
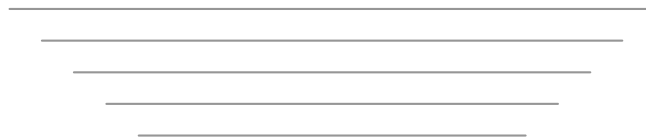


Method Performance Study for Total Solids and Fat in Coconut Milk and Products

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Content

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- Results and conclusion of the study

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Background of the Study

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

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

- **No analytical method was endorsed**

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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)

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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

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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 $102 \pm 2^\circ$ C
 - Hot air oven $130 \pm 3^\circ$ C
 - Vacuum oven 70° C, 70 mmHg

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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

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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)

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8th International Food Data Conference

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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:

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Single Laboratory analysis
Table1. Summary of total solids level in 16 test materials

No.	Total solids (g /100 g),, mean \pm SD, N=10		
	hot air oven 102 \pm 2 $^{\circ}$ C	Hot air oven 130 \pm 3 $^{\circ}$ C	Vacuum oven 70 $^{\circ}$ C,70 mmHg
1	7.68 \pm 0.01	7.63 \pm 0.01	8.38 \pm 0.02
2	8.66 \pm 0.01	8.66 \pm 0.02	8.67 \pm 0.14
3	9.05 \pm 0.02	9.05 \pm 0.02	9.10 \pm 0.02
4	7.82 \pm 0.02	7.84 \pm 0.01	7.84 \pm 0.01
5	12.14 \pm 0.02	12.06 \pm 0.02	12.19 \pm 0.02
6	18.86 \pm 0.02	18.59 \pm .02	18.77 \pm .02
7	20.47 \pm 0.03	20.35 \pm 0.02	20.72 \pm 0.21
8	21.87 \pm 0.02	21.66 \pm 0.02	21.69 \pm 0.02
9	22.17 \pm 0.05	22.05 \pm 0.01	22.34 \pm 0.03
10	24.69 \pm 0.01	24.64 \pm 0.01	24.72 \pm 0.02
11	25.35 \pm 0.02	25.24 \pm 0.02	25.42 \pm 0.02
12	26.32 \pm 0.02	26.32 \pm 0.02	26.46 \pm 0.01
13	26.43 \pm 0.02	26.85 \pm 0.03	26.66 \pm 0.03
14	29.24 \pm 0.01	28.13 \pm 0.02	27.52 \pm 0.01
15	27.96 \pm 0.03	27.80 \pm 0.02	28.05 \pm 0.02
16	98.92 \pm 0.02	98.93 \pm 0.03	98.95 \pm 0.03

No significant difference ($p > 0.05$)

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Single Laboratory analysis
Table 2. Summary of fat level in 16 test material

No	Fat (g /100 g),, mean \pm SD, N=10		
	Direct extraction	Acid hydrolysis	Alkaline hydrolysis
1	4.65 \pm 0.02 ⁽¹⁾	4.97 \pm 0.01	5.07 \pm 0.02
2	5.01 \pm 0.02	5.10 \pm 0.02	5.06 \pm 0.01
3	6.03 \pm 0.02	6.37 \pm 0.04	6.38 \pm 0.12
4	6.22 \pm 0.03	6.37 \pm 0.04	6.40 \pm 0.10
5	7.43 \pm 0.02	8.92 \pm 0.02	9.19 \pm 0.01
6	14.55 \pm 0.01	14.76 \pm 0.01	15.05 \pm 0.11
7	15.72 \pm 0.01	16.97 \pm 0.02	17.50 \pm 0.02
8	17.52 \pm 0.01	17.67 \pm 0.02	17.60 \pm 0.01
9	19.20 \pm 0.02	19.34 \pm 0.02	20.00 \pm 0.10
10	19.72 \pm 0.02	21.35 \pm 0.01	21.80 \pm 0.02
11	20.36 \pm 0.02	21.02 \pm 0.02	21.00 \pm 0.27
12	20.45 \pm 0.02	20.64 \pm 0.01	20.53 \pm 0.03
13	21.47 \pm 0.01	22.88 \pm 0.01	22.27 \pm 0.03
14	21.49 \pm 0.02	21.76 \pm 0.01	21.76 \pm 0.16
15	22.17 \pm 0.02	22.52 \pm 0.01	21.74 \pm 0.02
16	41.44 \pm 0.03	41.52 \pm 0.02	41.64 \pm 0.03

No significant difference ($p > 0.05$)

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Candidate method

- Total solids analysis: hot air oven 102 \pm 2°C
 - Why:
 - Hot air oven: basic instrument, cheaper, easier to operate and calibrate
 - 102 \pm 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

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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

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Inter-laboratory study

- 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

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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)

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Inter-laboratory study

Table 3: Conclusion of method performance for total solids determination

Test materials	Light coconut milk		Coconut milk	Coconut cream		Coconut milk powder	
No. of laboratory (No. of outlier)	17(1)	17(1)	17(3)	17(0)	17(3)	17(0)	17(0)
Mean (g/100g)	8.84	10.57	20.90	27.01	28.97	98.82	99.25
Repeatability standard deviation, S_r (g/100g)	0.02	0.03	0.12	0.49	0.15	0.04	0.10
Repeatability relative standard deviation, %RSD _r	0.21	0.30	0.59	1.83	0.53	0.04	0.10
Repeatability limit, $r=2.8 S_r$	0.05	0.09	0.35	1.38	0.43	0.12	0.28
Reproducibility standard deviation, S_R (g/100g)	0.04	0.11	0.22	0.60	0.28	0.21	0.31
Reproducibility relative standard deviation, %RSD _R	0.50	1.05	1.05	2.24	0.95	0.21	0.32
Reproducibility limit, $R=2.8 S_R$	0.12	0.31	0.61	1.69	0.77	0.59	0.88

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Inter-laboratory study

Table 4: Conclusion of method performance for fat determination

Test materials	Light coconut milk		Coconut milk	Coconut cream		Coconut milk powder	
	17(2)	17(4)	17(1)	17(1)	17(3)	17(4)	17(3)
Mean (g/100g)	5.88	8.28	16.85	21.83	23.02	42.90	42.44
Repeatability standard deviation, S_r (g/100g)	0.03	0.16	0.76	0.23	0.28	0.11	0.17
Repeatability relative standard deviation, %RSD _r	0.59	1.88	4.49	1.04	1.22	0.26	0.41
Repeatability limit, $r=2.8 S_r$	0.10	0.44	2.12	0.63	0.79	0.31	0.49
Reproducibility standard deviation, S_R (g/100g)	0.16	0.15	0.89	0.74	0.46	0.34	0.40
Reproducibility relative standard deviation, %RSD _R	2.7	1.77	5.30	3.4	2.02	0.80	0.94
Reproducibility limit, $R=2.8 S_R$	0.44	0.41	2.50	2.08	1.30	0.97	1.11

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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)

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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.

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