GC/MS Analysis of Butter, Butter Substitutes, & Cooking Oil

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Objective

To determine the concentration of oleic acid and linoleic acid in samples of vegetable oil, peanut oil, butter, and I Can't Believe It's Not Butter!® butter substitute using GC/MS.

Introduction

In human diets, fats and oils are a part of the human diet. These fats and oils consists of saturated and unsaturated fats, where saturated fats contain only C-C single bonds and unsaturated fats have at least one C=C double bond. Due to the structures of the fats, unsaturated fats are generally in liquid form while saturated fats are solid. Generally speaking, it is thought that unsaturated fats are healthier than saturated fats. Consequently, it is advised that using liquid oils are a better substitute for cooking than solid fats, such as butter. In butter, butter substitutes, and oils, oleic acid and linoleic acid are two of the common unsaturated fats found.

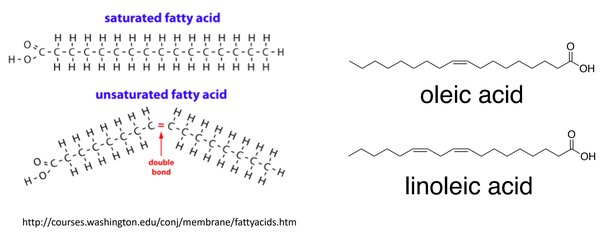

Figure 1. Side by side comparison of saturated and unsaturated fatty acid (on left) and chemical structures of oleic acid and linoleic acid (on right)

The main interest in this experiment is to determine the concentration of oleic acid in I Can't Believe It's Not Butter!® butter substitute, since out of all the samples that are analyzed, the butter substitute does not contain any accessible information on the chemical composition.

The hypothesis is that as a substitute made from vegetable oils, there would be higher concentrations of the unsaturated fatty acids than butter. However, due to its butter-like properties, it is expected to have lower concentrations of unsaturated fatty acids than the other cooking oils.

Methods

- Transesterification of the samples to prepare for GC/MS sampling using excess methanol and potassium hydroxide
- Phases were separated and washed to obtain ester product
- Esters were dissolved in n-heptane to prepare for injection into GC/MS
- Use similarity search to determine the identities of the peaks that were analyzed

Results & Discussion

- Methyl oleate has a molecular mass of 296.4879 g/mol and a molecular formula of $C_{19}H_{36}O_2$, meaning that the m/z ratio of all single-charged mass spectra peaks should be no higher than 296.
- The gas chromatogram of all four samples had absolutely no traces of methyl oleate.
- The spectra for vegetable oil, peanut oil, and I Can't Believe It's Not Butter butter substitute look nearly identical with each other. These three chromatograms all had a methanol peak at 1.860 min, various hydrocarbon peaks from 4.5 min to 5 min, and cyclohexane peak at 5.3 min as shown in Figure 2.
- No methyl oleate peak at all.

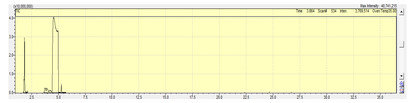

Figure 2. Gas chromatogram of peanut oil. Vegetable oil and I Can't Believe It's Not Butter butter substitute both look identical to this chromatogram.

- Land O'Lakes butter chromatogram looked different
- It had three peaks at 1.805 min, 2.025 min, and 2.240 min that were identified as water, a heptane peak at 4.550 min, and various cyclosiloxane peaks from 10.955 min to 32.210 min.
- Similarity search identified peaks at 10.955 min and 16.875 min as cyclotrisiloxane and cyclotetrasiloxane, respectively. This trend continues until the cyclodecasiloxane peak at 32.210 min..
- Still no methyl oleate peak identified by the instrument (**Figure 3**).

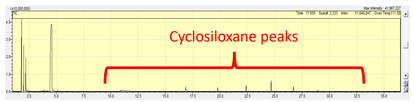

Figure 3. Gas chromatogram of Land O'Lakes butter.

- Peak for methyl oleate should show up at around 16.875 min, which is the same time as the cyclotetrasiloxane peak. This is because cyclotetrasiloxane has a molecular mass of 296.616 g/mol, which is very close to methyl oleate's molecular mass of 296.4879 g/mol.
- Mass spectrum of methyl oleate, however, does not resemble the expected mass spectrum of cyclotetrasiloxane.
- Mass spectrum of methyl oleate contains many more peaks than the spectrum of cyclotetrasiloxane, and it has a few characteristic peaks that identify it as methyl oleate.
- There is one peak at m/z = 296. This would make sense since the molar mass of methyl oleate is 296 g/mol.
- There is a peak at m/z = 264. This peak is a result in the loss of a methoxy group and one hydrogen.[7] The methoxy group has a mass of 31, the rest of the molecule has a mass of 264, and the hydrogen has a mass of 1.[7] All of this adds to 296, which is the mass of the methyl oleate. This proves that there is a methoxy group in methyl oleate. The fragments of this peak is displayed in Figure 4.

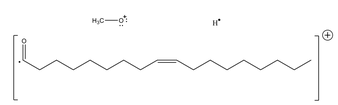

Figure 4. Fragmentation pattern of m/z = 264.

- There is a peak at m/z = 74. This peak corresponds to a fragment called the McLafferty ion, an ion that is used to identify most ester derivatives of fatty acids.
- Fragmentation of the McLafferty ion is shown in Figure 5.

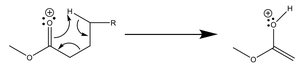

Figure 5. The McLafferty ion

- The three peaks are sufficient to show that the mass spectrum contains methyl oleate (Figure 6).

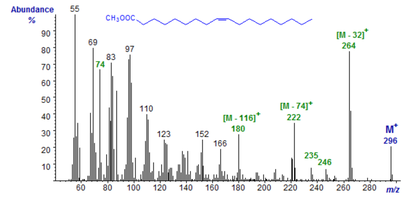

Figure 6 Expected Methyl Oleate Mass Spectrum

- Experiment did not yield expected results.
- Main explanation is that layer separation did not occur during transesterification process in order to prepare the samples for analysis
- Since experiment was adapted from the Perkin Elmer procedure, some changes made may have not been appropriate for the experiments.
- It was originally thought that the scale that Perkin Elmer executed the experiment was unnecessarily large, therefore everything was scaled down.
- It could be possible that the step mentioned above is more scale dependent than previously thought.

Conclusions

- The experiment did not go as planned, but there are still many things to be learned from this experiment.
- Most of the samples found in everyday life are complex, and impure and require processes in order to process for analysis.
- The experiment provided additional experience in using the GC/MS instrument.
- Possible future direction to take include making adjustments to Perkin Elmer procedure until separation is visible

Literature Cited

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