Method Performance Study for Total Solids and Fat in Coconut Milk and Products

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Content

• Background and objective of the study
• Framework of the study
• Literature review
• Single laboratory study
• Interlaboratory study
• Results and conclusion of the study
Background of the Study

- **CODEX STAN 240-2003: Codex standard for aqueous coconut products**
  - Coconut milk and coconut cream

<table>
<thead>
<tr>
<th>Product</th>
<th>Total Solids (% m/m)</th>
<th>Non-fat Solids (%m/m)</th>
<th>Fat (%m/m)</th>
<th>Moisture (%m/m)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) Light Coconut Milk</td>
<td>6.6-12.6</td>
<td>1.6</td>
<td>5.0</td>
<td>93.4</td>
<td>5.9</td>
</tr>
<tr>
<td>(b) Coconut Milk</td>
<td>12.7-25.3</td>
<td>2.7</td>
<td>10.0</td>
<td>87.3</td>
<td>5.9</td>
</tr>
<tr>
<td>(c) Coconut Cream</td>
<td>25.4-37.3</td>
<td>5.4</td>
<td>20.0</td>
<td>74.6</td>
<td>5.9</td>
</tr>
<tr>
<td>(d) Coconut Cream Concentrate</td>
<td>37.4 min</td>
<td>8.4</td>
<td>29.0</td>
<td>62.6</td>
<td>5.9</td>
</tr>
</tbody>
</table>

- No analytical method was endorsed

Objectives of the Study

- To find the appropriate methods for determination of total solids and fat in coconut milk and products

- To propose the appropriate methods to CODEX committee on method of analysis and sampling (CCMAS)
Literature review

- Methods for total solids analysis
  - Oven drying method: variety of food products
    - Vacuum oven: fruit and products, confectionery, beverage and honey
    - Hot air oven: milk, cream, flour, cereal and products
  - Distillation method: spice
  - Titrimetric method (the Carl Fisher method): low moisture foods: dried vegetables
  - Instrumental method: rapid, costly, in-plant quality control
    - NMR, NIR

Methods Selection for single laboratory study

- Total solids analysis
  - Consideration for selection the methods
    - simple, basic instrument, low cost
  - Selected methods: Oven drying method
    - Hot air oven 102 ± 2°C
    - Hot air oven 130 ± 3°C
    - Vacuum oven 70°C, 70 mmHg
Methods for fat analysis

- Solvent extraction method: variety of food products
  - Direct solvent extraction: butter, spreadable fat,
  - Acid hydrolysis-solvent extraction (Schmid-Bondzynski-Ratzlaff principle): cereal, seafoods
  - Alkaline hydrolysis-solvent extraction (Roese-Gottlieb principle): milk, cream, ice-cream, fermented milk

- Non-solvent extraction method: raw milk
  - Babcock method, Gerber method

- Instrumental method: Rapid, costly, in-plant quality control
  - IR, NMR

Methods Selection for single laboratory study

- Fat analysis
  - Consideration for selection the method
    - common method
    - approved method for many kinds of food
    - basic instrument, low cost
  - Selected method: 3 techniques of solvent extraction method
    - Direct solvent extraction
    - Acid hydrolysis-solvent extraction (Schmid-Bondzynski-Ratzlaff principle)
    - Alkaline hydrolysis-solvent extraction (Roese-Gottlieb principle)
Single laboratory study

• Test materials:
  • 16 samples of aqueous coconut milk, coconut cream and coconut milk powder

• Equipment:
  • Calibrated hot air oven and vacuum oven

• Determination:
  • 10 replicates for each sample

• Results:

