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A White Paper from FOSS

Comparing methods for fibre determination in food and feed

This White Paper gives an overview of different fibre determination methods and also an indication of relative performance for animal feed based on results reported from the proficiency testing scheme of the Association of American Feed Control Officials.

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Dedicated Analytical Solutions

Crude fibre

In the so-called Weende method, (fig. 1) Crude Protein, Crude Fat and Crude Ash are determined and the moisture and Carbohydrate content can then be calculated by difference: Carbohydrates = amount of total sample – Moisture – Crude Protein – Crude Fat.

In addition, Crude Fibre determination by acid hydrolysis with 1,25% H2SO4 is used for the extraction of sugars and starch, followed by alkaline hydrolysis with 1,25% NaOH, which removes proteins and some hemi-cellulose and lignin (fig. 2).Crude Fibre is commonly used to estimate the quality of foods of plant origin on the premise that it constitutes their least digestible fraction. The Nitrogen Free Extracts (NFE) are calculated by forming the difference (total) Carbohydrates minus Crude Fibre.

Though developed in the beginning of the 19th century many estimates of the nutritive value of vegetables and forages are still calculated on basis of the Weende method and crude fibre values even though there are several challenges with the crude fibre method as an estimate of the amount of fibre or plant cell wall.

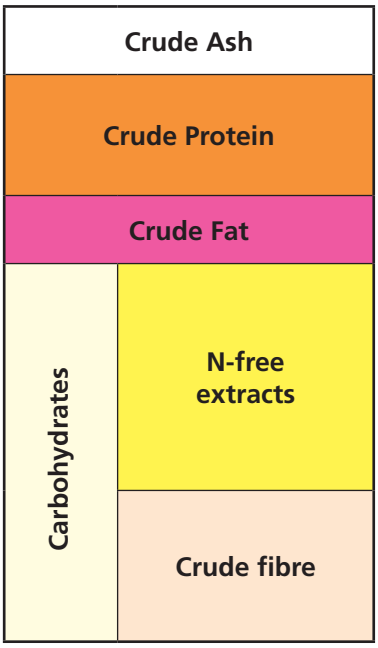

Fig. 1, Weende method.

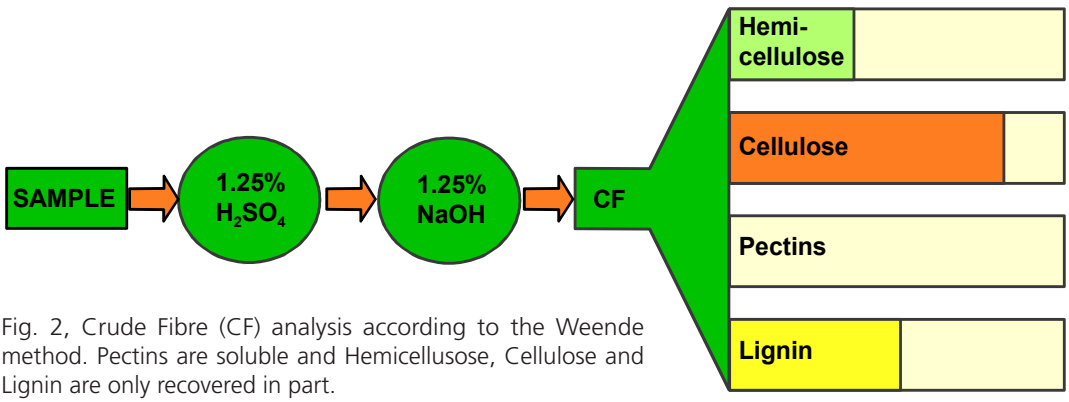

Fig. 2, Crude Fibre (CF) analysis according to the Weende method. Pectins are soluble and Hemicellusose, Cellulose and Lignin are only recovered in part.

On average, 80% of the hemicellulose or pentosans and from 50 to 90% of the lignin are removed by the acid and alkaline sequential extraction, while cellulose recovery is 50-80% (fig. 2). Thus, much of the hemicellulose and lignin appears in the nitrogen-free-extract (NFE) to be counted as available carbohydrate (fig. 1). The NFE of straws and grasses may contain as much as 90% of these substances (1). Because of the failure of the crude fibre method to recover indigestible substances, NFE appears less digestible than crude fibre in a significant number of cases. In the case of vegetables and cereals, the error is less because of the relatively lower content of hemicelluloses and lignin. However, it may be substantial.

Detergent fibre

There have been various attempts to replace the crude fibre method with a system of analysis which gives a better characterisation of the less nutritive fraction of food. Most successful has been the concept of detergent fibres developed by van Soest and colleagues (1), see figures 3 and 4.

