

Extraction of Oils from Oilseeds by Accelerated Solvent Extraction (ASE)

INTRODUCTION

Accelerated Solvent Extraction (ASE[®]) is an extraction method that significantly streamlines sample preparation. A commonly used solvent is pumped into an extraction cell containing the sample, which is then brought to an elevated temperature and pressure. Minutes later, the extract is transferred from the heated cell to a standard collection vial for cleanup or analysis. The entire extraction process is fully automated and performed in minutes for fast and easy extraction with low solvent consumption.

Oils for foods and cooking are derived from oilseeds like canola, soybeans, corn, flax, cotton, etc. The production of oil from oilseeds is an important business, and agronomists are continuing to investigate ways to improve the oil output of the seeds as well as ways to control the composition of the oil itself.

A common method used to remove the oil from the oilseeds is solvent extraction. Existing solvent extraction methods use large volumes of solvent (typically several hundred milliliters) and long extraction times (8–16 h) to remove the oil from the seeds. Once the oil is removed, the weight percent of oil in the seeds can be determined, and the composition of the oils can be studied.

ASE is a relatively new extraction technique that uses elevated temperature and pressure to expedite the removal of analytes from various matrices. It is accepted by the U.S. EPA as Method 3545 for environmental samples, and it has been applied to other sample extraction areas. ASE can be used to extract a 10-g sample in about 14 min with a total solvent consumption of approximately 16 mL.

The procedures described in this application note apply to the determination of oil content in oilseeds. The oil content is determined by collecting the extracts in preweighed vials, evaporating the solvent with a nitrogen stream and reweighing the vials.

PROCEDURE

Air samples were passed through a solid sorbent tube (4 mm i.d. × 7 cm long) containing 150 mg of impregnated charcoal (Barnebey-Cheney Co., Columbus, OH, No. 580-20, M-2820) at 50–200 mL/min. for 3–5 h. The charcoal was removed to a tube containing 15.0 mL of 0.1% H₂O₂ and shaken for one hour, then sonicated for 20 min. The solution was filtered through a 0.45 μm membrane filter, then injected into an Ion Chromatograph.

EQUIPMENT

ASE 200 Accelerated Solvent Extractor* equipped with 11 or 22 mL cells

Analytical balance

Dionex vials for collection of extracts (40 mL, P/N 49465; 60 mL, P/N 49466)

**ASE 150 and 350 can be used for equivalent results.*

SOLVENTS

Petroleum ether (pesticide quality or equivalent)

ASE 200 CONDITIONS

Oven Temperature:	105 °C
Pressure:	1500 psi*
Oven Heatup Time:	5 min
Static Time:	10 min
Flush Volume:	100%
Purge Time:	60 s
Solvent:	Petroleum ether
Static Cycles:	3

**Pressure studies show that 1500 psi is the optimum extraction pressure for all ASE applications.*

It may be necessary to perform three complete extractions of each sample and combine the results from each extraction. If the samples contain unsaturated fats that may be oxidized during extraction, and these fats are to be analyzed, the solvent should be degassed to avoid potential oxidative degradation.

SAMPLE PREPARATION

It is important to reduce the particle size of all samples by grinding or another appropriate procedure. The particle diameter should be less than 3 mm. Samples should also be mixed with an inert filler such as clean sand to prevent sample compaction during extraction. Wet samples (>10 wt%) should be dried in an oven at 105 °C for at least 2 h or mixed with a desiccant such as ASE Prep DE (diatomaceous earth), P/N 062819, (1:1 w/w) prior to extraction. Depending on the density of the samples, it may be necessary to use the 22-mL cells.

Sample Size	4 g ground seed
Oven	130 °C, 2 h
Extract	4 h, drain solvent and cool
Regrind	7 min
Extract	2 h, drain solvent and cool
Solvent	Petroleum ether
Total Volume Solven Used	150–250 mL
Total Time	10.5 h

Extraction Temp. (°C)	PV (µg/L or ppm)	FFA (%)
50	2.24	0.57
70	3.15	0.80
110	3.23	1.05
130	2.08	1.28

PROCEDURE

Grind the sample to an appropriate particle size (< 2- to 3-mm diameter). Place a cellulose disk at the outlet end of the extraction cell. Weigh 3 to 10 g of sample in a beaker. Mix with ASE Prep DE if the sample is wet. If dry, mix with sand (approximately 1:1) and load into the extraction cell. Fill any void volume with clean sand.

Place closed cells into upper carousel with the appropriate number of clean, preweighed collection vials. (It is best to weigh the vials without the lids and septa before extraction and then again after solvent evaporation to eliminate any contribution caused by the loss of material from the lids or septa.) Set the method conditions on the ASE 200 system and initiate the run.

Upon completion of the extraction, place the collection vials in an evaporator with nitrogen to evaporate the solvent. Then, weigh the residue to determine the percent of oil in the original sample.

