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Applikationsbericht

Extraction of Fat from Animal Feed Products using a Waters MV-10 ASFE System

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Abstract

In this note, we have demonstrated that SFE can be used as an alternative extraction technique to the industry-dominant ASE technique for determining the fat content in animal feed. The SFE-based approach also has the potential to eliminate hydrolysis steps, often required when other extraction techniques are used.

Benefits

- SFE can be used as an alternative extraction technique to ASE for fat analysis.
- Software-controlled extraction process offers superior accuracy and precision for fat extraction and ensuing analysis.
- The SFE-based approach has the potential to eliminate hydrolysis steps associated with fat analysis.
- Positive impact on environmental sustainability, and substantial cost savings.

Introduction

Fat content is an important nutritional and quality control parameter in the manufacturing of animal feed. As a result, a fast and reliable measurement is required for process optimization. Various solvent extraction techniques, including soxhlet and accelerated solvent extraction (ASE), followed by gravimetric analysis, are commonly used for fat content determination. Typical solvents used for these extractions include hexane and ether.

With the increasing awareness of environmental sustainability, supercritical fluid extraction (SFE) has been used to remove fat in various matrices. The low polarity of supercritical CO_2 (SC-CO_2) makes it an ideal solvent for fat.[1-2] Furthermore, the solvation power can be tuned by varying the pressure and temperature, and/or mixing with organic solvents to achieve the desired selectivity. Other advantages of SFE include: carbon dioxide is relatively nontoxic, noncombustible, and inexpensive; easy recovery of the extract; and reduced organic solvent consumption and disposal.

In this application note, we present the performance evaluation of a Waters MV-10 ASFE System for the extraction of fat from a variety of animal feed products. The results are compared to those obtained using

an ASE-based methodology. The prospect of adopting SFE for routine fat analysis is also discussed.

Experimental

Sample description

Various commercially available animal feeds were used as received unless noted otherwise.

Method conditions

SFE conditions

For each SFE experiment, approximately 3 g of finely ground animal feed were loaded into a 5-mL extraction vessel. The extracted fat content was collected into a pre-weighed scintillation vial. After the solvent in the extract was removed, using a Zymark Turbovap LV, the vial was reweighed. The weight difference was considered the fat content in the sample.

SFE conditions

Temp.:	40 °C
Pressure:	3625 psi
Co-solvent:	Methanol
Co-solvent%:	10%
Flow rate:	8 mL/min
Software:	ChromScope
Total run time:	25 min

ASE conditions

For each ASE experiment, approximately 3 g of finely ground animal feed were loaded into a 10-mL

extraction cell. The extracted fat content was collected into a pre-weighed collection bottle vial. After the solvent in the extract was removed, using a Zymark Turbovap LV, the bottle was reweighed. The weight difference was considered the fat content in the sample.

Temp.:	125 °C
Pressure:	1000 psi
Extraction solvent:	Hexane
Oven heat up time:	5 min
Oven heat up cycles:	2
Static time:	3 min
Static cycles:	2
Flush volume:	60%
Purge time:	1 min
Purge cycles:	2
Total run time:	18 min

Results and Discussion

Comparison between SFE and ASE

Table 1 summarizes the fat analyses of 15 different animal feed products. To gauge the general applicability of SFE in fat analysis, a conscious effort was made in sample selection to ensure the sample diversity in both product form and intended animals. It is also noteworthy that samples 8 and 9 were hydrolyzed by acid prior

to ASE. In ASE-based methodology, it is a common practice to include a hydrolysis step prior to extraction to disintegrate the sample, to disrupt the plant cell walls, and to liberate the fat.

Vial No.	Type	Target animal	Claim (%)	Fat by ASE (%)	Fat by SFE (%)
1	Pellet	Equine	12.000	12.950	12.209
2	Textured	Cattle	2.000	4.300	2.093
3	Pellet	Equine	7.000	7.006	8.033
4	Ground	Poultry	8.000	7.945	8.652
5	Hay	Cattle	4.000	3.978	4.227
6	Textured	Multispecies	5.000	4.966	5.840
7	Textured	Equine	2.750	3.447	2.602
8	Dried bakery product	Ingredient	7.000	6.800*	7.090
9	Pellet	Dog	14.000	13.600*	14.693
10	Pellet	Sheep	2.000	2.833	2.646
11	Pellet	Lamb	2.000	2.735	2.120
12	Textured	Equine	10.000	9.811	10.070
13	Dried beet pulp	Ingredient	0.250	0.671	0.238
14	Textured	Cattle	3.500	3.589	4.081
15	Pellet	Equine	12.000	12.400	12.265
*Samples were hydrolyzed by acid prior to ASE.					

Table 1. Fat content of 15 different animal feed products.

The hydrolysis step, however, didn't seem necessary for the SFE approach. For both samples 8 and 9, the fat content extracted by SFE was slightly higher than those by ASE. This is likely due to the unique properties of SC-CO_2. Its gas-like diffusivities and liquid-like solvating strengths enable CO_2 to easily penetrate those otherwise difficult matrices, such as glutenous particles in dried bakery product (sample 8), and dissolve and transport the fat contents.

