SPECTRA OF THE MONTH

FLOUR VARIETY ANALYSIS WITH NIR ABSORBANCE SPECTROSCOPY

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INTRO

BACKGROUND OF APPLICATION

Flour is a staple ingredient in cuisines worldwide. While typically derived from wheat, alternative flours like rice, chickpea, and almond have gained popularity, particularly due to the rise in gluten sensitivities and autoimmune disorders like celiac disease, which are estimated to be nine times more prevalent than 50 years ago. For individuals with such conditions, consuming gluten-free products is critical. However, some alternative flours advertised as gluten-free may be contaminated with wheat during production. To ensure purity, near-infrared (NIR) absorbance spectroscopy can identify contaminants. This method leverages the well-characterized properties of flour components—such as fats, carbohydrates, sugar, fiber, and protein—to detect and quantify gluten content effectively.

This experiment aims to measure differences in flour samples using NIR absorbance spectroscopy. Absorbance peaks in the NIR region include those associated with fiber, starch, lipids, and protein. Five flour varieties were used, including wheat flour, almond flour, coconut flour, chickpea flour, and rice flour (Figure 1). These samples were specifically chosen due to their variation in fiber, carbohydrate, fat, and protein content. Other differences in nutritional content of the samples include concentrations of elements such as calcium, potassium, and sodium.

FIGURE #1 Flour samples used for this experiment (from left to right: wheat flour, almond flour, coconut flour, chickpea flour, and rice flour).

DESCRIPTION OF SPECTROSCOPY SETUP

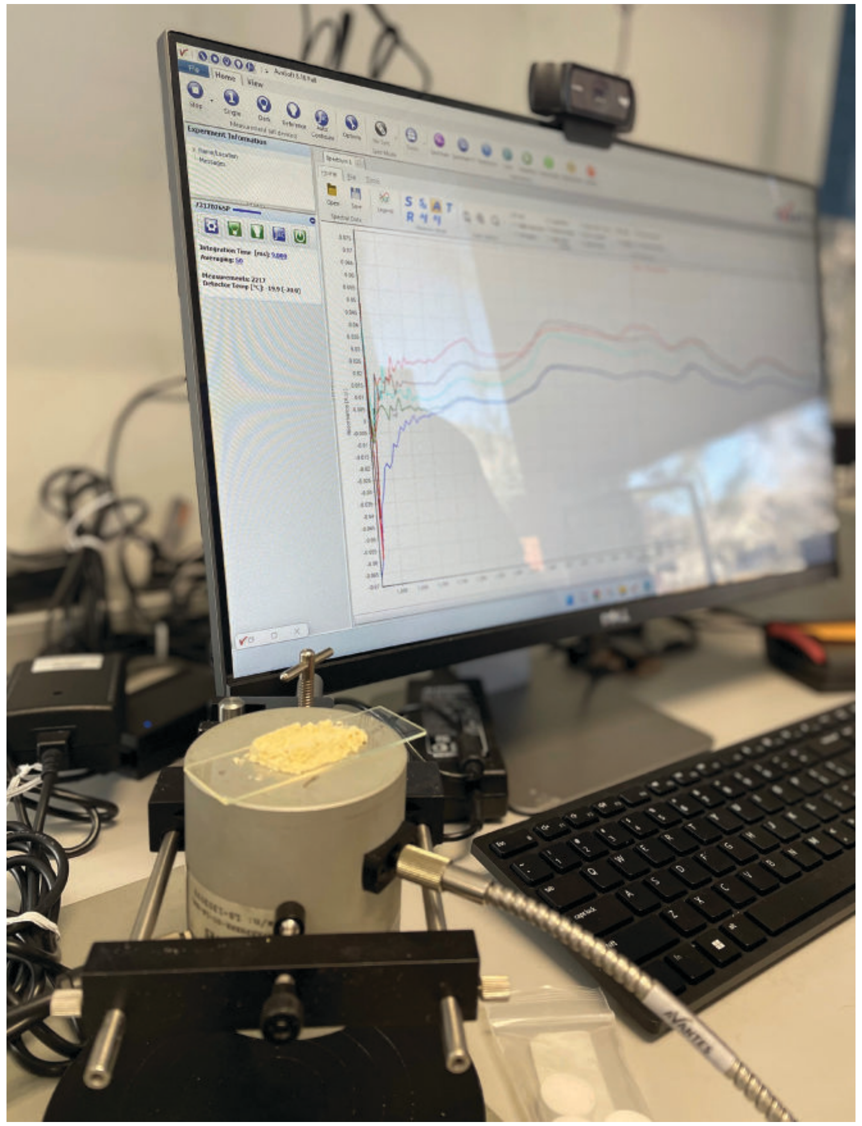
The setup for this experiment (Figure 2) utilized our AvaSpec-NIR256-2.5-HSC-EVO spectrometer. Specifically for measurements in the NIR range up to 2.5 µm, this model pairs our high-sensitivity optical bench with next generation electronics for exceptional performance, including 0.54 ms/scan sample speed and integration times as fast as 10 µs. The AvaSpec-NIR256-2.5-HSC-EVO is equipped with our trusted InGaAs (Indium-Gallium-Arsenide) array detector, a thermally electric-cooled optical bench, and our ultra-low-noise electronics board with both USB3.0 and Giga-Ethernet connection ports onboard. Additional features include multiple grating and replaceable slit options, as well as digital and analog I/O ports, which can be used to control the shutter or pulse of connected light sources and the gain setting of the spectrometer, with either High Sensitivity or Low Noise. The instrument used in this experiment had a wavelength range of 1000-2500 nm and a 50-micron slit installed.

