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Soxhlet Extraction of Fat from French Fries

Beverly Swaile, University of Cincinnati, Cincinnati, OH

INTRODUCTION

Description

Lipids are a group of substances that, in general, are soluble in ether, chloroform, and other organic solvents but are relatively insoluble in water. An accurate and precise quantitative analysis of lipids in foods is important not only for nutritional labeling, but also for determining whether the food meets the standards for identity and uniformity, and for understanding the effects of fats and oils on the functional and nutritional properties of foods. The validity of the fat analysis of a food depends on many factors, including proper sampling and preservation of the sample before the analysis.

Because of commercial regulations, it is important for food producers to be able to report fat content in a serving size of a food item. The Soxhlet procedure allows for the calculation of total lipid (fat) content in french fries or other food substances. In many of its published methods, the U.S. Environmental Protection Agency requires Soxhlet extraction of components from a variety of sample matrices including foods and soils.

This experiment involves the extraction of fat from commercial (fast-food) french fries by an exhaustive extraction with solvent using a Soxhlet extractor apparatus.

Student Audience

This activity is appropriate for chemical technology students.

Goals for the Activity

The following is a method for the extraction and determination of percent fat content in a food sample by weight. Students will develop skills including use of a micro-Soxhlet extractor and a sample concentrator and the ability to calculate percent fat content by weight in a sample.

Recommended Placement in the Curriculum

This activity should be conducted during analytical or organic chemistry.

STUDENT HANDOUT
Soxhlet Extraction of Fat from French Fries

Purpose
This experiment involves the extraction of fat from commercial (fast-food) french fries by an exhaustive extraction with solvent using a Soxhlet extractor apparatus.

Scenario
A recent marketing study has concluded that french fries served at Company X are more "greasy tasting" to consumers than fries sold by Companies Y and Z. As a top technician at Company X, you have been personally assigned by the CEO to a team that will evaluate this problem by extracting the fat from the finished fry product and comparing the average lipid percentage to that of your competitors. Your first mission is to conduct library research on methods for fat extraction. After searching for an acceptable extraction technique, you discover that although many techniques for extraction of fats exist (including supercritical fluid extraction, assisted solvent extraction, and liquid-liquid extraction), many of the methods published by governmental regulatory agencies such as FDA, USDA, and EPA require the use of Soxhlet extraction. You present this information to the team, and since you are presently interested only in total fat content, a decision is made to use Soxhlet extraction for the lipid analysis.

Materials
Per group
- organic solvent
- sodium sulfate
- thimble (cellulose or slitted glass)
- french fry sample
- Teflon® boiling chips or glass beads (see instructor)
- glass wool
- nitrogen gas
- mortar and pestle
- micro-Soxhlet glassware with 25-mL round-bottomed flask
- heating mantle
- concentrator (evaporation) apparatus
- rubber tubing
- analytical balance

Safety, Handling, and Disposal
It is your responsibility to specifically follow your institution's standard operating procedures (SOPs) and all local, state, and national guidelines on safe handling and storage of all chemicals and equipment you may use in this activity. This includes determining and using the appropriate personal protective equipment (e.g., goggles, gloves, apron). If you are at any time unsure about an SOP or other regulation, check with your instructor.

This experiment uses hazardous solvents that must be disposed of in appropriate waste containers. Organic solvents tend to be highly flammable. Use appropriate precautions. **Extraction and post-extraction evaporation steps must be performed in a vented hood!**

Procedure

1. Obtain a sample of 10–15 french fries from a package. Weigh the sample on an analytical balance. Mash and grind the sample using a mortar and pestle until the sample is as homogenous as possible. Mix the crushed fry samples with sodium sulfate in a 1:1 (w/w) ratio. Once the sample is thoroughly mixed, place 2.0 g of the sample in a glass or cellulose thimble, filling it two-thirds to three-fourths full. Tap down and place a plug of glass wool on top of the sample to prevent spillage.
   Caution: Extraction and post-extraction evaporation steps must be performed in a vented hood! The micro-Soxhlet apparatus is expensive and very fragile and should be handled with care to prevent breakage.

