Non-targeted Determination of Olive Oil Adulterations with a Simple Dilute and Shoot Method

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Introduction

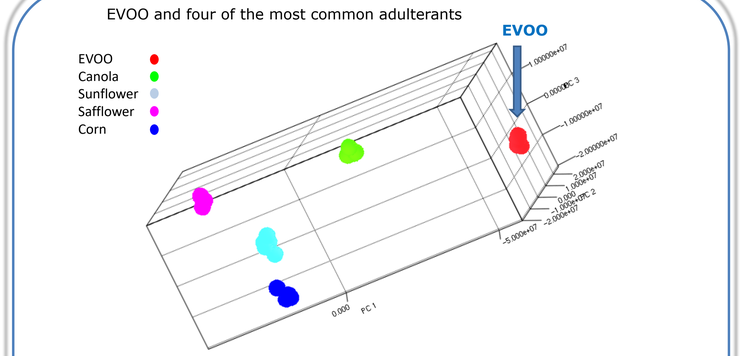
Extra virgin olive oil (EVOO) is the highest quality olive oil and its health benefits have been extensively studied. When consumed as part of a traditional Mediterranean diet, EVOO has shown to be beneficial in preventing heart- and obesity-related health issues. The health benefits together with the great taste make the demand for EVOO High and a lucrative product for adulteration.

While less than 10% of world olive oil production meets the criteria for labeling as extra-virgin, it has been estimated that up to 50% of retail oil is labeled "extra-virgin". Some oil labeled "extra-virgin" is diluted with cheaper olive oils or other vegetable oils, making the detection of such adulterations an important task.

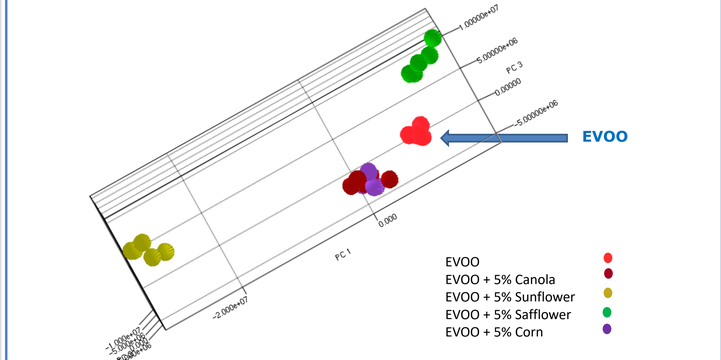
Methods

Standards of EVOO, safflower oil, corn oil, canola oil, and sunflower oil were diluted 100,000 times with acetonitrile and filtered to remove particles. 5% additions of the other edible oils to the EVOO were prepared in the same way. 5uL was injected on a C-18 column with a 24 min. gradient starting at 40% A to 100% B. A: water/CAN - 60/40, 0.1% formic acid; B: CAN/IPA - 25/75, 0.1% formic acid.

After non-targeted acquisition with Bruker Compact QTOF high resolution accurate mass instrument (Fig. 3), the data is processed by Principal Component Analysis using the MetaboScape software package to distinguish the different edible oils.



A: EVOO and other pure edible oils in a PCA plot. The oils are well separated from each other



B: EVOO and adulterated EVOO. 5% addition of other edible oils added to EVOO

Fig. 1: Principal components plots of extra virgin olive oil and other edible oils. In the top plot (A) the pure oils are plotted and in the bottom plot (b) all are EVOO but four out of the five have a 5% addition of one of the other edible oils.

On this side of the volcano plot, in the red box, are the compounds from the adulteration.

The other side of the plot has the compounds that are more characteristic for the olive oil.

The black dots in the middle are the compounds that both the adulteration and the olive oil have in common.

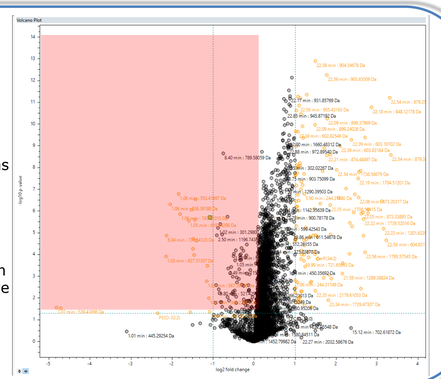


Fig. 2: The volcano plot is an easy and convenient way to identify differences between two sample sets. In this graph the dots on the left are compounds belonging to the adulterating oil, the dots on the right belong to the EVOO, and the dots in the middle are compounds that they have in common.

Fig. 3: Bruker Compact QTOF and Elute UHPLC used for the analysis

Results

After a simple 100,000 times dilution and µ-filtering of all the standards, the analysis were done with five injections of each sample, to gain statistical significance and more reliable results. Analysis of the data from different oil standards was automated by Bruker MetaboScape statistical and annotation software. The results of the Principal Component Analysis (PCA) are shown in Fig. 1A. All the different edible oils are clearly pulled apart and separated in a PCA plot.

The "adulteration" of the EVOO with only 5% of other edible oils was done before dilution and filtration. These "adulterated" EVOO samples were also injected 5 times each. Even in this more challenging case, the different oils were clearly separated by the Bruker MetaboScape statistical analysis, as can be seen in Fig. 1. B.

Once sample sets are analyzed with this non-targeted workflow, software generated volcano plots can be used to identify the specific biomarkers for each adulterant (Fig. 2). Thus, once separated and identified, these biomarkers can be used for accurate, rapid and highly automated targeted detection of specific adulterants in EVOO.

Conclusions

- We developed an easy dilute-and-shout method for detection of extra virgin olive oil adulteration with other edible oils.
- The combination of highly automated non-targeted and targeted workflows allows to achieve both high accuracy and expandability of the method, while keeping it fast and simple.

Food Chemistry

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