Table 1. Summary of total solids level in 16 test materials

<table>
<thead>
<tr>
<th>No.</th>
<th>Total solids (g/100 g), mean ±SD, N=10</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Hot air oven 102 ± 2 °C</td>
</tr>
<tr>
<td>1</td>
<td>7.68 ± 0.01</td>
</tr>
<tr>
<td>2</td>
<td>8.66 ± 0.01</td>
</tr>
<tr>
<td>3</td>
<td>9.05 ± 0.02</td>
</tr>
<tr>
<td>4</td>
<td>7.82 ± 0.02</td>
</tr>
<tr>
<td>5</td>
<td>12.14 ± 0.02</td>
</tr>
<tr>
<td>6</td>
<td>18.86 ± 0.02</td>
</tr>
<tr>
<td>7</td>
<td>20.47 ± 0.03</td>
</tr>
<tr>
<td>8</td>
<td>21.87 ± 0.02</td>
</tr>
<tr>
<td>9</td>
<td>22.17 ± 0.05</td>
</tr>
<tr>
<td>10</td>
<td>24.69 ± 0.01</td>
</tr>
<tr>
<td>11</td>
<td>25.35 ± 0.02</td>
</tr>
<tr>
<td>12</td>
<td>26.32 ± 0.02</td>
</tr>
<tr>
<td>13</td>
<td>26.43 ± 0.02</td>
</tr>
<tr>
<td>14</td>
<td>29.24 ± 0.01</td>
</tr>
<tr>
<td>15</td>
<td>27.96 ± 0.03</td>
</tr>
<tr>
<td>16</td>
<td>98.92 ± 0.02</td>
</tr>
</tbody>
</table>

No significant difference (p>0.05)
Candidate method

- Total solids analysis: hot air oven 102±2°C
  - Why:
    - Hot air oven: basic instrument, cheaper, easier to operate and calibrate
    - 102±2°C: general principle for the determination of moisture in foods (boiling point of water= 100°C)

- Fat analysis: Alkaline hydrolysis-solvent extraction (Roese-Gottlieb principle)
  - Why:
    - easier and fewer steps than acid hydrolysis
Inter-laboratory study

- Participating laboratories (17 laboratories: 7 government, 10 private)
- Test material
  - Types of test materials:
    - Phase I: 3 samples (light coconut milk, coconut cream, coconut milk powder)
    - Phase II: 8 samples (four sets of blind duplicates; light coconut milk, coconut milk, coconut cream and coconut milk powder)
  - Preparation:
    - approx. 50 g. for each sample in sealed plastic bottles and labeled

Test material (cont.)
- Homogeneity testing
- Distribution of test materials
  - Including: Instruction for participant, determination method, sample receipt form, reporting form
- Instruction for the participants
  - Store samples at 2-8°C
  - Analyze in 1-2 days after received the samples
  - Report the results within 3 weeks
  - Follow the method exactly
Inter-laboratory study

- Statistic analysis: Guidelines for collaborative study procedures to validate characteristics of a method of analysis (Horwitz, 2002)
  - Outliers testing:
    - Cochran’s test
    - Grubbs’ test
  - Precision:
    - Repeatability (r)
    - Reproducibility (R)

Table 3: Conclusion of method performance for total solids determination

<table>
<thead>
<tr>
<th>Test materials</th>
<th>Light coconut milk</th>
<th>Coconut milk</th>
<th>Coconut cream</th>
<th>Coconut milk powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>No. of laboratory (No. of outlier)</td>
<td>17(1)</td>
<td>17(1)</td>
<td>17(3)</td>
<td>17(0)</td>
</tr>
<tr>
<td>Mean (g/100g)</td>
<td>8.84</td>
<td>10.57</td>
<td>20.90</td>
<td>27.01</td>
</tr>
<tr>
<td>Repeatability standard deviation, Sr (g/100g)</td>
<td>0.02</td>
<td>0.03</td>
<td>0.12</td>
<td>0.49</td>
</tr>
<tr>
<td>Repeatability relative standard deviation, %RSDr</td>
<td>0.21</td>
<td>0.30</td>
<td>0.59</td>
<td>1.83</td>
</tr>
<tr>
<td>Repeatability limit, r=2.8 Sr</td>
<td>0.05</td>
<td>0.09</td>
<td>0.35</td>
<td>1.38</td>
</tr>
<tr>
<td>Reproducibility standard deviation, Sr (g/100g)</td>
<td>0.04</td>
<td>0.11</td>
<td>0.22</td>
<td>0.60</td>
</tr>
<tr>
<td>Reproducibility relative standard deviation, %RSDR</td>
<td>0.59</td>
<td>1.05</td>
<td>1.05</td>
<td>2.24</td>
</tr>
<tr>
<td>Reproducibility limit, R=2.8 Sr</td>
<td>0.12</td>
<td>0.31</td>
<td>0.61</td>
<td>1.69</td>
</tr>
</tbody>
</table>
Inter-laboratory study

