A Novel Continuous Oil Seed Extraction Method for Jet Fuel Production

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### U.S. Dependence on Foreign Oil

**Have Oil**

- Saudi Arabia: 26%
- Iraq: 11%
- Kuwait: 10%
- Iran: 9%
- UAE: 8%
- Venezuela: 6%
- Russia: 5%
- Libya: 3%
- Mexico: 3%
- China: 3%
- Nigeria: 2%
- U.S.: 2%

**Use Oil**

- U.S.: 26%
- Japan: 7%
- China: 6%
- Germany: 4%
- Canada: 4%
- Russia: 3%
- Brazil: 3%
- S. Korea: 3%
- France: 3%
- India: 3%
- Mexico: 3%
- Italy: 2%

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*The U.S. uses more than the next 5 highest consuming nations combined.*

U.S. production and net import shares of petroleum and other liquid demands in 2012

Data source: [http://www.eia.gov/energy_in_brief/article/foreign_oil_dependence.cfm](http://www.eia.gov/energy_in_brief/article/foreign_oil_dependence.cfm)
The Bioeconomy Roadmap

Task 1: Agricultural Processing

Biomass

Task 2: Biofuel production

- Bagasse
- Distiller grains
- Lignin
- Protein rich meals
- Coproducts

Crude oil

Task 3: Coproducts development

- Chemicals & Polymers
- Composites
- Carbon Materials

Task 4: Work with communities to build rural economy
Challenges

Development of commercially viable biomass utilization technologies.

Sustainable and economic production of advanced biofuels.

Compatibility with today’s transportation infrastructure.

Oil quantity and fatty acid contents in oil are two major factors responsible for suitability of oil for aviation fuel.

Advanced biofuels from algae and non-food oil seeds are promising.
3rd Generation Advanced Biofuels

“Drop-In” bioderived equivalent for gasoline, diesel and JP-8

Infrastructure compatible, energy dense

Safflower, carinata, camelina, canola, several others

Photoautotrophs: conversion of CO2 - Engineered organisms

Modes of Production

Refining of vegetable oils
Opportunity

- US Navy initiative to use 50% blends of green diesel and jet fuel by 2020
- Annual market for 336 million gallons of fuel
- Larger national market of 140 billion gallons of fuel
Department of Defense Interest in Biofuels

- Navy sailing great green fleet in 2014
  - Green FA-18 Hornet
  - Green AV-8B Harrier
    - [http://youtu.be/K-P9_U0SzE8](http://youtu.be/K-P9_U0SzE8)
Biofuels from Non-Food Oilseeds:
Understanding the linkages between Oilseed species, oil extraction processing, oil quality, fat acid profile, production costs and extraction efficiency.

Catalytic Cracking
Extraction
Processing
Animal Feed
Oilseeds
Crude Oil
Jet Fuel
Solid fraction
Enzymatic/microbial conversion

Extrusion Pretreatment
Microbial Conversion
Feeding Trial
Objective

To evaluate and compare three oil extraction technologies, solvent extraction, cold press, and novel continuous extraction, for efficiently extracting oils from different non-food oilseeds for further conversion into aviation fuels.
Oilseed Varieties

Camelina seeds

Carinata seeds

Flax seeds

Sunflower seeds

Canola seeds

Safflower seeds
Cold press for oil extraction
# Oil Characterization

## Oil extraction from canola seeds at different speeds

<table>
<thead>
<tr>
<th>Extraction speed</th>
<th>pH value</th>
<th>Seed water content, %</th>
<th>Oil water content, %</th>
<th>Density (g/ml)</th>
<th>Viscosity (cP @20°C)</th>
<th>Extraction efficiency, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>25HZ</td>
<td>6</td>
<td>4.89</td>
<td>0.06</td>
<td>0.833</td>
<td>85.57</td>
<td>84.25</td>
</tr>
<tr>
<td>20HZ</td>
<td>6</td>
<td>4.89</td>
<td>0.06</td>
<td>0.82</td>
<td>84.97</td>
<td>84.73</td>
</tr>
<tr>
<td>15HZ</td>
<td>6</td>
<td>4.89</td>
<td>0.05</td>
<td>0.893</td>
<td>87.23</td>
<td>86.25</td>
</tr>
</tbody>
</table>

