Fighting food fraud – a unique approach to tackle extra virgin olive oil adulteration

Virgin and extra virgin olive oils are adulterated with cheaper vegetable oils and lower grade olive oils due to the high demand and price of high-grade olive oil. Not only is it important to authenticate olive oil to prevent fraud, it is also necessary to determine purity for the health and safety of consumers. For example, olive oil can be adulterated with hazelnut or peanut oils and this can result in major allergic risk for some consumers. A 2010 report published by the University of California Davis Olive Center found that "69 percent of imported olive oils labeled as extra virgin failed the International Olive Council's sensory standard". In other words, most imported extra virgin olive oils (EVOOs) were not extra virgin at all. This led, in 2014, to the State of California approving stricter standards on the labeling and grading of olive oil.

Photo courtesy of UC Davis.

The first center in North America dedicated to olive oil research

UC Davis launched the first university-based olive research and education center in North America in 2008, called the Olive Center. It is a self-funded university/industry coalition bringing together nearly 60 UC faculty members, research specialists, and farm advisors to address the research and education needs of California olive growers and processors. As part of the university's Robert Mondavi Institute for Wine and Food Science, the Olive Center hopes to do for olives and olive oil what UC Davis has done for grapes and wine - helping move California olive oil into the world's highest rankings.

"Traditional methods involve time-consuming extractions, excessive solvent use, and lengthy data analysis"

— Dr. Selina Wang
Research Director, UC Davis Olive Center[1]

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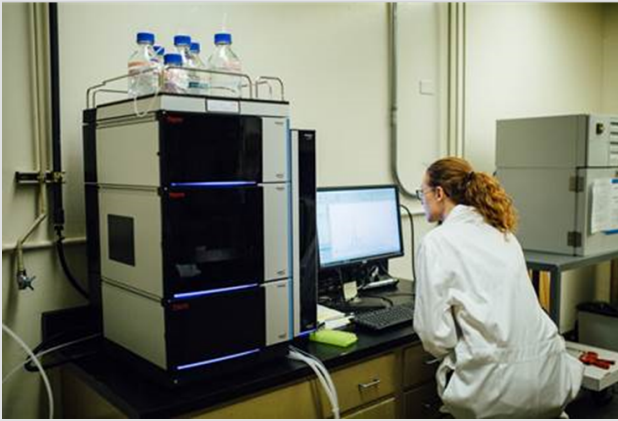
The Thermo Scientific™ Vanquish™ UHPLC System UHPLC System with Charged Aerosol Detector being operated by Hilary Green at the UC Davis Olive Center

"We were able to quickly setup the Vanquish CAD system and our method with the help of Thermo Fisher Scientific staff"

— Hilary Green

The UC Davis Olive Center's mission:

• To pursue innovation – applying modern scientific knowledge and technology to the olive oil crop.

• To provide services – offering best practices, university resources, and educational events.

• To provide independent, research-based information to professional buyers and regulators to promote consumer embrace of quality.

Thermo Fisher Scientific is proud to support the Olive Center. Their goals complement our mission to make the world healthier, cleaner, and safer.

Addressing high value edible oil adulteration
The research director, Dr. Selina Wang, her graduate student, Hilary Green, and assistant director, Xueqi Shirley Li, at the Olive Center evaluated methods capable of authenticating EVOO and identifying the type and amount of adulterant oils present. They found that traditional methods measuring either fatty acid or sterol profiles were complex, costly, and time consuming. Furthermore, these methods used excessive amounts of solvents that needed to be disposed of correctly. What Dr. Wang wanted was a routine, simple to use, and quick method that required minimal sample preparation and the elimination of excessive solvent usage.

Coincidently, Thermo Fisher Scientific application chemists were developing a straightforward, direct, and rapid method for EVOO purity by measuring an oil's triacylglycerol (TAG) profile using the Thermo Scientific™ Vanquish™ Flex UHPLC system with a Charged Aerosol Detector (CAD) and principal component analysis (PCA). Thermo Fisher Scientific staff presented a poster on the preliminary method at an American Oil Chemists Society Conference. This was of great interest to Dr. Wang. She sent samples to our analytical chemists to show feasibility of the method and the very fruitful collaboration between the Olive Center and Thermo Fisher Scientific began.

"The Vanquish CAD system is intuitive to work with and requires minimal maintenance, which gives it potential to be used by industry for in-house testing"

— Hilary Green

A Vanquish Flex UHPLC with CAD system was installed at the Olive Center and it started to generate data immediately. Hilary further developed the analytical method and used it to analyze the Olive Center's extensive library of olive oil samples.

A new, quicker and easier approach to authenticate olive oil

TAGs are the most abundant class of compounds in oil samples. Different oils have different TAG profiles. Therefore, changes in TAG patterns can be useful in determining oil purity. However, the measurement of TAGs is extremely challenging. TAGs are difficult to resolve using traditional high performance liquid chromatography (HPLC) approaches. Furthermore, they do not possess a strong chromophore so UV/Vis absorbance detection is not an option. Mass spectrometric approaches require the analyte to form gas phase ions, and mass spectrometers are expensive to purchase and maintain, and require a dedicated operator.

