Case Study #4: Characterization of Edible Oils, Butters and Margarines by Fourier Transform Infrared Spectroscopy with Attenuated Total Reflectance

Intro

- We consume oil, butter and margarine on a daily basis, but do we actually know what is inside each of these products? How do they differ in terms of fat and water content?
- In a paper by Safar et al., the authors characterized various types of these lipid-rich foods using Fourier Transform Infrared Spectroscopy (FTIR) to determine their chemical composition.

Part A

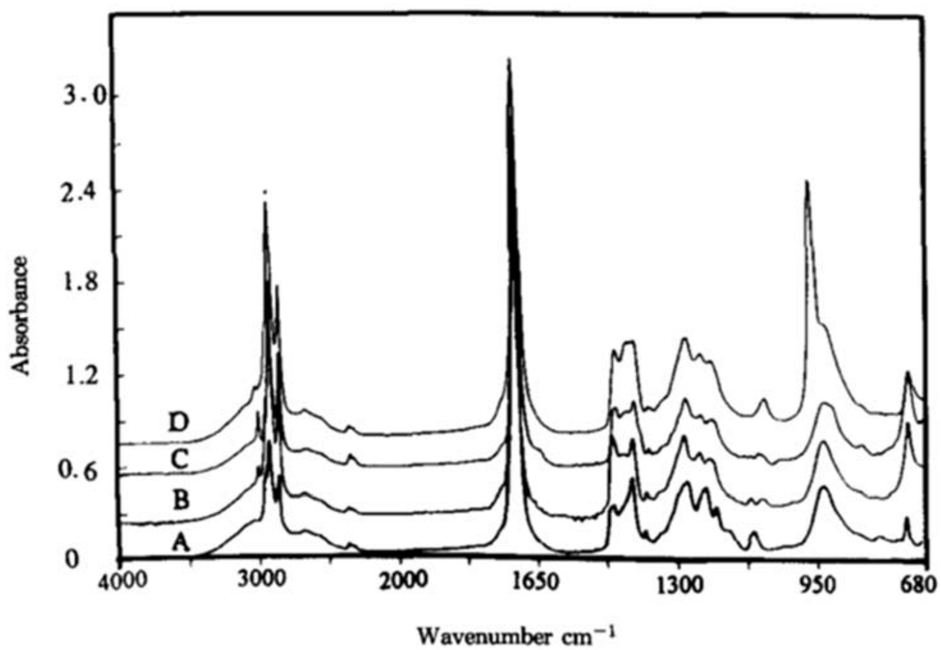
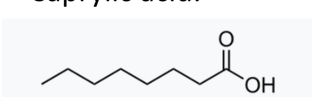
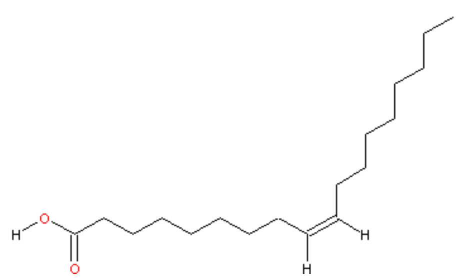


Figure 1. Infrared spectra of A. caprylic acid; B. oleic acid; C. linoleic acid; D. linolelaidic acid[1].

Caprylic acid:

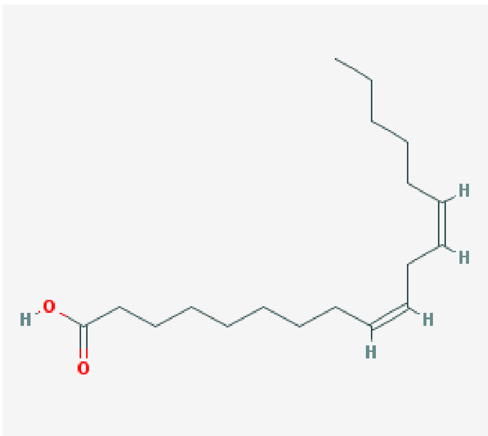


Oleic acid:

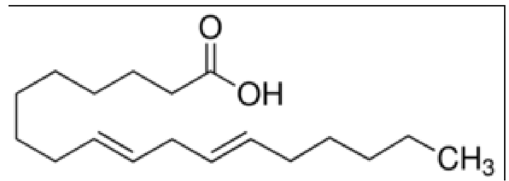


[1] 1. Safar, M., Bertrand, D., Roberta, P., Devaux, M. F. & Genot, C. Characterization of Edible Oils, Butters and Margarines by Fourier Transform Infrared Spectroscopy with Attenuated Total Reflectance. *J. Am. Oil Chem. Soc.* **71,** 371–377 (1994).

Linoleic acid:



Linoleaidic acid:



Characteristic Attenuated Total Reflectance Infrared Absorption Band of Oil

Wavenumber[a]	Group	Type of vibration	Remarks
3450*m	O-H	str.	intermolecular bonded (water)
3005 w	C-H	sym. str.	-CH=CH-(*cis* olefin)
2953 m	C-H	asym. str.	aliphatic (-CH_3)
2922 s	C-H	asym. str.	aliphatic (-CH_2)
2853 s	C-H	sym. str.	aliphatic (-CH_2)
1743 s	C=O	str.	ν(C=O) ester
1640*m	O-H	def.	δ(O-H) water
1462 m	C-H	scissoring	aliphatic (-CH_2)
1377 w	C-H	sym. def.	aliphatic (-CH_3)
1238 m	C-H	out-of-plane bend	aliphatic (-CH_2)
1162 s	C-O	str.	ν(C-O) ester
1025**vw	C-O-C	str.	ν(C-O-C) ester
966 vw	C-H	out-of-plane bend	*trans* (-CH=CH-)
722 m	C-H	rocking	aliphatic (-CH_2)

[a]Abbreviations: s, strong; m, medium; w, weak; vw, very weak; * water; ** ν(C-O-C) of unsalted butter and salted butter, asym., asymmetrical; def., deformation; str., stretching; sym., symmetrical.

