
  - fish oil methyl esters/
  - fish oil ethyl esters
- **Feed volume:**
  - 1500 mL (FAME)
  - 1350 mL (FAEE)
- **Pressure:** \(1.2 \times 10^{-1} \text{ mbar}\)
- **Oil temperature in evaporator unit:**
  - 205ºC (FAME)
  - 215ºC (FAEE)
- **Separation time:**
  - 3 h
  - 2.45 h

**Distillate product:**

- **Product type:** Saturated and unsaturated fatty acids methyl/ethyl esters
- **Product volume:**
  - 636 mL (FAME)
  - 566 mL (FAEE)

**Bottom product:**

- **Product type:** PUFA methyl/ethyl esters
- **Product volume:**
  - 760 mL (FAME)
  - 700 mL (FAEE)
Separation conditions:

Feed type:
- fish oil methyl esters/
- fish oil ethyl esters
Feed volume:
- 1400 mL (FAME)
- 1300 mL (FAEE)
Pressure: \(1.2 \cdot 10^{-1} \text{ mbar}=12 \text{ Pa}\)
Oil temperature in evaporator unit:
- 205ºC (FAME)
- 215ºC (FAEE)
Separation time:
- 3 h (FAME)
- 2.45 h (FAEE)

Distilate product:

Product type:
- Saturated and unsaturated fatty acids methyl/ethyl esters
Product volume:
- 500 mL (FAME)
- 470 mL (FAEE)

Bottom product:

Product type:
- PUFA methyl/ethyl esters
Product volume:
- 780 mL (FAME)
- 750 mL (FAEE)
**Separation conditions:**

**Feed type:**
Bottom products from DSL5 batch1 and DSL5 batch2

**Feed volume:**
1540 mL (FAME)
1450 mL (FAEE)

**Pressure:** 7.2 $ \cdot 10^{-2}$ mbar = 7.2 Pa

**Oil temperature in evaporator unit:**
210ºC (FAME)
220ºC (FAEE)

**Separation time:**
4 h (FAME)
3.5 h (FAEE)

**Distillate product:**

Product type: Saturated and unsaturated fatty acids methyl/ethyl esters

Product volume:
550 mL (FAME)
500 mL (FAEE)

**Bottom product:**

Product type: PUFA methyl/ethyl esters

Product volume:
940 mL (FAME)
900 mL (FAEE)
DSL5 1st distillation step products

DSL5 Batch 1 products

DSL5 2nd distillation step products

DSL5 Batch 2 products
**Separation conditions:**

Feed type: **Bottom product from DSL5 2nd distillation step**

Feed volume: 940 mL (FAME)  
900 mL (FAEE)

Pressure: $1 \cdot 10^{-3}$ mbar

Evaporator unit temperature: 105-125ºC

Separation time: 1.45 h

**Light product:**

Product type: Saturated and unsaturated fatty acids methyl/ethyl esters

Product volume: 280 mL (FAME)  
250 mL (FAEE)

**Heavy product:**

Product type: PUFA methyl esters  
PUFA ethyl esters

Product volume: 615 mL (FAME)  
590 mL (FAEE)
Result: 5 samples of distillate and bottom products collected at 5 different temperatures (105°C, 110°C, 115°C, 120°C, 125°C)
Experimental results – FAME/FAEE Gas chromatography analysis

- The fatty acids esters composition is analyzed with Agilent Technologies 7890A Chromatograph with Mass Spectrometer Detector Agilent Technologies 5975C.

- Chromatographic method:

| INLET | |COLUMN| |OWEN |
|-------|---|-----------------|-----------------|
| **Heater (ºC)** | 250 | **Type** | J&W122-5532, 30m x 25 μm x 0.25 μm |
| **Pressure (psi)** | 7.76 | **Flow (mL/min)** | 1 |
| **Total Flow (mL/min)** | 84 | **Pressure (psi)** | 10.5 |
| **Purge flow (mL/min)** | 3 | **Rate (ºC)** | **Value (ºC)** | **Hold time (ºC)** | **Run time (ºC)** |
| Initial | 100 | 1 | 1 | 1 |
| Ramp 1 | 5 | 160 | 1 | 14 |
| Ramp 2 | 2 | 190 | 5 | 34 |
| Ramp 3 | 2 | 200 | 20 | 59 |
| Ramp 4 | 3 | 230 | 10 | 79 |
• Raw material GC-MS chromatogram