In a first step, the sample is treated with a neutral detergent solution (NDS) and rinsed with a heat-stable amylase to make the sugars, starches and pectins soluble. The remaining residue consists of the non or less-digestible cell wall substances hemicellulose, cellulose and lignin. In a second step, hemicellulose is made soluble using an acid detergent solvent (ADS). The residue, consisting of cellulose and lignin is then treated with concentrated sulfuric acid, dissolving the cellulose and leaving the lignin in the residue. These steps can be performed consecutively or separately to determine the neutral detergent fibre (NDF), acid detergent fibre (ADF) and acid detergent lignin (ADL).

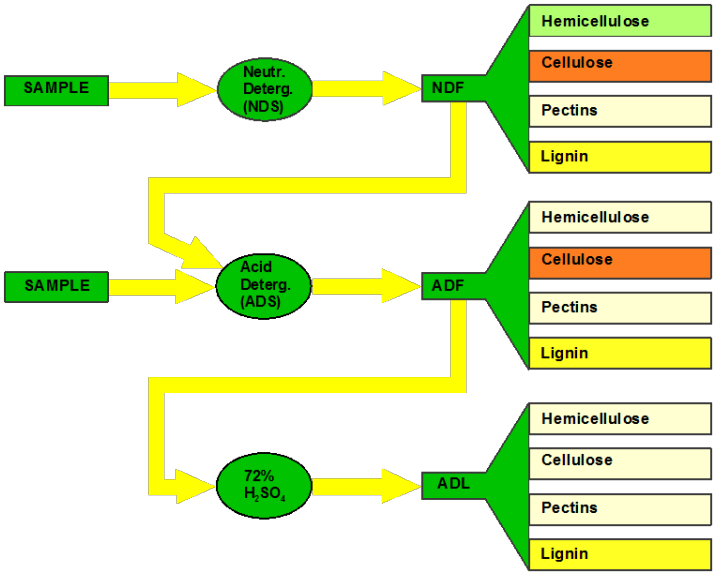

Fig. 3, Detergent Fibre according to van Soest

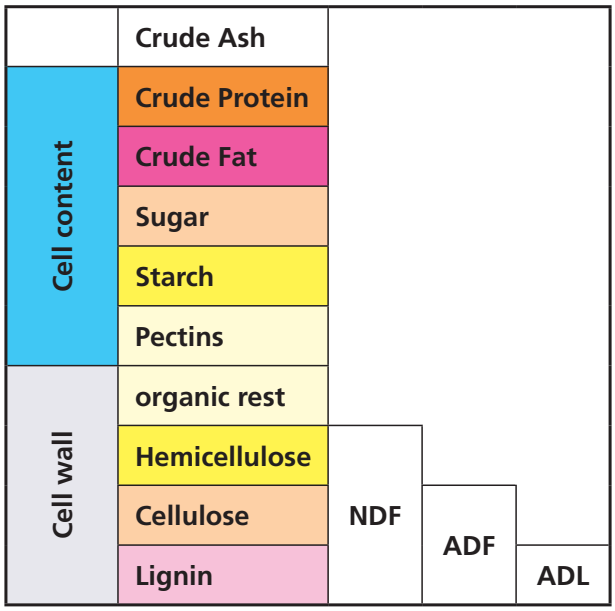

Fig. 4, Modified Weende method according to van Soest

Global standards

Recently some new global standards for aNDF (2) and ADF/ADL (3) have been presented in the FOSS journal *In Focus*. Both are using the so-called Fibertec (or crucible) method.

A criterion for evaluating fibre methods is not only the recovery of indigestible plant residues, but also the analytical performance of the methods and their official status. For crude fibre, numerous methods may be in use, but only a few have reached the status of an international standard (see table 1). ISO 5498 describes a general method and ISO 6541 a method for some food products. ISO 12099 gives guide lines for the application of NIR spectrometry and underlines the importance of using validated standard methods for the calibration and validation of NIR methods. One of the most widely spread methods is the so called crucible or Fibertec method according to ISO 6865, which has also been adopted as official method within the European Union (4).

ISO 5498:1981	**Agricultural food products** Determination of crude fibre content - General method
ISO 6541:1981	**Agricultural food products** Determination of crude fibre content - Modified Scharrer method
ISO 12099:2010	**Animal feeding stuffs, cereals and milled cereal products** Guidelines for the application of near infrared spectrometry
ISO 6865:2000	**Animal feeding stuffs** Determination of crude fibre content - Method with intermediate filtration

Table 1, International standards for the determination of crude fibre

Evaluation of methods for animal feed

Different methods for the determination of fibre have been evaluated by analysing results reported with the proficiency testing scheme of the Association of American Feed Control Officials (www.aafco.org). With some 300 participating laboratories reporting data on hundreds of different methods and parameters, the AAFCO PTS is one of the most comprehensive proficiency testing schemes. Table 2 shows the different fibre methods reported within this scheme, with the AAFCO reporting code in the first column.