These analytical challenges may seem insurmountable, but they were no problem for the Vanquish UHPLC system with CAD.

- UHPLC gives improved chromatographic resolution and sample throughput.

- The CAD is a powerful universal detector capable of measuring all non-volatile and many semi-volatile species contained within the sample. CAD response is uniform and independent of chemical structure.

- The Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software offers superior instrument control, automation, and data processing.

Furthermore, sample preparation was straightforward — a simple dilution prior to analysis — no need for toxic solvents.

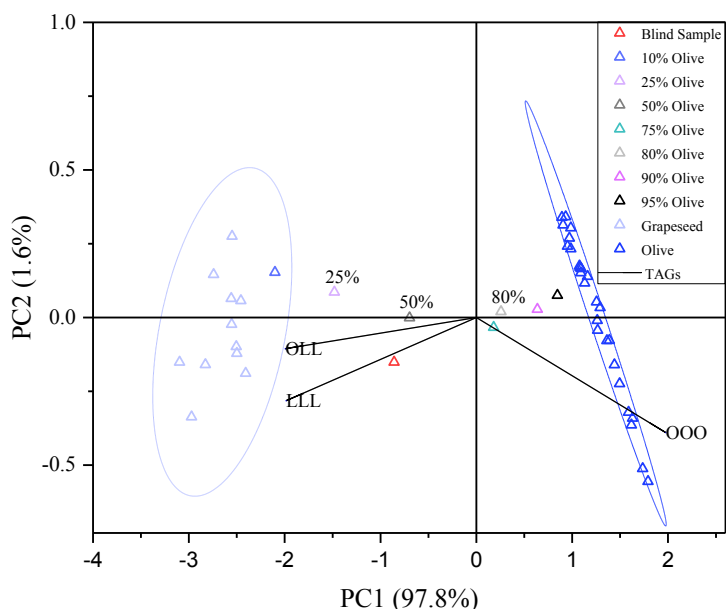### Olive oil adulteration – easily visualized with PCA

TAG profiles were determined for different olive oils, adulterant oils, and olive oil/adulterant oil blends. The TAG data were transferred from the Chromeleon CDS to a commercially available PCA software. Oil purity, the type of oil, and amount of adulteration could easily be visualized using PCA:

A typical PCA plot showing different olive oil samples, the potential adulterant grapeseed oil, and blends of these two oils. Similar samples will cluster together based on their TAG profiles. A sample of unknown composition, "Blind Sample", is also shown and was used to evaluate the performance of the method. Differences in TAG profiles can be used to determine adulteration of olive oil with as little as 5-10% vegetable oil. For greater detail see: Green et al., (2020)[1] and Application Note: AN73174.[2]

The effectiveness of the UHPLC-CAD method to identify EVOO adulteration was tested by the analysis of intentionally blended "blind samples". The method was easily able to detect adulteration of EVOO with cheaper vegetable oils and lower-quality olive oils at or below the 10% level, supporting the value of this approach for authentication of commercially available EVOO samples.

To the future

The method applying a Vanquish UHPLC System with CAD shows great great promise for routine olive oil testing. It is easier, quicker and more cost effective than traditional methods. The extensive use of toxic solvents is eliminated. The method can be readily implemented for in-house use, thus eliminating the need for companies to send samples out for testing.

About Hilary Green

Hilary Green is a second year PhD student in the Agricultural and Environmental Chemistry Graduate group. She completed her Bachelors of Science in Chemistry from the University North Carolina at Chapel Hill. Throughout undergrad, her research focused on water and air pollution, but she has always had a passion for food and agriculture. Now at UC Davis, she is excited to be working on method development for the food industry. Her current project focuses on developing a more time efficient and cost-effective way to determine the purity and quality of edible oils. She hopes to use her degree to continue research either working for industry or with the government.

The Olive Center team is currently expanding their database to include different domestic and international olive oil varietals and to study the impact of growing region and season on TAG profiles. They will be using their Vanquish UHPLC System with CAD to look at other potential markers relevant to olive oil adulteration and expand the method to include other high value edible oils.

For more information about the UC Davis Olive Center please visit https://olivecenter.ucdavis.edu/

References

1. A rapid method for the detection of extra virgin olive oil adulteration using UHPLC-CAD profiling of triacylglycerols and PCA – Green, HS; Li, X; De Pra, M; Lovejoy, KS; Steiner, F; Acworth, IN; and Wang, SC. Food Control, Volume 107, January 2020.

2. AN 73174 Determination of olive oil purity based on triacylglycerols profiling by UHPLC-CAD and Principal Component Analysis. Authors: Hilary S. Green, Hilary S., Li, Xueqi, De Pra, Mauro, Lovejoy, Katherine, Steiner, Frank, Acworth, Ian N., Wang, Selina

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