Crude Fiber Determination in Dog Food according to Weende Method


Tested with VELP Scientifica FIWE 6 Fiber Analyzer (Code SA30520200).
Introduction

Fibre is the indigestible part of plant foods. It is classed as a complex carbohydrate that is resistant to digestion in the small intestine. Fibre is important to keep the digestive system healthy. As it is not digested in the small intestine, fibre passes through unchanged until it reaches the large intestine. In the large intestine, bacteria act on the fibre to ferment it. The primary function and benefit of fibre is to increase bulk and water in intestinal contents, maintain optimum levels of good bacteria and promote gut health.

Fibre has been successfully used in pet foods to help with weight management, diabetes mellitus, diarrhoea and constipation. A pet food that is high in slowly fermentable fibre increases the bulk of the stomach contents, helping pets feel full and satisfied. This ensures they eat fewer calories and is helpful in long term weight management. Slow and rapidly fermentable fibres have beneficial effects on blood glucose in diabetic animals. Fibre can absorb water content in cases of diarrhoea and adds moisture in cases of constipation.

The performance of VELP FIWE Fiber analyzer was evaluated by participating in the proficiency testing program organized by BIPEA.

Sample of Dog food is analyzed using FIWE Fiber analyzer and the obtained results (as % CF) were compared with the BIPEA tolerance range.

Fiber Determination in Dog food according to Weende method

The method is based on the solubilization (digestion) of non-cellulosic compounds by sulfuric acid and potassium hydroxide solutions. Crude fiber is the loss on ignition of the dried residue remaining after digestion of the sample and determined by weight difference. This method is applicable to grains, meals, flours, feeds, and fiber-bearing material from which fat can be extracted to leave workable residue.

Reagents

1. Sulfuric acid (H₂SO₄) 1.25% - 0.255 ± 0.005 N. 12.5g, 98% concentrated to 1000 ml with distilled water
2. Potassium hydroxide (KOH) 1.25% - 0.223 ± 0.005 N, free from carbonate. 12.5 g to 1000 ml with distilled water
3. n-octanol as antifoam
4. Anhydrous acetone
5. Petroleum ether 40-60 °C

As filtration aid it's possible to use Celite (code A00000097), calcined in a muffle for 4 hours at 500 °C.

Sample

BIPEA Dog food ID: 007-1913-0145
Crude Fiber Assigned value: 2.6 % Tolerance range: 2.3 – 2.9 %

Analysis Procedure

Before the fiber determination it's necessary to defat the feed sample, since its fat content is more than 10 %.

Defatting procedure:
1. Weigh 1 g ± 0.0001 g (Msample) of homogeneous sample into each crucible containing about 1 g of celite, if necessary.
2. Place the crucibles on the cold-extraction unit COEX and extract 3 times with about 30 ml of petroleum ether 40-60°C. Apply vacuum to remove traces of solvent, air-dry for 10 min to ensure that all traces of solvent are removed.
The diagram below shows the steps involved in the fiber determination procedure:

1. Move the crucibles from COEX directly to FIWE directly. It’s not necessary to weigh them.
2. Add 1.25% sulfuric acid up to the 150 ml notch, after preheating by RC2 hot plate (cod. F20700430) in order to reduce the time required for boiling.
3. Add 3-5 drops of n-octanol as antifoam agent.
4. Boil 30 minutes exactly from the onset of boiling.
5. Connect to vacuum for draining sulfuric acid.
6. Wash three times with 30 ml of hot deionized water (crucible filled up to the top), connecting each time to compressed air for stirring the content of crucible.
7. After draining the last wash, add 150 ml of preheated potassium hydroxide (KOH) 1.25% and 3-5 drops of antifoam.
8. Boil 30 minutes.
10. Perform a last washing with cold deionized water aimed to cool the crucibles and then wash three times the crucible content with 25 ml of acetone, stirring each time by compressed air.
11. Remove the crucibles and determine the dry weight after drying in an oven at 130 °C for 2 hours or at 105 °C for at least 8 hours. Let cool in a desiccator. This weight represents the crude fiber plus ash content ($M_{\text{dry}}$).
12. The crucibles are then placed in a muffle and heat up to 525 °C for three hours and reweighed after cooling in a desiccators ($M_{\text{ash}}$).
13. Remove ash and if necessary clean the crucibles by an oxidizing procedure.

Calculation

\[
\% \text{ Crude Fiber} = \frac{M_{\text{dry}} - M_{\text{ash}}}{M_{\text{sample}}} \times 100
\]

$M_{\text{sample}}$ = sample weight
$M_{\text{dry}}$ = crucible weight with fiber and ashes, after drying in an oven at 130 °C for 2 hours
$M_{\text{ash}}$ = crucible weight with ashes, after muffle at 525 °C for three hours
CRUDE FIBER DETERMINATION IN DOG FOOD

Results on Dog food sample:

<table>
<thead>
<tr>
<th>$M_{sample}$ (g)</th>
<th>$M_{dry}$ (g)</th>
<th>$M_{ash}$ (g)</th>
<th>Fiber %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0120</td>
<td>31.6378</td>
<td>31.6099</td>
<td>2.76</td>
</tr>
<tr>
<td>1.0820</td>
<td>31.1630</td>
<td>31.1330</td>
<td>2.77</td>
</tr>
<tr>
<td>1.0557</td>
<td>30.6406</td>
<td>30.6118</td>
<td>2.73</td>
</tr>
<tr>
<td>1.0131</td>
<td>31.4996</td>
<td>31.4720</td>
<td>2.72</td>
</tr>
<tr>
<td>1.0001</td>
<td>31.7794</td>
<td>31.7522</td>
<td>2.72</td>
</tr>
<tr>
<td>0.9916</td>
<td>31.0927</td>
<td>31.0658</td>
<td>2.71</td>
</tr>
</tbody>
</table>

Average ± SD% 2.74 ± 0.02

RSD% * 0.84

Tolerance range: 2.3 – 2.9 % CF

*RSD% = (Standard Deviation * 100) / Average

Conclusion

The obtained results are reliable and in accordance with the assigned value.

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.*