ADF and ADL Determination in Oatmeal

Reference: **ISO 13906:2008**, **AOAC 973.18** Animal feeding stuffs - Determination of Acid Detergent Fibre (ADF) and Acid Detergent Lignin (ADL) contents.

Tested with **VELP Scientifica FIWE 6 Fiber Analyzer** (Code F30520200).

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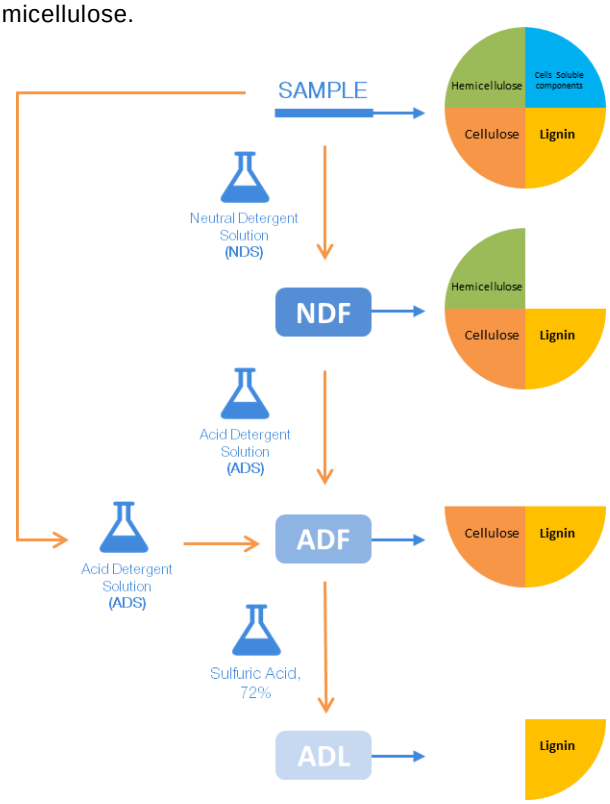
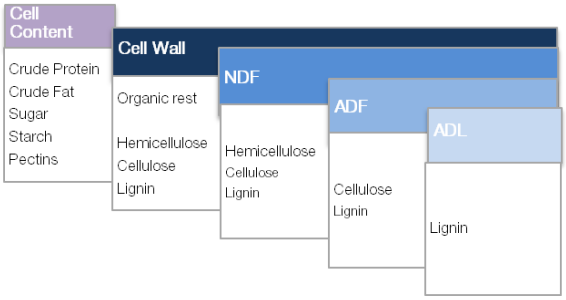
ADF and ADL DETERMINATION IN OATMEAL

Introduction

Oats are a whole-grain cereal, known scientifically as *Avena sativa*. They are mainly grown in North America and Europe. This whole-grain are a very good source of fiber, and are high in vitamins, minerals and antioxidants.
Oats contain more soluble fiber than other grains, leading to slower digestion, increased satiety and suppression of appetite. Most of the soluble fiber is composed of beta-glucans.
Beta-glucans are known to lower cholesterol levels and increase excretion of bile acids. They are also believed to cause a reduction in blood sugar and insulin levels after a carbohydrate-rich meal.
Daily consumption of beta-glucans has been shown to lower cholesterol, especially LDL (the "bad") cholesterol, and may therefore decrease the risk of heart disease.Oats also contain insoluble fibers, including lignin, cellulose and hemicellulose.





Determination of ADF and ADL contents in Oatmeal

ADF is determined in the first stage of the method. The Acid detergent solution (ADS) solubilizes the hemicellulose while lignin and cellulose remain insoluble. The residue is weighed for the determination of ADF and it includes cellulose and lignin. In the second stage, the remaining residue is solubilized by 72 % sulfuric acid, leaving the lignin (ADL) which is determined gravimetrically.

Reagents

1. ADS: Add 20 g cetyl(trimethyl)ammonium bromide ($C_{19}H_{42}BrN$) to 1 l of 0,5 mol/l sulfuric acid. Agitate to aid dissolution. Store the ADS at room temperature. If precipitation occurs, warm the solution to 25 °C and mix before use.
2. Sulfuric acid 72 % mass fraction, 12.00 mol/l (H_2SO_4)
3. Anhydrous acetone
4. n-Octanol, as antifoaming agent

Note: to simplify filtration, 1.00g of celite can be added to the crucible.

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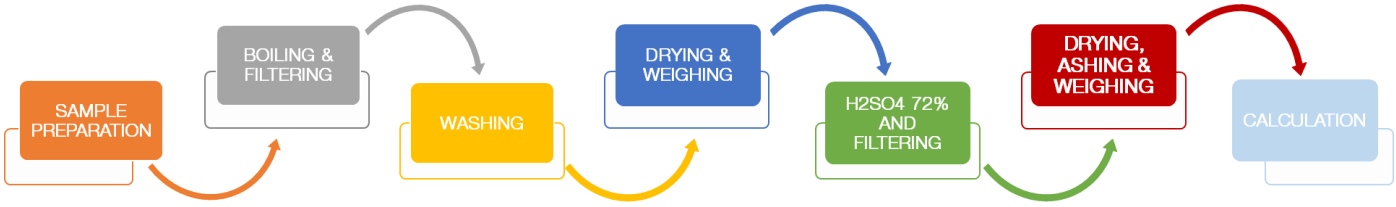
Sample

Oatmeal (VELP code A00000318)

