Information sheet on the NIR analysis of olive oil

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Sensory testing

The taste impressions “fruity”, “bitter”, and “sharp” are largely characterised by the type and quantity of phenolcarboxylic acids, phenols and polyphenols. It was therefore possible to develop a method using NIR that correlated extremely well to the data determined by the panel. However, this should not or cannot replace sensory testing.

Harmony

When comparing different olive oils it is important to quantify the quality using a single parameter. To this end, in 2011 Bongartz and Oberg developed the term “harmony” and introduced this in sensory testing to help identify the sensory quality differences in the “extra virgin” category.

The quality of an olive oil is predominantly determined by its age, type of harvesting, and the flavour profile. The objective was therefore to calculate “harmony” on the basis of NIR analysis. The various analytical parameters for the age, the oxidative changes (POV, 1,2-DG, K-values, PPP, MonoxTG) and the flavour profile are used for this calculation.

In many cases, the value given in the NIR analysis for “harmony” correlated to the value determined in sensory testing for harmony. In this way, the probability of a sensory error can also be limited, though it cannot be specifically predicted as sensory errors can only be attributed to minor components, they are not caused by polyphenols or phenolcarboxylic acids.

FFA% – free fatty acids

The free fatty acid content (FFA%) together with the proportion of 1,2-diglycerides is a key quality parameter. Encouraged by elevated temperatures, free fatty acids are released via lipase-catalysed enzymatic hydrolysis of the triglycerides during olive storage *before* pressing. High levels of FFA are therefore a sign of poor quality because the olives were stored too long. Olive oils produced from flawless olives have a very low level of free fatty acids (approx. 0.1 %) immediately after harvest and this increases after just one day of storage to approx. 0.2–0.3 %. Following pressing and removal of residual water via filtration, the FFA level increases only slightly at approx. 0.01 %/month.

70 % of all olive oils with more than 0.5 % FFA generally also have a sensory defect. The legislator has specified a limit of 0.8 % FFA for “extra virgin olive oil”, which is considered excessively high.

POV – peroxide value

Peroxide value is another analytical parameter. The POV measures the level of primary oxidation products in the oil, which can increase as a result of secondary reactions in the oil. However, it can also decrease again. As a result of their manufacture, virgin oils have a higher POV of more than 4 compared with refined oils, which can have levels of 0 meq O2/kg. Good oils generally have values of 8–12. Oils aged for more than 12 months sometimes have POVs of more than 12. A value of 15 is virtually never achieved although the legislator has even set the threshold at 20.

It is wrongly assumed that a rancid taste is related to a high peroxide value. The POV is therefore of minor importance to the assessment and is only used as supplementary information. The POV increases during storage and only correlates to the K232 value to a certain extent. Poor storage conditions include using clear glass, (sun)light and heat, which encourage fat oxidation, thereby negatively changing the values for POV, K232, PPP and 1,2 diglycerides.

K232/K270 – UV absorption

In addition to POV, UV absorption has been considered a key quality parameter for a long time due to the lack of other more suitable criteria, or lack of these included in EU regulation.

In general, the spectrophotometric behaviour of oils during storage or thermal treatment changes in the wavelength range 270–232 nm. Oxygen and light (auto- or photooxidation) catalyse changes in unsaturated fatty acids by forming hydroperoxides (K232) or forming conjugated fatty acids (K270). The K232 value correlates slightly with the peroxide value and increases with storage. In comparison, K270 values change less during storage. However, both K values are also influenced by the type of olive oil, which means both values can only be used for orientation purposes. For instance, Spanish oils often have significantly lower K values than Greek olive oils, which may also be a result of the different harvesting methods.

Elevated K270 values (over 0.22) are almost certainly an indication of the presence of refined olive oil and of the presence of olive pomace oil.