Figure 1 shows the correlation of the fat content extracted by SFE and ASE. It is evident there is a general agreement between the two extraction techniques, suggesting SFE could become a potential alternative to ASE in fat analysis.

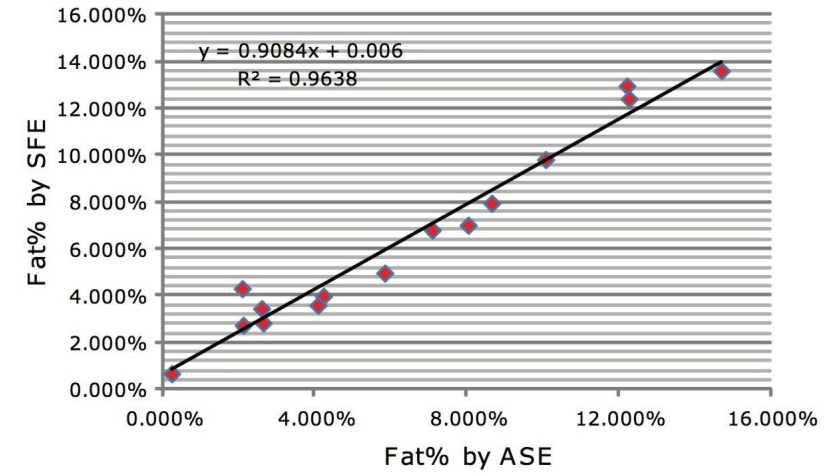

Figure 1. Correlation of fat content extracted by SFE and ASE.

Figure 2 shows the distribution of the Fat%, based on the SFE approach. The Fat% ranges from 95% to 132%, with the majority of the samples (11 out of 15) ranging from 95% to 110%, showing excellent agreement with the label claims.

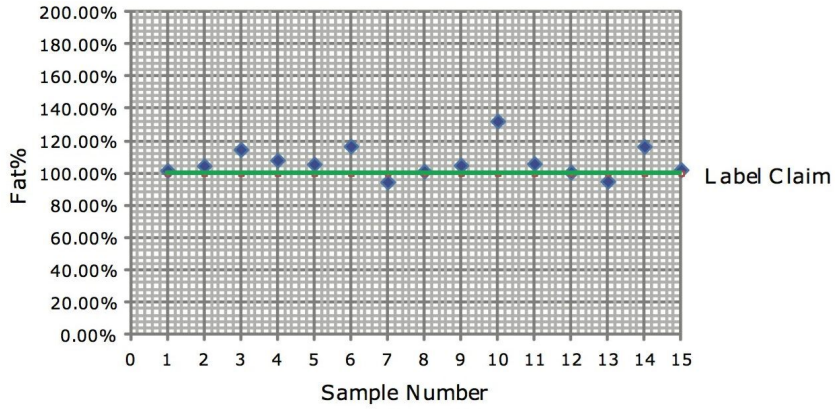

Figure 2. Extracted Fat% by SFE vs. label claims for 15 samples.

Table 2 summarizes a reproducibility study of the SFE-based approach for fat analysis. The RSD% of the five replicate experiments is less than 2%. The MV-10 ASFE System is controlled by ChromScope Software. After samples are loaded onto the extraction vessels, there is no user intervention required for the extraction; thus, offering superior precision for the ensuing analyses.

Vial No.	Empty vial (g)	Fat + vial (g)	Sample wt. (g)	Fat%
1	24.6961	24.8251	3.15	4.095
2	24.7558	24.8848	3.09	4.175
3	24.6134	24.7424	3.07	4.202
4	24.9672	25.0962	3.22	4.006
5	24.8170	24.9486	3.18	4.138
Mean				4.123
STD				0.000767
RSD%				1.861%

Table 2. Fat analysis results of five replicate experiments.

It should be noted that an excessive extraction time (25 min) was used for all SFE experiments to ensure the thoroughness of the extraction. In practice, the fat content started to elute out in less than 5 min. Preliminary time course study indicated that the optimal extraction time for fat analysis can be shortened to less than 10 min.

Conclusion

In this note, we have demonstrated that SFE can be used as an alternative extraction technique to the industry-dominant ASE technique for determining the fat content in animal feed. Software controlled extraction process offers superior accuracy and precision for the overall analyses. The SFE-based approach also has the potential to eliminate hydrolysis steps, often required when other extraction techniques are used. Finally, in addition to the positive impact on environmental sustainability, the use of methanol in SFE compared to hexane used in ASE could also result in substantial cost savings.

References

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2. King JW, Eller FJ, Snyder JM, Johnson JH, McKeith FK, Stites CR. Extraction of fat from ground beef for nutrient analysis using analytical supercritical fluid extraction. *J Agric Food Chem*. 1996 Sept; 44:2700-4.

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MV-10 ASFE System <https://www.waters.com/134661549>

ChromScope Software <https://www.waters.com/134647658>

720004446, September 2012