## Oil extraction from flax seeds at different speeds

<table>
<thead>
<tr>
<th>Extraction speed</th>
<th>pH value</th>
<th>Seed water content, %</th>
<th>Oil water content, %</th>
<th>Density (g/ml)</th>
<th>Viscosity (cP @ 20°C)</th>
<th>Extraction efficiency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>27HZ</td>
<td>5</td>
<td>6.27</td>
<td>0.06</td>
<td>0.894</td>
<td>51.7</td>
<td>70.54</td>
</tr>
<tr>
<td>22HZ</td>
<td>5</td>
<td>6.27</td>
<td>0.09</td>
<td>0.900</td>
<td>50.2</td>
<td>74.9</td>
</tr>
<tr>
<td>17HZ</td>
<td>5</td>
<td>6.27</td>
<td>0.07</td>
<td>0.907</td>
<td>50.3</td>
<td>77.56</td>
</tr>
</tbody>
</table>

- Different oilseeds required different extraction conditions.
- Extraction speed and time affect oil yield and properties.
- Higher speed and shorter time led to lower oil yield.
### Characterization of Cold Pressed Camelina Oil

<table>
<thead>
<tr>
<th>Screw speed, Hz</th>
<th>Die Dia, inch</th>
<th>Extraction Efficiency, %</th>
<th>Heating value, MJ/kg</th>
<th>Viscosity, cP</th>
<th>Density, kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>0.22</td>
<td>78.6</td>
<td>39.72 ± 0.1</td>
<td>56.1 ± 0.1</td>
<td>913.7 ± 11.9</td>
</tr>
<tr>
<td>20</td>
<td>0.22</td>
<td>83.3</td>
<td>39.73 ± 0.1</td>
<td>57.0 ± 0.1</td>
<td>908.3 ± 5.9</td>
</tr>
<tr>
<td>25</td>
<td>0.22</td>
<td>81.0</td>
<td>39.74 ± 0.2</td>
<td>56.4 ± 0.3</td>
<td>910.0 ± 14.2</td>
</tr>
<tr>
<td>15</td>
<td>0.25</td>
<td>76.2</td>
<td>39.78 ± 0.2</td>
<td>56.1 ± 0.2</td>
<td>914.0 ± 4.6</td>
</tr>
<tr>
<td>20</td>
<td>0.25</td>
<td>76.2</td>
<td>39.77 ± 0.2</td>
<td>56.2 ± 0.1</td>
<td>912.3 ± 11.6</td>
</tr>
<tr>
<td>25</td>
<td>0.25</td>
<td>73.8</td>
<td>39.96 ± 0.2</td>
<td>55.9 ± 1.1</td>
<td>908.7 ± 3.8</td>
</tr>
<tr>
<td>15</td>
<td>0.28</td>
<td>73.8</td>
<td>39.95 ± 0.1</td>
<td>55.2 ± 1.2</td>
<td>911.7 ± 6.5</td>
</tr>
<tr>
<td>20</td>
<td>0.28</td>
<td>73.8</td>
<td>39.90 ± 0.2</td>
<td>56.0 ± 0.6</td>
<td>907.3 ± 6.7</td>
</tr>
<tr>
<td>25</td>
<td>0.28</td>
<td>71.4</td>
<td>39.95 ± 0.2</td>
<td>56.2 ± 0.3</td>
<td>903.7 ± 12.8</td>
</tr>
</tbody>
</table>

