Evaluation of methods for the determination of the residual oil content in rapeseed and other oilseeds meals and cakes

PROLEA
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afnor NORMALISATION

***The accurate assessment** of the residual oil content in the industrial meals and cakes is critical for the optimization of the oil extraction process because of its higher economical value. Accuracy of oil content is also important to take in account the actual energy value of the meals when included in feedstuffs.*

***Comparison of data** collected in France through a long-term survey on the quality of rapeseed and sunflower meal indicated that an inadequate analysis method (dedicated to feedstuff and using solvent extraction without hydrolysis) may underestimate the oil content. Evaluation of these methods of analysis was then necessary.*

***A collaborative study** was conducted with most of the laboratories involved in the French committees of standardization AFNOR V18A (feedstuffs) and T60D (oilseeds and meals) to evaluate the performance of 3 types of methods that are currently used or dedicated for oilseed meals, cakes and feedstuffs.*

Material and methods

Preparation of samples for the collaborative study

Meals and cakes were collected in France to be representative of the current production. The samples (50 g) were prepared by CETIOM laboratory (45-Ardon) by using a riffle-type dividing sampler.

Samples		Process
1-RSM	Rapeseed meals	Regular de-oiled meals from 5 different plants using **Pressing**, **solvent extraction** and **desolventization**
2-RSM		
3-RSM		
4-SFM	Sunflower meal	
5-SBM	Soymeal	
6-RSC	Rapeseed cake	Cold pressing (in farm)
7-ESB	Full fat extruded soybean	Cooking-Extrusion
8-LSC	Linseed cake	Cold pressing (industrial)

Determination of moisture

According to NF V03-921 (103 °C during 4 h).

Determination of the oil content

***Methods A :** dedicated for « feedstuffs » and based on a **simple solvent extraction** (petroleum ether or hexane during 4 h) :*
- NF V18-117 (part A) or Directive 98/64/CE (part A)

***Methods B :** dedicated for « feedstuffs » and based on solvent extraction **with hydrolysis**:*
- NF V18-117 (part B) or Directive 98/64/CE (part B)

Method B = hydrolysis + method A

Methods C :** dedicated for « oilseeds and meals » based on solvent extraction **with grinding:
- ISO 734-2 : ball mill with solvent 10 min + solvent extraction 1h (rapid method for meals)
- ISO 659 : solvent extraction 4 h + (ball mill + solvent extraction 2 h) x 2 + solvent extraction 2 h (reference method for seeds)
- V03-908 : ball mill with solvent 5 min + solvent extraction 3h (rapid method for seeds)

Method C = grinding + method A

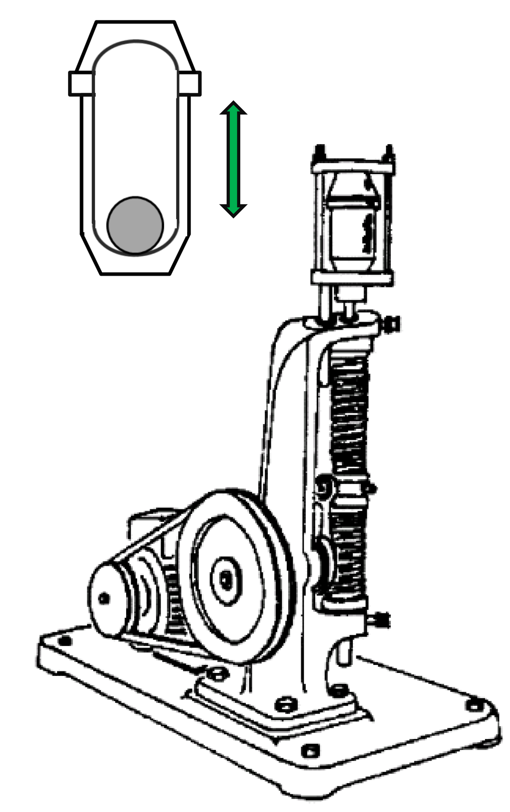


Figure : Dangoumau type mill with ball chamber used in Methods C to improve the oil extraction.

Participating laboratories

12 laboratories from the following sectors participated to the collaborative study : animal feeding (6), crushing industry (3), agricultural research centers (2), government (1).