Table 4: Conclusion of method performance for fat determination

<table>
<thead>
<tr>
<th>Test materials</th>
<th>Light coconut milk</th>
<th>Coconut milk</th>
<th>Coconut cream</th>
<th>Coconut milk powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>No. of laboratory (No. of outlier)</td>
<td>17(2)</td>
<td>17(4)</td>
<td>17(1)</td>
<td>17(3)</td>
</tr>
<tr>
<td>Mean (g/100g)</td>
<td>5.88</td>
<td>8.28</td>
<td>16.85</td>
<td>21.83</td>
</tr>
<tr>
<td>Repeatability standard deviation, Sr (g/100g)</td>
<td>0.03</td>
<td>0.16</td>
<td>0.76</td>
<td>0.23</td>
</tr>
<tr>
<td>Repeatability relative standard deviation, %RSDr</td>
<td>0.59</td>
<td>1.88</td>
<td>4.49</td>
<td>1.04</td>
</tr>
<tr>
<td>Repeatability limit, r=2.8 Sr</td>
<td>0.10</td>
<td>0.44</td>
<td>2.12</td>
<td>0.63</td>
</tr>
<tr>
<td>Reproducibility standard deviation, Sr(100g)</td>
<td>0.16</td>
<td>0.15</td>
<td>0.89</td>
<td>0.74</td>
</tr>
<tr>
<td>Reproducibility relative standard deviation, %RSDR</td>
<td>2.7</td>
<td>1.77</td>
<td>5.30</td>
<td>3.4</td>
</tr>
<tr>
<td>Reproducibility limit, R=2.8 Sr(100g)</td>
<td>0.44</td>
<td>0.41</td>
<td>2.50</td>
<td>2.08</td>
</tr>
</tbody>
</table>

Conclusion

- The Delegation of Thailand proposed these methods to CODEX committee on methods of analysis and sampling (CCMAS) on 9-13 Mar, 2009, in Hungary

- the Committee agreed to endorse both methods as type I (defining method)
Participating laboratories

- IQA Laboratory Co., Ltd.
- South East Asian Laboratory Ltd., (SEAL)
- Institute of Nutrition, Mahidol University
- SGS (Thailand) Limited.
- Asia Medical and Agricultural Laboratory and Research Center Co., Ltd.
- Nutrition and Food Analysis.
- Thailand Institute of Scientific and Technological Research.
- Thai Agri Foods Public Company Limited.
- Center of Export Inspection and Certification for Agricultural Products
- Laboratory Center for Food and Agricultural Products Company Limited.
- National Food Institute (Thailand).
- Analytical Laboratory Service Co., Ltd.
- Department of Science Service, Ministry of Science and Technology.
- Food Quality Assurance Center.
- Intertek Testing Services (Thailand) Ltd.
- Chemlab Services (Thailand) Limited.
- Bureau of Quality and Safety of Food. Department of Medical Sciences.

Acknowledgement

- The manufacturers who prepared test materials for this study

  - Thai Agri Foods Public Company Limited.
  - Ampol Food Processing Ltd
  - Asiatic Agro Industry Co., Ltd.
  - Theppadungporn Coconut Co., Ltd