FIGURE #2: setup for flour measurements: amples were placed on glass slides over the integrating sphere port, connected to the NIR spectrometer via a fiber optic cable. A WS-2 reference tile served as the reference, and a BS-2 tile (not pictured) was used for the dark measurement

The light source used for the absorbance measurements was a built-in halogen light in our AvaSphere-50-LS-HAL-12V integrating sphere. While specifically designed for reflection applications, this integrating sphere is also useful for low reflecting materials and NIR spectral measurements, where signal strength can be limited. The built-in halogen light source provides diffused halogen light on the sample without the losses associated with fiber-optic coupling, with up to 160 times more light on the sample compared to our standard reflectance integrating sphere. The AvaSphere-50-LS-HAL-12V has an internal diameter of 50 mm, a 10 mm sample port, an SMA-terminated reference port, and a direct collimated SMA-port for collection of signal with any of our AvaSpec spectrometers.

Other accessories used for this experiment included a 600-micron core fiber optic cable (FC-UVIR600-1-BX) to connect the integrating sphere to the NIR spectrometer, a WS-2 white reference tile, a BS-2 black reference tile, and a mounting stage to hold the integrating sphere in place for the absorbance measurements.

DESCRIPTION OF METHODOLOGY



The flour samples used for this experiment were wheat flour, almond flour, coconut flour, chickpea flour, and rice flour, all purchased from a local grocery store. For the absorbance measurements, dark and reference spectra were taken using our BS-2 black reference tile and WS-2 white reference tile, respectively, with the internal halogen light of the integrating sphere turned on. A small pile of each flour sample was then individually placed on a glass slide, which was then placed over the opening port of the integrating sphere and measured. No additional preparation was done for the samples, such as sifting, besides slightly pressing the samples onto the glass slides to minimize the amount of air gaps in the samples.

For data analysis, we used Absorbance mode in AvaSoft, our custom software package. As the name suggests, Absorbance mode is designed for absorbance applications, where the reference measurement will report 0 A.U. (absorbance units) and 5 A.U. for the dark measurement. In this experiment, the WS-2 white reference tile was used as the reference and the BS-2 black reference tile was used as the dark. While the dark measurement is often just a measurement with the light source disabled, this method created too significant of a difference for the absorbance measurements where all the flour measurements reported below 0.05 A.U. By using the BS-2 as the dark measurement, the absorbance spectra for the flour samples were converted to a much more useable A.U. range. We used an integration time of 9 ms, which can be adjusted to increase or decrease the amount of light being measured at one time and affects the overall magnitude of the reported spectrum. We set averaging to 50, meaning 50 values were averaged together to provide more consistent spectra results.