### Characterization of Cold Pressed Carinata Oil

<table>
<thead>
<tr>
<th>Screw speed, Hz</th>
<th>Die Dia, inch</th>
<th>Extraction Efficiency, %</th>
<th>Heating value, MJ/kg</th>
<th>Viscosity, cP</th>
<th>Density, kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>0.22</td>
<td>79.1</td>
<td>40.40 ± 0.2</td>
<td>70.0 ± 0.8</td>
<td>872.7 ± 8.5</td>
</tr>
<tr>
<td>20</td>
<td>0.22</td>
<td>81.4</td>
<td>40.32 ± 0.2</td>
<td>70.4 ± 1.2</td>
<td>870.7 ± 2.8</td>
</tr>
<tr>
<td>25</td>
<td>0.22</td>
<td>79.1</td>
<td>40.57 ± 0.3</td>
<td>70.5 ± 1.3</td>
<td>874.6 ± 10.4</td>
</tr>
<tr>
<td>15</td>
<td>0.25</td>
<td>79.1</td>
<td>40.19 ± 0.7</td>
<td>70.3 ± 0.8</td>
<td>878.3 ± 6.2</td>
</tr>
<tr>
<td>20</td>
<td>0.25</td>
<td>76.7</td>
<td>40.51 ± 0.1</td>
<td>71.5 ± 1.1</td>
<td>874.7 ± 4.9</td>
</tr>
<tr>
<td>25</td>
<td>0.25</td>
<td>74.4</td>
<td>40.47 ± 0.1</td>
<td>71.4 ± 0.9</td>
<td>871.4 ± 10.5</td>
</tr>
<tr>
<td>15</td>
<td>0.28</td>
<td>86.0</td>
<td>40.44 ± 0.1</td>
<td>69.9 ± 0.9</td>
<td>875.1 ± 6.3</td>
</tr>
<tr>
<td>20</td>
<td>0.28</td>
<td>65.1</td>
<td>40.36 ± 0.3</td>
<td>70.5 ± 1.1</td>
<td>876.9 ± 11.2</td>
</tr>
<tr>
<td>25</td>
<td>0.28</td>
<td>65.1</td>
<td>40.48 ± 0.1</td>
<td>69.5 ± 0.7</td>
<td>879.5 ± 2.6</td>
</tr>
</tbody>
</table>
Accelerated Solvent Extraction (ASE)

- Effect of extraction temperature, extraction time and solvents on oil recovery was evaluated for canola seeds using ASE method
<table>
<thead>
<tr>
<th>No.</th>
<th>Solvent</th>
<th>Boiling point (°C)</th>
<th>Density (g/ml)</th>
<th>Viscosity (cp) at 25 °C</th>
<th>Flash point (°C)</th>
<th>Auto ignition temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Hexane</td>
<td>69</td>
<td>0.6548</td>
<td>0.294</td>
<td>-26</td>
<td>234</td>
</tr>
<tr>
<td>2</td>
<td>Ethanol</td>
<td>78</td>
<td>0.789</td>
<td>1.074</td>
<td>14</td>
<td>363</td>
</tr>
<tr>
<td>3</td>
<td>Ethyl acetate</td>
<td>77</td>
<td>0.897</td>
<td>0.426</td>
<td>-4</td>
<td>460</td>
</tr>
<tr>
<td>4</td>
<td>1-butanol</td>
<td>118</td>
<td>0.81</td>
<td>3.0</td>
<td>35</td>
<td>343</td>
</tr>
</tbody>
</table>

### Oil extraction from canola seeds using different solvents

<table>
<thead>
<tr>
<th>S No</th>
<th>Temp [°C]</th>
<th>Time [min]</th>
<th>% Extraction Efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hexane</td>
</tr>
<tr>
<td>1</td>
<td>80</td>
<td>40</td>
<td>69.3</td>
</tr>
<tr>
<td>2</td>
<td>120</td>
<td>40</td>
<td>81.7</td>
</tr>
<tr>
<td>3</td>
<td>80</td>
<td>90</td>
<td>74.6</td>
</tr>
<tr>
<td>4</td>
<td>120</td>
<td>90</td>
<td>87.1</td>
</tr>
<tr>
<td>5</td>
<td>80</td>
<td>40</td>
<td>72.9</td>
</tr>
<tr>
<td>6</td>
<td>120</td>
<td>40</td>
<td>87.1</td>
</tr>
<tr>
<td>7</td>
<td>80</td>
<td>90</td>
<td>65.4</td>
</tr>
<tr>
<td>8</td>
<td>120</td>
<td>90</td>
<td>100.2</td>
</tr>
<tr>
<td>9</td>
<td>100</td>
<td>65</td>
<td>84.9</td>
</tr>
</tbody>
</table>