ADF range: 10.27 ± 0.97 %
ADL value: < 1.5 %

Analysis Procedure

The diagram below shows the steps involved in the procedure:



Procedure for ADF determination

1. Dry the crucibles containing 1 g of celite in an drying oven at 102-105 °C for 2 h to 4 h. Cool them in a dessicator and weigh the tare (M_{tare} and B_{tare}) to the nearest 0.0001 g. Include a blank test every 20-30 samples.
2. Weigh 1 g ± 0.002 g of sample portion into each crucible (M_{sample})
3. Place the crucibles in the FIWE unit and from the top of the glass columns pour 100 ml of ADS to each crucible and mix using back pressure
4. Add from 2 to 4 drops of n-octanol to prevent foaming and heat to boiling point (set heating power at 10)
5. Adjust heater at power 5-7 and allow to boil for 60 min ± 5 min.

   Note: Measure the boiling time from when the solution reaches the boiling point
6. Connect to vacuum to start the filtration through crucibles. If plugged, the crucibles may be backflushed using minimum back pressure
7. Wash three times with 30 ml of hot water
8. Fill crucibles with 25 ml of acetone and use minimum back pressure to disperse the particles. Soak from 3 min to 5 min and evacuate. Repeat the acetone wash
9. Remove the crucibles from the unit and air-dry for 10 min to remove acetone
10. Dry the crucibles at 130 °C ± 2 °C for 2 h or at 105 ± 2 °C for at least 8 h. Cool to room temperature in a desiccator and weigh to the nearest 0,0001 g. Record the mass of sample and blank as M_{dry1} and B_{dry1} respectively.

Procedure for ADL determination

1. Place the crucibles containing acid detergent fibre in the FIWE unit. Add 25 ml sulfuric acid 72 % cooled to 15 °C and stir at hourly intervals with back pressure.
2. After 3 h, filter as complete as possible with vacuum, and wash with hot water until acid-free.
3. Dry the crucibles at 130 °C ± 2 °C for 2 h or at 105 ± 2 °C for at least 8 h. Cool to room temperature in a desiccator and weigh to the nearest 0,0001 g. Record the mass as M_{dry2} and B_{dry2}.
4. Ignite the crucibles with the residue in a furnace at 525 ± 15 °C for at least 3 h or until carbon-free. Leave to cool in a desiccator and weigh to the nearest 0,0001 g (M_{ash} and B_{ash}).
5. Remove ash and if necessary clean the crucibles by an oxidizing procedure.

Calculation

ADF

ADF %= (M_{dry1} - M_{tare} - (B_{dry1}-B_{tare})) *100/M_{sample}

M_{dry1} = sample weight after drying

ADL

ADL %= (M_{dry2} - M_{ash} - (B_{dry2}-B_{ash})) *100/M_{sample}

M_{dry2} = sample weight after drying

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M_{tare} = tare of the sample
M_{sample} = sample weight
B_{dry1} = blank weight after drying
B_{tare} = tare of the blank

M_{ash} = sample weight after ashing
M_{sample} = sample weight
B_{dry2} = blank weight after drying
B_{ash} = blank weight after ashing

Typical Results on Oatmeal

M_{tare} (g)	M_{sample} (g)	M_{dry1} (g)	ADF %	M_{dry2} (g)	M_{ash} (g)	ADL %
32.1910	Blank	32.1792		30.7160	30.7096	
31.7299	1.0288	31.8102	**9.57**	31.7469	31.7293	**1.09**
31.6613	0.9989	31.7379	**9.49**	31.6772	31.6579	**1.09**
31.2783	0.9999	31.3563	**9.62**	31.2853	31.2677	**1.12**
31.6248	0.9975	31.7021	**9.57**	31.6481	31.6273	**1.07**
31.2756	0.9898	31.353	**9.66**	31.2923	31.2726	**1.04**
		Average ± SD%	**9.58 ± 0.06**		**Average ± SD%**	**1.08 ± 0.03****
		RSD% *	**0.7**		**RSD% ***	**2.7*****

ADF Blank (B_{dry1}-B_{tare}) results: – 0.0182 g
ADL Blank (B_{dry2}-B_{ash}) results: + 0.0064 g
***** RSD% = (Standard Deviation * 100) / Average
**According to reference method ISO 13906:2008, ADL mass fraction resulting < 1.5 % are reported as “ADL < 1.5 %”.
*** According to reference method ISO 13906:2008

Conclusion

The obtained results are reliable and in accordance with the expected range.
The use of an extraction apparatus purposely devised for this method as FIWE unit, makes very easy the standardization of analytical conditions.
The FIWE Series is suitable for Crude Fiber (CF), Neutral Detergent Fiber (NDF), Acid Detergent Fiber (ADF) and Acid Detergent Lignin (ADL).

Benefits of FIWE are:

- 3 or 6 positions simultaneously: FIWE units can support up to 3 (FIWE 3) or 6 (FIWE 6) crucibles. Samples can also be processed individually
- Time saving: fast analysis (2 hours with FIWE vs. 6 hours manually)
- Easy to use: convenient filtration, with pump and air pressure
- Precision and accuracy: high reproducibility of the results: **± 1%** relative or better

In order to avoid losses of fiber, it’s important to remember that crucibles life is around 20-30 analysis, because the fritted filter could be damaged from basic and acid solutions. Hence it’s suggested to change them after 20-30 analysis.

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