TEST DATA AND RESULTS

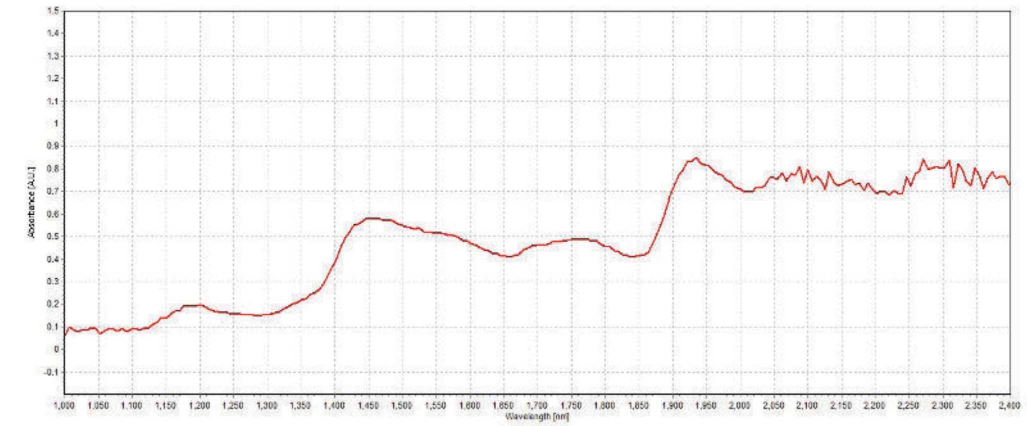


FIGURE #:3 Absorbance spectrum of wheat flour sample.

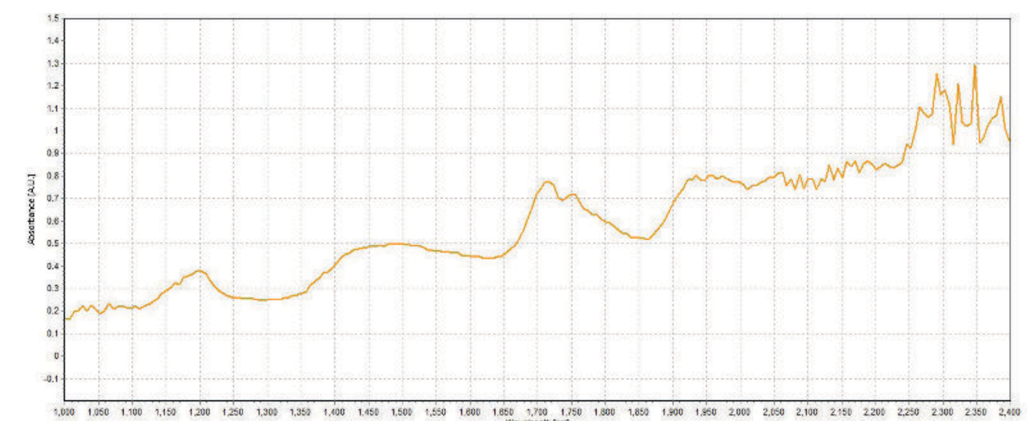


FIGURE #4: Absorbance spectrum of almond flour sample.

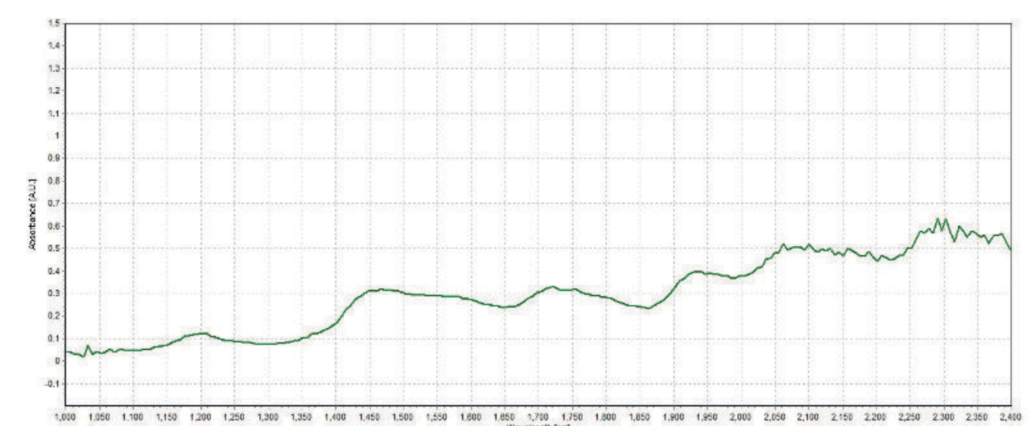


FIGURE #5: Absorbance spectrum of coconut flour sample.