<table>
<thead>
<tr>
<th>Component (FAEE)</th>
<th>Comp. Position (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C14:0</td>
<td>6.75</td>
</tr>
<tr>
<td>C16:0</td>
<td>12.14</td>
</tr>
<tr>
<td>C16:1</td>
<td>17.22</td>
</tr>
<tr>
<td>C16:4</td>
<td>3.4</td>
</tr>
<tr>
<td>C18:3</td>
<td>1.2</td>
</tr>
<tr>
<td>C18:2</td>
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<td>14.26</td>
</tr>
<tr>
<td>C22:5</td>
<td>2.96</td>
</tr>
</tbody>
</table>

PUFA: 35.01

**PUFA (Polyunsaturated Fatty Acids)**
1st step high vacuum fractionation - DSL5
Bottom product GC-MS chromatogram

<table>
<thead>
<tr>
<th>Composition (%)</th>
<th>C16:1</th>
<th>C16:0</th>
<th>C16:4</th>
<th>C18:3</th>
<th>C18:2</th>
<th>C18:1</th>
<th>C18:0</th>
<th>C20:5</th>
<th>C20:4</th>
<th>C20:1</th>
<th>C22:6</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>1.2</td>
<td>3.2</td>
<td>5.42</td>
<td>2.5</td>
<td>12.43</td>
<td>6.15</td>
<td>6.2</td>
<td>24.22</td>
<td>2.56</td>
<td>2.32</td>
<td>21.78</td>
<td>4.54</td>
</tr>
</tbody>
</table>

PUFA 55.42
2nd step High Vacuum Fractionation-DSL5

1st step bottom product GC-MS chromatogram

Light product GC-MS chromatogram

Bottom product GC-MS chromatogram

55.42% PUFA

83.51% PUFA
Molecular distillation – KDL5
Heavy product KDL5 GC-MS chromatogram

FAEE

105°C

FAEE

110°C

FAEE

115°C
Molecular distillation – KDL5

- Heavy product KDL5 GC-MS chromatogram

FAEE

- C20:5
- C20:4
- C20:1

120°C

- C22:6
- C22:5

86.77% PU FA

125°C

- C22:6
- C22:5

86.76% PU FA
<table>
<thead>
<tr>
<th>Experimental Plan</th>
<th>PUFA Compositions (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C20:5</td>
</tr>
<tr>
<td>Transesterification</td>
<td>15</td>
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<tr>
<td>DSL5 1st distillation step</td>
<td>24.22</td>
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<tr>
<td>DSL5 2nd distillation step</td>
<td>31.45</td>
</tr>
<tr>
<td>KDL5 distillation</td>
<td>21.04</td>
</tr>
</tbody>
</table>

**Fish oil ethyl esters 35.01%**

**DSL5 FIRST DISTILLATION**
- Bottom product 55.42%

**DSL5 SECOND DISTILLATION**
- Bottom product 83.51%

**KDL5 DISTILLATION**
- Heavy product ≈88%
- Raw material GC-MS chromatogram

<table>
<thead>
<tr>
<th>Comp position (%)</th>
<th>C14:0</th>
<th>C16:1</th>
<th>C16:0</th>
<th>C16:4</th>
<th>C18:3</th>
<th>C18:2</th>
<th>C18:1</th>
<th>C18:0</th>
<th>C20:5</th>
<th>C20:4</th>
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<td>11.47</td>
<td>3.99</td>
<td>16.11</td>
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<td>1.14</td>
<td>15.98</td>
<td>2.98</td>
</tr>
<tr>
<td>PUFA</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td><strong>42.01</strong></td>
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</table>
1st step high vacuum fractionation - DSL5
Bottom product GC-MS chromatogram