DISCUSSION AND RESULTS

The example used here (canola seeds) contain approximately 45 wt percent oil. To understand the advantages of ASE, it is important to understand the currently used extraction methodology. The method currently used is specified as AOCS (American Oil Chemist Society) Official Method AM 2-93 which is based on the FOSFA (Federation of Oil Seeds and Fat Association) Official Method. The specifics of this method are given in Table 1.

There are several ways to compare the performance of ASE against that of the standard method. First, the weight percent of oil in the seeds was determined to be 44.9% with 0.31% RSD (n = 3) as compared to 45.2%

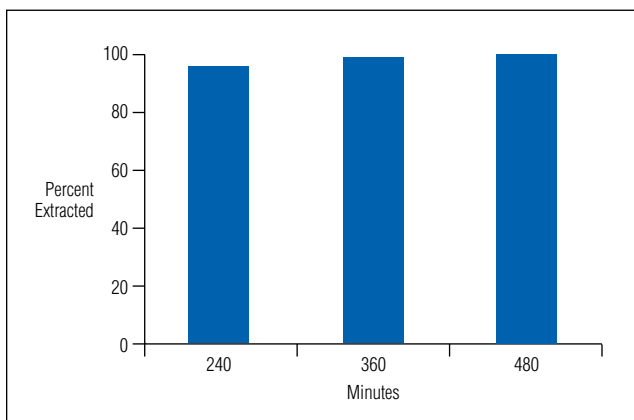


Figure 1. Extraction of oil from oilseeds: FOSFA.

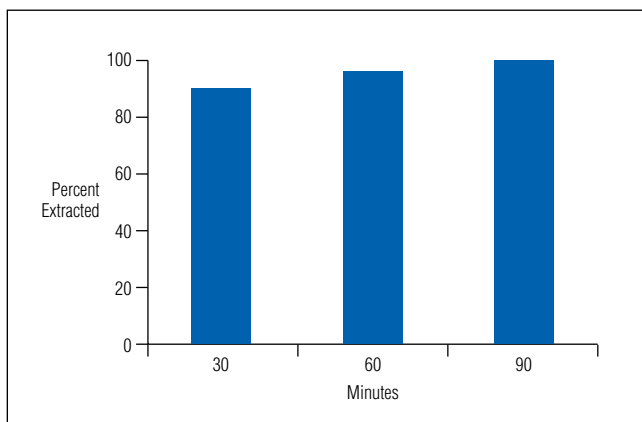


Figure 2. Extraction of oil from oilseeds: ASE.

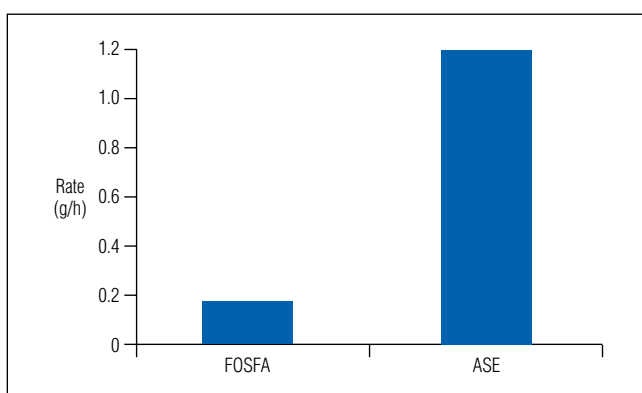


Figure 3. Rate of oil extraction from oilseeds.

with 0.24% RSD ($n = 12$) for the AOCS method. Second, the percent of the total oil extracted as a function of time can be compared for the two techniques (see Figures 1 and 2). Third, the weight of oil extracted per unit time can be contrasted for ASE and the FOSFA procedure (see Figure 3). As can be seen, ASE gives comparable results faster and with less solvent usage.

Studies were performed to determine the effect of the elevated temperatures on the triglycerides during the extraction by ASE. Two values, peroxide value (PV) and free fatty acid (FFA), were determined for oils that had been extracted at different temperatures. The resulting data are given in Table 2.

The PV is a measure of the oxidation of the triglycerides, and any value less than 5 ppm shows no significant oxidation as compared to Soxhlet. The FFA is a measurement of triglyceride degradation, and typical values from Soxhlet are 0.5–1.0%. These data show that no significant oxidation or triglyceride degradation occurs during ASE.

CONCLUSION

The data presented here demonstrate that the ASE system can be used to extract and determine the oil content of oilseeds. The data generated are equivalent to those obtained by existing methods, but the time and solvent usage are significantly less with the ASE system.

ACKNOWLEDGMENTS

The authors would like to thank the Grain Research Laboratory, Canadian Grain Commission, Winnipeg, Manitoba for providing the values for peroxide and free fatty acid contents.

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LPN 0786-03 PDF 4/11
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