For this study, the PTS samples from the year 2011, as shown in table 3, have been selected. The assigned values for the different methods, based on robust statistics, and their robust standard deviations have been studied. Several thousand reported data have been evaluated.

On an average some 130 labs (100%) reported data for crude fibre for the 12 samples, while 33% also reported data for ADF and 27% for NDF. The significance of differences between methods has been defined as the absolute difference between the results for the respective methods divided by the standard deviation. If this value is larger than 3 a significant difference can be stated.

In this study, with some exceptions, these values were below 1, i.e. no significant difference between methods. This indicates that the most common methods currently in use are robust and reliable.

AAFCO code	Standard	Description
004.00	AOAC 962.09 ISO 5498:2000	Crude Fibre, older general method
004.01	AOCS Ba6-84	Single Filt method
004.03	AOAC 978.1	Fritted Glass method
004.06	**ISO 6865 : 2000**	**Fibertec method**
004.07	AOCS Ba6a-05	ANKOM method
004.11	ISO 12099:2010	NIR methods
004.99	non standard Crude Fiber	Miscellaneous methods
008.02	**AOAC 973.18 ISO 13906:2008**	**Acid Detergent Fibre, Standard method**
008.05	non standard ADF	Acid Detergent Fibre-Hach Method
008.08	non standard ADF	Acid Detergent Fibre, ANKOM method
008.99	non standard ADF	Miscellaneous methods
009.04	non standard NDF	Methods with no enzym. Pretreat.
009.07	**AOAC 2002.04 ISO 16472:2006**	**Neutral Detergent Fibre with enz. Pretreat. Standard method**
009.09	non standard NDF	ANKOM method
009.99	non standard NDF	Miscellaneous methods

Table 2, Methods evaluated. Standard methods in bold, Global official methods highlighted

AAFCO no	Proficiency testing sample	CF	Difference Ftec - Ankom	Significance (dev/sd)	Precision rsd%
201121	Pig grower pellet	3,75	0,39	1,40	11,80
201122	Senior pig starter	2,74	0,03	0,10	15,90
201123	Infant Pig starter	2,48	0,08	0,20	16,00
201124	Lamb starter	6,25	0,39	0,90	8,00
201125	Dry Dog Food	4,65	0,18	0,50	11,10
201126	Chicken Starter	3,98	0,25	0,70	8,30
201127	Calf milk replacer	0,25	-0,29	-1,50	78,30
201128	Special beef feed lot	9,15	-0,93	-1,20	8,50
201129	Pig starter	2,28	0,19	0,70	15,80
201130	Chicken starter	3,44	0,30	1,00	9,50
201131	Corn protein concentrate	1,28	0,22	1,10	16,20
201132	Swine grower	3,11	0,15	0,50	10,30

Table 3, Studied samples and average results for crude fibre (CF), neutral detergent fibre (NDF) and acid detergent fibre (ADF)

The average significance of differences for crude fibre was 0,2 and for ADF and NDF 0,3. The average relative standard deviation of reported results was about 10%.

Significant differences were found for reported ADF and NDF values for the calf replacer sample between the Fibertec values and the Ankom teabag method values. On the other hand the reported values were quite low, below the limits of quantification for these methods.

For the special beef feed lot sample significantly higher values were reported for the Fibertec than for the Ankom teabag method. For ADF 13,6 vs 11,9 % and for NDF 22,5 vs 20,8%. The reason for this is not understood.

Also the reported NDF values for the corn protein concentrate sample show significant differences.

Conclusion

Although alternative methods may be faster and allow higher samples volumes to be run, the adherence to official methods is mandatory in cases of dispute and for labeling purposes. Automation options for the official methods would add further reliability while making them highly resource efficient.

References:

(1) Van Soest, P.J. and McQueen, R.W.: *The chemistry and estimation of fibre,* Proc. Nutr. SOC. (1973), vol. 32, p 123-130

(2) Möller, J.: Feed Control – *New Standard for Determination of Amylase-Treated Neutral Detergent Fibre (aNDF),* In Focus, Vol. 29, No 2, 2005, p 12-14

(3) Möller, J.: *Animal feeding stuff - Global Standard for the Determination of Acid Detergent Fibre (ADF) and Lignin,* In Focus, Vol. 32, No 2, 2008, p 22-24

(4) COMMISSION REGULATION (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed, published 26.2.2009 in the Official Journal of the European Union L 54/1.

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