TEST DATA AND RESULTS

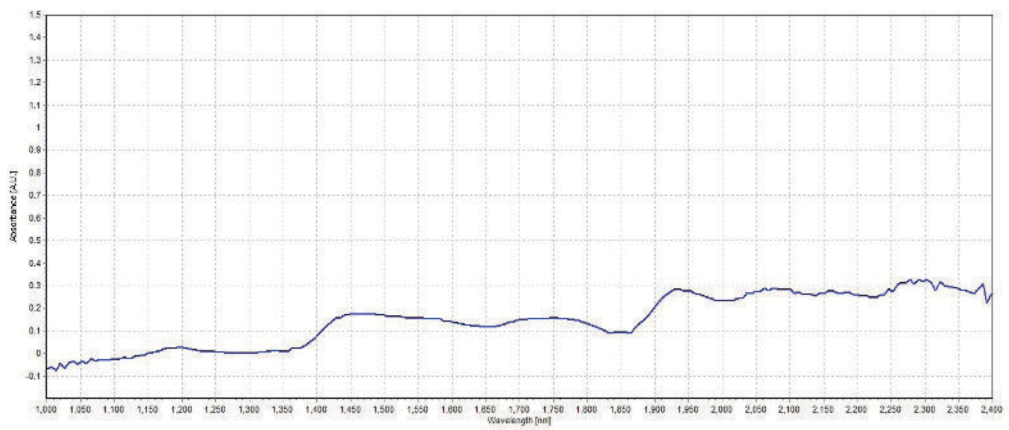


FIGURE #:6 Absorbance spectrum of chickpea flour sample.

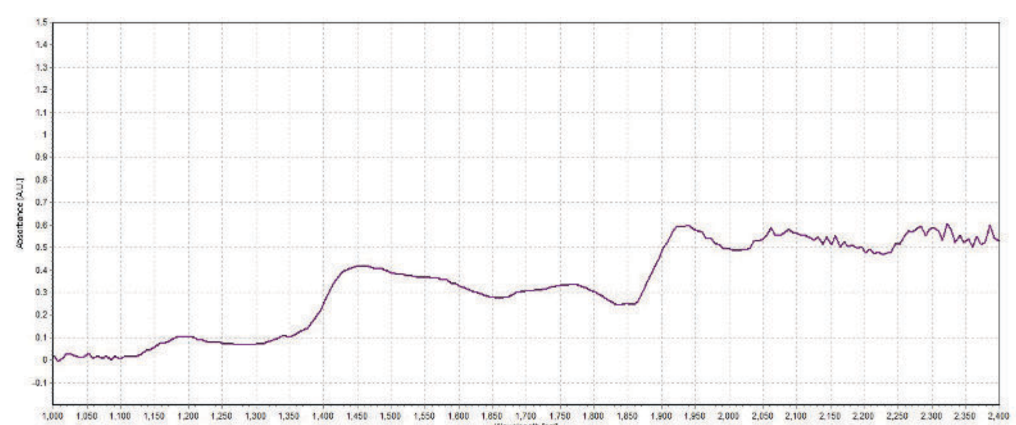


FIGURE #7: Absorbance spectrum of rice flour sample.

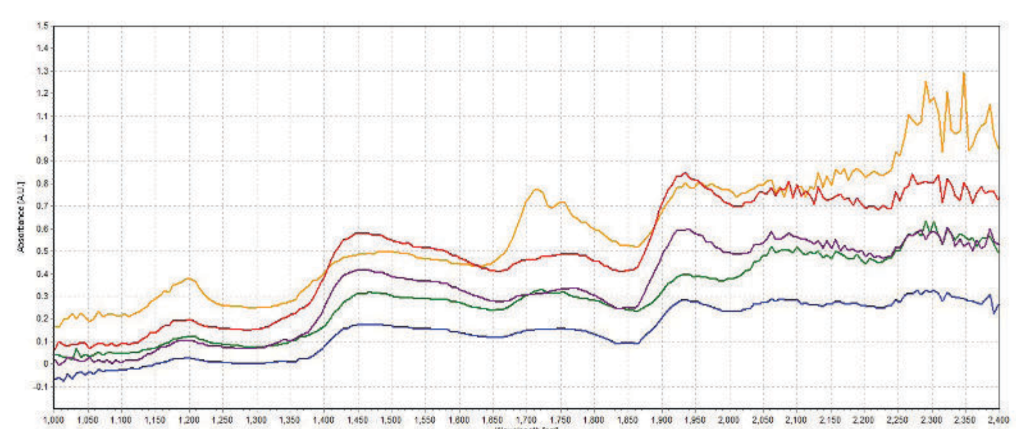


FIGURE #8: Absorbance spectra of wheat flour sample (red), almond flour sample (orange), coconut flour sample (green), chickpea flour sample (blue), and rice flour sample (purple), shown together for comparison.