Program

Each of the 8 samples were analysed for the moisture (1 determination by 1 method) and the residual oil contents (2 replicates by each of the methods A, B and C). Analyses were carried out from January to July, 2009,

Statistical treatment

Precision data were determined according to ISO 5725-2. Statistical stragglers and outliers were detected by the Cochran and Grubbs tests. Only outliers were discarded. Critical probabilities for no difference between methods for each observation (sample x lab) were determined from the reproducibility variance and the normal distribution function .

Results and discussion

***Precision data**:* results showed that the repeatability and reproducibility data were in agreement with those determined in previous ring-tests and mentioned in standards: NF V18-117 Part 1 for method A, NF V18-117 Part 2 for method B and ISO 734-2 for method C.
The precision achieved in the study allowed to **make significant most of the differences** observed between the three methods, particularly for samples heated during the crushing process. Increasing contents of residual oil in deoiled oilseed meals were given by methods A, C and B, respectively

Hydrolysis effect : the difference between results from methods B and A corresponding to the linked fat released by hydrolysis, **ranged from 0.7 (soybean meal) to 1.4 (rapeseed meal) and 2.2 g/100 g (extruded soybean),** the larger difference, the more drastic hydrothermal treatment during the process (desolventisation, extrusion). Low effect was logically observed on cold pressed cakes.

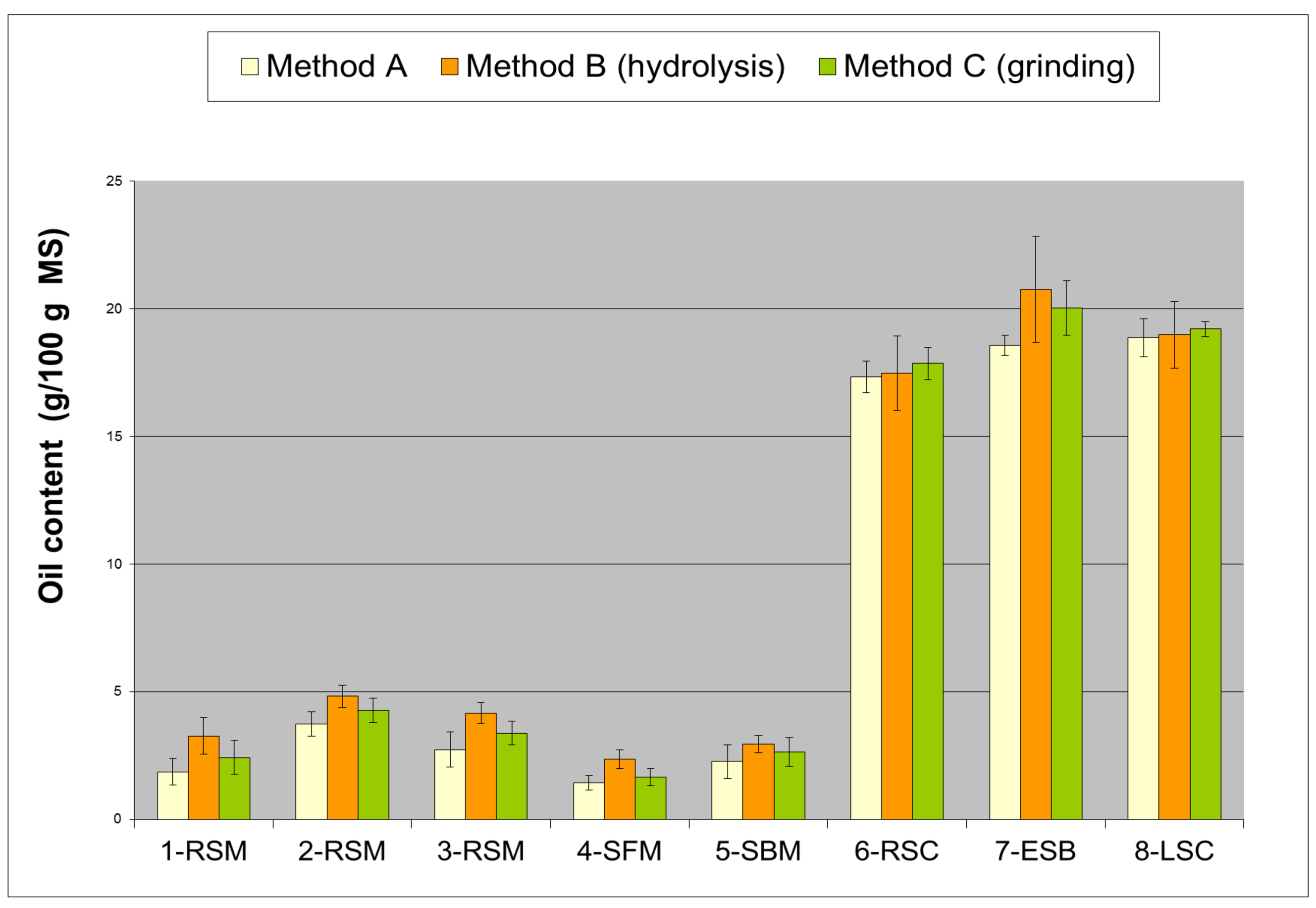
Grinding effect : the difference between results from methods C and A, representing the fraction of the entrapped oil not easily extractible by the solvent, was around **0.6 g/100g for rapeseed meal** and was lower for sunflower meal and soymeal. A high effect was observed with extruded soybean, probably due to the absence of a pressing treatment.