2. Pour approximately 10 mL of solvent into a 25-mL round-bottomed flask. Add 3–5 boiling chips to the flask. Place the Soxhlet extractor connection on top of the flask and place the thimble with the sample in the extractor fitting. (See Figure 1.) Pour an additional 5–7 mL of solvent into the thimble compartment. Connect the Allihn condenser on top of the Soxhlet extractor and turn on the water flow. It is advisable to flow the water in from the bottom outlet and out from the top outlet of the condenser. A minimum of three samples should be extracted per student.

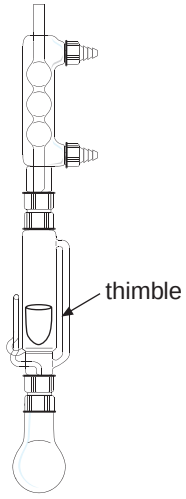

Figure 1: Soxhlet Extractor

3. Begin heating the round-bottomed flask with a mantle. Monitor the heating process. The solvent should be brought to the vaporization stage but not to a rolling boil. Ideally, solvent flushes through the sample should occur every 5–10 minutes. Adjust heat as necessary to achieve regular flushing. Allow the extraction to continue approximately 18–20 hours.

4. Empty the round-bottomed flask contents into a pre-weighed Erlenmeyer flask. Place the flask in the heated water bath of the concentrator apparatus. Remove the solvent by flowing a steady stream of nitrogen over the sample. Once all the solvent has been removed, dry the excess water from the outside of the flask and weigh the extracted fat. Then calculate the weight of fat extracted from the original french fry sample.

   Fat content for commercial french fry products is reported as follows: The difference between the weight of the flask before addition of the sample and after concentration of the sample, multiplied by 100% and divided by the original weight of the sample, gives you the percentage of fat from the sample. This calculation assumes 100% extraction of fat from the sample.

5. Turn in a formal write-up of your findings including a diagram of the apparatus and a discussion of fat content for different brands of fries.

Questions

1. What role does sodium sulfate play in this experiment?

2. Why is a cellulose thimble used to hold the sample?

3. What makes the solvent used for the extraction a good choice? Suggest an alternate choice.

INSTRUCTOR NOTES
Soxhlet Extraction of Fat from French Fries

Time Required
This activity will take 2–3 lab periods. (Take special care to turn off heating mantles and cover the tops of condensers 18–20 hours after the first lab.) The extractors can sit until the next lab period, or the units may be disassembled and the round-bottomed flasks may be removed, covered, and stored separately.

Group Size
Availability of glassware and hood space will probably determine group size. It is advisable to put students in teams of 2–3 and have them run multiple extractions. Each team may be assigned one brand of french fry to prepare and analyze according to the procedure in the Student Handout.

Materials
Per group
- solvent (see Procedural Tips and Suggestions)
- sodium sulfate
- thimble (cellulose or slitted glass)
- french fry sample
- Teflon boiling chips or glass beads
- glass wool
- nitrogen gas
- mortar and pestle
- micro-Soxhlet glassware with 25-mL round-bottomed flask
- heating mantle
- concentrator (evaporation) apparatus
- rubber tubing for water flow to Allihn condenser
- analytical balance

Safety, Handling, and Disposal
As the instructor, you are expected to provide students with access to SOPs, MSDSs, and other resources they need to safely work in the laboratory while meeting all regulatory requirements. Before doing this activity or activities from other sources, you should regularly review special handling issues with students, allow time for questions, and then assess student understanding of these issues.

Organic solvents should be collected in a waste container and disposed of properly. All extraction and drying-down procedures should be performed in the hood.

Points to Cover in the Pre-lab Discussion
It is advisable to divide the class into groups that can perform the analysis on one type of product (i.e., Group 1 performs extraction on Brand Y, Group 2 works with Brand X, etc.). Once the experiments are completed, it is effective to collect the data from each group, present it on the board, and have the students critically evaluate the data as a whole. Statistical manipulation of the data can be performed and conclusions drawn if a fairly large number of samples per product are extracted.

Procedural Tips and Suggestions

- Soxhlet extraction can be used to determine the percent-by-weight of fat in french fries. However, lipids cannot be effectively extracted with ethyl ether from moist foods because the solvent cannot easily penetrate the moist food tissues. The ether, which is hygroscopic, becomes saturated with water and inefficient for lipid extraction. Drying the food at elevated temperatures is undesirable because some lipids become bound to proteins and carbohydrates, and bound lipids are not easily extracted with organic solvents. It is necessary to add sodium sulfate or some other drying agent to the sample. This agent will be unaffected by the organic solvent but will scavenge excess water from the sample.