PPP – pyropheophytin

The PPP value describes the relative level of decomposition products of the green plant pigment chlorophyll. Initially, this parameter was considered to be a sensitive parameter to detect thermal treatment. Since around the year 2000, olive oils have frequently been treated in a vacuum with steam to remove the off-flavour components. Using the so-called soft column method, temperatures of just 90–120°C for a few minutes are adequate for this refining. However, PPP are already formed at temperatures below 100°C which is why this parameter was proposed to detect this type of treatment.

The analytical criteria such as trans-fatty acids, K270 or stigmastadiene proposed in EC regulation to detect thermal treatment are unsuitable because their decomposition products are only formed at considerably higher temperatures (>150°C). As often only a portion of the oil is thermally treated, this makes detecting a raffinate in a virgin oil even more difficult. Unfortunately this parameter is also influenced by the storage conditions (temperature, oxygen and light). Light in particular (light-coloured bottles) rapidly changes or destroys the green plant pigment, which can thereby simulate the refining process.

As most oils are often correctly protected from light during storage, the age of an oil can also be estimated using the PPP value. As with 1,2-DG, the PPP level permits an indicative age assessment. The PPP value generally increases 0.5 % per month and generally reaches 6–8 % after a year.

MonoxTG – monomeric oxidised triglycerides

This parameter, in a single analytical value, describes all the oxidative changes of the fats. Its correlation with the other analytical parameters is currently still being investigated.

1,2-DG – 1,2-diglycerides

Together with the free fatty acids (FFA %), the 1,2-diglyceride proportion is currently the best parameter to assess the quality of the harvest, the type of storage before pressing, and the age of an olive oil. 1,2-diglycerides are natural components of the oil and are formed as intermediates during fat (triglyceride) biosynthesis catalysed by acyltransferases. This synthesis stops after harvesting. During storage, and encouraged by higher temperatures, the proportion of 1,3-diglycerides increases as a result of the lipase-induced enzymatic decomposition of the triglycerides until the olives are pressed and there is no more water available to react with the lipases. If pressing

takes place promptly after harvesting, the 1,2-diglyceride proportion is above 90 %, stopping the lipase-induced enzymatic decomposition of the triglycerides. After being stored for too long under unfavourable conditions, values of just 50 % 1,2-DG are not uncommon.

The 1,2-DG molecules gradually rearrange themselves chemically during storage into the 1,3 form. After a year, most oils still have a 1,2-DG level of about 50–60 % if they have been stored and pressed properly. Cooling the oils can slow down the isomerisation of the diglycerides. Extra virgin olive oils with a 1,2-diglyceride level of less than 45 % would generally be expected to have a sensory defect. These oils could no longer be marketed as “extra virgin olive oils” in the event of a sensory defect.

FFA and 1,2-DG content are also good criteria for evaluating age and determining the best before dates. Depending on the FFA content, 1,2-diglycerides rearrange themselves fairly quickly into 1,3-diglycerides. The lower the FFA content, the slower the rearrangement. Thermal treatment, though, cannot be identified based on the 1,2-diglyceride content.

IV – iodine value

The iodine value is an indicator of the identity of the olive oil and is a chemical parameter to characterise fats and oils. Generally it is a measure of the unsaturated fatty acid content of an oil. Irrespective of whether they are refined or virgin, olive oils have iodine values in the range 75 to 88.

Fatty acid composition (C16:0...C18:3)

The fatty acid composition is also an indicator of the olive oil identity. Together with the triglyceride pattern, this parameter permits the detection of any suspected adulteration with other oils. The origin of the oil and its identity, for example, between the samples taken and the shop sample can be checked using these values.

Triglyceride distribution (POP, PLL, OOO, OLL, OLO, LLL)

The fatty acid distribution in the individual triglycerides (triglyceride distribution) can be used together with the fatty acid distribution to determine the identity of the olive oil or to detect adulteration with other oils. The percentages of the individual triglycerides, determined via NIR, cannot be compared with the percentage distribution, determined via GC or HPLC. The results of the different methods cannot be directly compared with each another.

Literature

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