ANALYSIS

All five flour samples showed similar absorbance peaks throughout the NIR spectrum. Absorbance peaks seen at around 1200 nm, 1450 nm, 1930 nm, and 2300 nm can be correlated to carbohydrate/starch content (Figures 3-7). The 1200 nm peak could be associated with the presence of sugar or fiber, as both have reported peaks in this range. The 1450 nm, 1930 nm, and 2300 nm peaks can be linked to O-H overtones, O-H deformation, and C-H bonds, respectively, in various starch molecules such as sucrose, glucose, and fructose. Known absorbance peaks for fats/lipids are 1725 nm and 2310 nm, both of which seems clearly present in the almond flour spectrum. Reported NIR absorbance peaks for protein have a range of 2150-2200 nm, though we did not observe peaks of much significance in this range.

With the main peaks identified, we can compare each flour sample to see if the peaks correlate to differences in nutritional content. Below is a table containing the nutritional information for each flour sample based on the package labels. While all labels reported nutritional values in terms of volume, the mass between them is not consistent due to varying fineness of the flour. Because of this, each serving size was adjusted to a consistent 40g serving, and the nutritional values were adjusted accordingly.

TABLE #1: Nutritional value information for flour samples. All values based on 40g serving size.

Flour	Wheat	Almond	Coconut	Chickpea	Rice
Fat/Lipids (g)	1	20	5	2	0
Carbs (g)	29	10	30	28	34
Fiber (g)	1	7	17	7	0
Sugar (g)	0	3	10	1	0
Protein (g)	5	13	10	7	2

The peak around 1200 nm has been correlated to both sugar and fiber. While almond has the clear most intense peak here, it does not have the highest content of either sugar or fiber. The peaks at 1450 nm, 1930 nm, and 2300 nm have strong correlations to carbohydrate/starch content, so it is no surprise that the wheat and almond samples had the two highest intensities for those three peaks. The chickpea flour had surprisingly low magnitudes at these peaks, which may suggest that the carbohydrate composition between this flour type varies compared to the other two. The peak around 2300 nm is also linked to fat/lipid content, which may explain why this area has a significantly high magnitude for the almond flour sample compared to the other flour samples. The two significant peaks in the almond flour spectrum between 1700-1750 nm are also likely indicative of the significantly higher fat/lipid content in this flour type compared to the other. Interestingly, these same peaks are seen at a much smaller magnitude in the coconut flour sample, which has the second-highest total fat content. In addition to the main peaks mentioned, a few minor absorbance peaks were also observed. The rice flour sample had a peak around 1337 nm, which has been reported as a means of classifying rice flour. A minor absorbance peak also seemed to be present around 1363 nm in the wheat flour, coconut flour, and chickpea flour samples, though there is no clear indication for what this could be demonstrating. A plot of all five spectra is also provided to illustrate the differences in peak intensity (Figure 8).

ANALYSIS

The results of the measurements were somewhat limited in their ability to directly correlate peak magnitudes to nutritional values, but further post-processing models can be implemented to apply more complex analysis. These peaks are also only identified to contents such as lipids or starches. Correlating these values to gluten content would also require more complex tools such as a chemometric model. Besides the discussed nutritional values, we hoped to detect some elemental characteristics in the samples as well, such as calcium, potassium, and sodium, but the absorbance for these elements is typically in the UV and lower visible range. Absorbance measurements were performed in these regions using our Nexos spectrometer and an AvaLight-DH-S, though no significant results were derived. Because of this, these results were excluded. Better results for this region may have been obtainable using a 45-degree reflection probe immersed in the flour, though this probe type was not readily available at the time of the measurements. A similar type of measurement was performed previously by our team a few years ago (https://avantesusa.com/spectra-near-infrared-absorption-reflection/).

CONCLUSION

In conclusion, the present experiment highlights the use of NIR absorbance measurements to determine components in different flour samples as well as perform some minor direct comparisons. More in-depth comparisons would require more complex post-processing such as a chemometric model. Despite this, the measured peaks still correlated well with previously reported absorbance peaks for contents such as fats/lipids and carbohydrates/starches. The AvaSpec-NIR256-2.5-HSC-EVO is a highly versatile NIR spectrometer with plenty of available options to match the bandwidth and requirements fitting your application. The AvaSphere-50-LS-HAL-12V is specifically designed for reflectance measurements but is effective for any applications where limited signal strength may be an issue, such as NIR measurements. Please contact Avantes for more information on the configuration that is best suited for your data collection.

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