- Sodium sulfate is a common chemical used to absorb excess water in Soxhlet extractions. By mixing the french fry sample in a 1:1 ratio (w/w), you will eliminate the aqueous layer in the solvent/lipid mixture at the end of the extraction. However, if you choose a solvent that is miscible with water, the water interference will be more subtle in that it will take much longer to blow down the solvent, and the water, if not completely removed, will cause erroneous total fat values.

- *Evaporation:* Some evaporation of the solvent has been observed over the 20-hour period due to poorly sealed joints between the flask and extractor. Care should be taken to ensure a good seal; however, stopcock grease is not recommended as it may contaminate the extracted fat. Instead use Teflon tape wrapped around the joint or a Teflon fitting, which may be purchased from companies such as ACE Glass.

- *Solvents:* Common solvents that are immiscible with and less dense than water include diethyl ether, toluene, and hexane. Those that are immiscible with but more dense than water include chloroform, dichloromethane, and carbon tetrachloride. Some of these solvents are rather hazardous, and it is recommended that dichloromethane, hexane, or petroleum ether be used instead of others due to the temperature, safety, and extraction capabilities with fats. Heptane may be substituted. Petroleum ether is the low boiling point fraction of petroleum and is composed mainly of pentane and hexane. It has a boiling point of 35–38°C and is more hydrophobic than ethyl ether. Additionally, it is selective for more hydrophobic lipids, cheaper, less hygroscopic, and less flammable than ethyl ether.

- *Concentrating apparatus:* Several companies, such as Zymark Corporation, sell a concentrating apparatus. However, such an apparatus is not necessary to perform this experiment. A rotovap onto which the round-bottomed flask is directly attached may be used. Alternatively, a piece of tubing may be attached to a nitrogen source at one end and to a disposable glass pipette at the other, and individual samples may be evaporated down manually, either in a heated water bath or at room temperature. The more volatile the solvent used, the more rapid the drying process.

- *Treatment of drying agent:* Sodium sulfate should be dried in the oven at approximately 150°C for several hours to drive off absorbed moisture prior to use in this experiment.

- *Size of extractor:* If a larger-sized Soxhlet extractor is used, the volume of solvent and the sample amount should be scaled up accordingly.

- *Boiling chips:* Boiling stones tend to absorb lipid into their pores and become yellow, causing lipid losses. Use Teflon boiling chips or glass beads in place of the stones.

Sample Results

Average percentage of fat by weight in french fries:

- brand X: 12.02% ± 3.5%
- brand Y: 10.42% ± 2.0%

Plausible Answers to Questions

1. What role does sodium sulfate play in this experiment?
   Na_2SO_4 is a water scavenger. The fries, like most foods, contain a certain percentage of water. Removal is necessary either prior to or, in this case, during the extraction to prevent mixture of immiscible solvents or lengthy drying time of final product.

2. Why is a cellulose thimble used to hold the sample?
   A thimble acts as a filter to prevent loose sample from clogging the extraction system and entering the round-bottomed flask, which serves as a collection container for the final product. Further, the holder allows for complete saturation of the sample with the solvent, thus giving an exhaustive extraction. Also some materials tend to float, so the thimble packed on top with the wool prevents particles of sample from straying.

3. What makes the solvent used for the extraction a good choice? Suggest an alternate choice.
   In keeping with the saying "like dissolves like," the choice of solvent used is dictated by the end product desired. In the case of lipids, which are typically nonpolar, a nonpolar solvent will be a better choice for extraction than a polar solvent.

Extensions and Variations

- Soxhlet is a workhorse extraction method. One could conceivably remove the fat from almost any food product, including peanut butter, pet food kibble, fish fillets, etc. French fries from various sources may be used. Additionally one could compare other potato products, such as hash browns or chips. Other fried foods such as onion rings might also be explored.

- One could apply this general exercise to oil-stained cloth or other material in a forensics-type scenario.

- Compare and contrast Soxhlet extraction with supercritical fluid extraction (SFE) and/or with assisted solvent extraction (ASE).

References

Nielsen, S. *Introduction to the Chemical Analysis of Foods;* Jones and Bartlett: Boston, 1994; pp 183–191.

AOAC. *Official Methods of Analysis (Method 945.16),* 15th ed.; Association of Official Analytical Chemists: Washington, DC, 1990.