- Increase in temperature and extraction time improved the oil recovery for all the solvents
- After hexane, acetate showed high potential for oil extraction followed by pinene, butanol and ethanol.
Green Solvent - Terpene

- Environmentally safer than Hexane
- Natural solvents existing both in citrus fruits and in many other plants
- Include hydrocarbons with C5H8 isoprene units
- d-Limonene: derived from citrus peels
- α-Pinene: from gum turpentine in pine gum
Solvent-Assisted Extrusion (SAE)

- Oil extraction methods
  - Using single screw extruder to determine the upper limit of available oil from the oilseeds
  - Using single screw extruder and green solvent simultaneously to extract the oil from the oil seeds
  - Quality analysis of the extracted oils
Experimental Designs

- Evaluating the effect of extrusion parameters on oil extraction yield & quality

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>40-60-60</th>
<th>40-60-80</th>
<th>40-60-100</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS (rpm)</td>
<td>40</td>
<td>60</td>
<td>80</td>
</tr>
</tbody>
</table>

- Evaluating the effect of green-solvent assisted extrusion parameters on oil extraction yield & quality.

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>40-80-80</th>
<th>40-80-120</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS (rpm)</td>
<td>80</td>
<td>150</td>
</tr>
<tr>
<td>R (W/W%)</td>
<td>10</td>
<td>15</td>
</tr>
<tr>
<td>solvents</td>
<td>α-Pinene</td>
<td>L-Limonene</td>
</tr>
</tbody>
</table>
Crambe oil, 80 °C, 80 rpm

Pennycress oil, 80 °C, 80 rpm

Single-screw (Brabender, Plasti-Corder (PL 2000))
Increasing T and SS reduced and increased the residual oil content of the extruded meal, respectively. Perhaps, the diminution of the SS increased the mean residence time of the extruding seeds and resulted in more oil release from the broken cell walls.

Increasing R, reduced the oil residual content in extrudates implying the positive effect of R on SAE processing.

Type of solvent did not show significant effect on oil residual content of the extrudates (α=0.05)
Pennycress seeds

Maximal oil recovery

![Bar chart showing extraction efficiency](image)

- **80 C, 80rpm**
  - Pinene-10%"
  - Pinene-15%"
  - No solvent

- **120 C, 80rpm**
  - Pinene-10%"
  - Pinene-15%"
  - No solvent

- **80 C, 150rpm**
  - Pinene-10%"
  - Pinene-15%"
  - No solvent

- **120 C, 150rpm**
  - Pinene-10%"
  - Pinene-15%"
  - No solvent

**Extrusion Conditions**

**Extraction Efficiency, %**
Crambe Seeds

Maximal oil recovery

Extraction Efficiency, %

80 C, 80rpm 120 C, 80rpm 80 C, 150rpm 120 C, 150rpm

Extrusion Conditions

- Pinene-10%"
- Pinene-15%"
- No solvent
Properties of Pennycress and Crambe Oils extracted using SAE (120 °C, 80 RPM)

<table>
<thead>
<tr>
<th></th>
<th>Pennycress</th>
<th>Crambe</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Pinene</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10%</td>
<td>15%</td>
</tr>
<tr>
<td>Density, (Kg/m3)</td>
<td>898 ± 20</td>
<td>901 ±20</td>
</tr>
<tr>
<td>Acid value (mgKOH/g)</td>
<td>0.61 ± 0.02</td>
<td>0.62 ±0.01</td>
</tr>
<tr>
<td>Heating Value (MJ/kg)</td>
<td>39.45 ± 0.13</td>
<td>39.39 ±0.14</td>
</tr>
</tbody>
</table>

- SAE operating parameters had no significant effect on solvent-extruded oil qualities, in terms of density, acid value, and heating value.
CH Pilot System

High pressure, high temperature

Five min reaction time

Carinata

Camelina

Plant Stand
Acknowledgements

- Agricultural Experiment Station, ABS College, SDSU
- US Department of Transportation
- NASA-EPSCoR, Oil Seeds Initiative
Questions???

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