	Sample	1 Rapeseed meal	2 Rapeseed meal	3 Rapeseed meal	4 Sunflower meal	5 Soymeal	6 Rapeseed cake	7 Extruded Soybean	8 Linseed cake
A Solvent Extraction	Number of labs	10	10	10	9	10	10	9	10
	Mean	**1.869**	**3.742**	**2.745**	**1.442**	**2.275**	**17.352**	**18.582**	**18.880**
	SD_r	0.051	0.053	0.053	0.067	0.048	0.119	0.152	0.147
	CV_r	2.7%	1.4%	1.9%	4.7%	2.1%	0.7%	0.8%	0.8%
	SD_R	0.260	0.238	0.339	0.146	0.327	0.308	0.200	0.372
	CV_R	13.9%	6.4%	12.4%	10.2%	14.4%	1.8%	1.1%	2.0%
B Hydrolyse + Solvent Extraction	Number of labs	9	9	8	9	9	9	9	9
	Mean	**3.277**	**4.825**	**4.178**	**2.366**	**2.949**	**17.487**	**20.764**	**18.985**
	SD_r	0.185	0.073	0.098	0.067	0.056	0.267	0.126	0.456
	CV_r	5.6%	1.5%	2.3%	2.8%	1.9%	1.5%	0.6%	2.4%
	SD_R	0.358	0.213	0.197	0.176	0.167	0.729	1.043	0.654
	CV_R	10.9%	4.4%	4.7%	7.4%	5.7%	4.2%	5.0%	3.4%
C Grinding + Solvent Extraction	Number of labs	6	6	7	7	6	7	7	5
	Mean	**2.433**	**4.266**	**3.384**	**1.662**	**2.640**	**17.867**	**20.038**	**19.211**
	SD_r	0.150	0.124	0.154	0.168	0.199	0.159	0.148	0.142
	CV_r	6.2%	2.9%	4.5%	10.1%	7.5%	0.9%	0.7%	0.7%
	SD_R	0.332	0.238	0.232	0.168	0.283	0.313	0.534	0.142
	CV_R	13.6%	5.6%	6.8%	10.1%	10.7%	1.8%	2.7%	0.7%
Hydrolysis effect	Difference B - A	1.41	1.08	1.43	0.92	0.67	0.14	2.18	0.10
	Probability B > A	99.9%	100.0%	100.0%	100.0%	97.4%	54.3%	98.7%	52.7%
Grinding effect	Difference C- A	0.56	0.52	0.64	0.22	0.37	0.52	1.46	0.33
	Probability C > A	90.2%	93.7%	93.0%	83.1%	80.8%	84.6%	99.3%	76.0%

SD: standard deviation; CV : coefficient of variation; r:repeatability ; R:reproducibility, Probability B>A (or C>A): probability of B >A (or C>A) for each laboratory

What economical impact of the difference between methods ?

The underestimated oil content of rapeseed meal by 0.6 g/100 g may lead to a non extraction of 3.3 litres of oil per ton of crushed seeds, and a lost of value of 2 €/t !



Conclusion

The study showed that the three methods logically gave different results according to their principle and to the type of process used for the production of meal or cake.

Because of the thermal treatment occurring in the processing (desolventisation) of regular industrial meal, differences were non negligible in all cases, particularly for rapeseed meal.

The choice of the most relevant method should be finally done considering the needs of the user of the results:

- **method B**, for a more economical feedstuff formulation ,
- **method C**, to optimize the oil extraction process in crushing plant.

The detailed results of this study will soon be published in the journal "*Oleagineux, Corps gras, Lipides »* (www.revue-ocl.fr)

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