<table>
<thead>
<tr>
<th>Composition (%)</th>
<th>C16:1</th>
<th>C16:0</th>
<th>C16:4</th>
<th>C18:3</th>
<th>C18:2</th>
<th>C18:1</th>
<th>C18:0</th>
<th>C20:5</th>
<th>C20:4</th>
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<th>C22:6</th>
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<tbody>
<tr>
<td></td>
<td>2.37</td>
<td>0.65</td>
<td>-</td>
<td><strong>4.66</strong></td>
<td>3.25</td>
<td>15.66</td>
<td>6.13</td>
<td><strong>28.25</strong></td>
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<td>3.74</td>
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<tr>
<td>PUFA</td>
<td></td>
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<td></td>
<td><strong>68.2</strong></td>
</tr>
</tbody>
</table>
2\textsuperscript{nd} step high vacuum fractionation – DSL5

1\textsuperscript{st} step Bottom product GC-MS chromatogram

Top product GC-MS chromatogram

Bottom product GC-MS chromatogram

FAME

68.2% PUFA

85.42% PUFA
Molecular distillation – KDL5
Heavy product KDL5 GC-MS chromatogram

FAME

105°C
88.8% PUF A

110°C
87.9% PUF A

115°C
89.6% PUF A
Molecular distillation – KDL5

- Heavy product KDL5 GC-MS chromatogram

![FAME GC-MS chromatogram at 120°C with 89.77% PUFA](chart1.png)

![FAME GC-MS chromatogram at 125°C with 90.76% PUFA](chart2.png)
### Molecular distillation KDL 5 results - summary

<table>
<thead>
<tr>
<th>Esters Composition (%)</th>
<th>C20:5</th>
<th>C20:4</th>
<th>C20:1</th>
<th>C22:6</th>
<th>C22:5</th>
<th>Total</th>
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<tbody>
<tr>
<td>Bottom product 105°C</td>
<td>33.86</td>
<td>3.94</td>
<td>1.89</td>
<td>40.54</td>
<td>8.56</td>
<td>88.8</td>
</tr>
<tr>
<td>Bottom product 110°C</td>
<td>32.19</td>
<td>3.96</td>
<td>1.94</td>
<td>40.87</td>
<td>8.88</td>
<td>87.9</td>
</tr>
<tr>
<td>Bottom product 115°C</td>
<td>28.81</td>
<td>3.57</td>
<td>1.95</td>
<td>44.77</td>
<td>9.29</td>
<td>89.6</td>
</tr>
<tr>
<td>Bottom product 120°C</td>
<td>25.8</td>
<td>3.47</td>
<td>1.98</td>
<td>45.57</td>
<td>10.07</td>
<td>89.77</td>
</tr>
<tr>
<td>Bottom product 125°C</td>
<td>23.4</td>
<td>2.3</td>
<td>2.45</td>
<td>51.28</td>
<td>11.04</td>
<td>90.76</td>
</tr>
</tbody>
</table>

**Fish oil methyl esters 42.01%**

**DSL5 FIRST DISTILLATION**

Bottom product 68.2%

**DSL5 SECOND DISTILLATION**

Bottom product 85.42%

**KDL5 DISTILLATION**

Heavy product 90%
9. Conclusions

- PUFA are essential fatty acids with high importance for human health, but their separation from saturated and unsaturated fatty acids mixtures is difficult.

- Advanced separation techniques based on high vacuum as **High vacuum fractionation** and **Molecular (Short path) distillation** can be successfully used to obtain concentrated omega-3 fatty acids methyl/ethyl esters fractions.

- Based on proposed conceptual separation scheme (**two consecutive high vacuum fractionations followed by one molecular distillation**), an experimental working plan was performed to concentrate omega-3 fish oil methyl esters from ~42% to ~90% and fish oil ethyl esters from ~35% to ~88%.
Future work

- New POC Project 2016-2020: **Advanced separation systems for bioresources valorization (ASPiRE) - P_37_449**
- Special distillation techniques energetically effective (dividing wall, middle vessel, cyclic distillation)
- Supercritical CO\(_2\) extraction (L-L and L-S)
- Non-conventional techniques (*pressurized liquid extraction, microwave assisted extraction, advanced vacuum distillation, molecular distillation*)
- New solvents for extraction (DIMCARB)
THANK YOU!

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