Table 1. Absorption band assignments of infrared spectra

Discuss with your group:

1. Look at the unit on the x-axis. That unit is used for wavenumbers. Can you convert the values to wavelength (e.g., nm)?
2. How do the nm values compare with what you saw previously in UV-vis and AA/AE spectra?
3. By comparing Figure 1 and Table 1, what do the peaks in Figure 1 correspond to?
4. What is the main difference in the peaks in Figure 1 as the degree of unsaturation of the fatty acids increase? Does this make sense? Why?

Part B

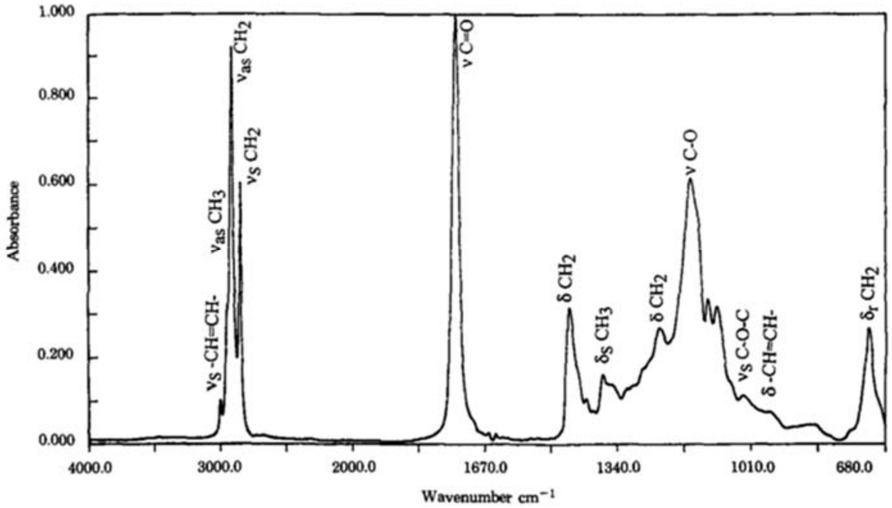


Figure 2. Infrared absorption spectrum of Olive Oil.

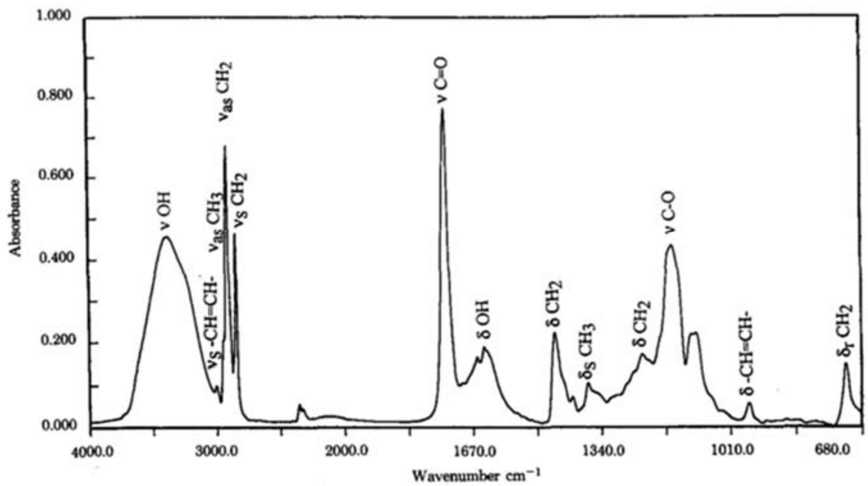


Figure 3. Infrared Spectrum of Margarine.

1. Refer to Figure 2 and 3 to answer the questions below:
    a. Which bond has a higher frequency: $_{\nu}$C-O or $_{\nu}$CH? Can you guess why?
    b. Which bond has a higher frequency: $_{\nu}$C-O or $_{\nu}$C=O? Can you guess why?
2. What are the main differences in the spectra of olive oil and margarine?
3. Based on what you know of the chemical composition of olive oil and margarine, make your best guess for why we see these differences.
4. Based on what you just saw in Part A and Part B, discuss advantages and disadvantages of using FTIR to determine adulteration of oils.

Part C

Answer these questions at home:

A company is producing extra-virgin olive oil, but there have been reports that the company has been mixing peanut oil with the olive oil to cut costs. You have been hired as an investigator to find out if this is true.

1. Design an experiment to see if there has been adulteration of the olive oil, and to what percentage. List in details the steps of your experiment.
2. Provide sample IR spectra for what you would expect to see for pure olive oil, as well as adulterated samples with the peanut oil.

Part D:

After you have discussed Part C with your team and the class, list three things you have learned through this case study.

1. ____

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2. ____

   ____

3. ____

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