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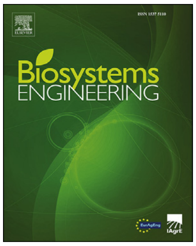

Research Paper

Use of visible and near infrared spectroscopy with a view to on-line evaluation of oil content during olive processing

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The aim of this preliminary feasibility study was to verify whether visible/near infrared(vis/NIR) spectroscopy could be used to predict the oil content of intact olives entering the mill, and of olive paste, pomace and paté during the milling process. Three different extraction methods (3-phase decanters, 2-phase decanters and 2.5-phase decanters) were considered, and two optical devices were tested: (i) a process device for non-contact analysis and (ii) a system equipped with an immersion probe for contact measurements, both working in the spectral range 400–1650 nm. 35 samples of olives were collected during the experimental tests, 50 samples of olive paste, 50 samples of pomace and 50 samples of paté. The collected samples (olives, olive paste, pomace and paté) were used to calculate partial least squares (PLS) regression models.

Results regarding the non-contact analyses were encouraging, except for the measures on olives. On pomace, satisfactory models were calculated for the vis/NIR range [Ratio Performance Deviation (RPD) > 2], and a good model with $R^2 = 0.81$ and RPD = 2.68 in validation was calibrated in the NIR range. The device equipped with an immersion probe achieved good predictive models for the oil content prediction on paté (R^2 and RPD values ranged 0.77–0.82 and 3.00–3.43).

The predictive models could be easily applied in an on-line system to monitoring the entire extraction plant and to perform a feed-forward control, allowing a reduction of oil leakage to minimise the oil losses and to maximise the extraction yield.

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1. Introduction

The food industry converts agro-materials into products using a variety of different processes. The choice of the process to achieve the best supply chain performance is carried out according to the efficiency and quality of the product to remain competitive on the market. For the production of an extra-virgin olive oil of excellent quality, a company should consider implementing and/or improving some parts of the olive oil production chain: olive harvesting and handling; carrying out the milling operation a short time after olive harvesting; using advanced mill plant equipped with suitable technologies to control the milling conditions. The last point is a good starting point for an optimisation of the process because differences from the standard operating conditions are the principal cause of failures to maintain the highest standards during processing and these affect the final product's quality. In the extra virgin olive oil extraction chain, control of the process and its management determines the conditions to produce a high quality extra-virgin olive oil, needed to retain consumer confidence, but process control is also important to evaluate potential losses of the plant's yield. Thus, process control is essential for achieving effective competitiveness.

The extra virgin olive oil extraction plant is set up as a number of interconnected machines arranged in series and in some cases, even in parallel (Leone et al., 2015). The main operations of the entire process are: olive cleaning, olive crushing to obtain a paste, paste malaxation, solid–liquid separation and liquids separation. Solid-liquid separation is one of the most important operations; it consists of separating the solids (pomace) from the other olive components (oil and waste water).

The solid–liquid centrifuges are classified according to the characteristics of the products in output from the decanter (Altieri, Di Renzo, & Genovese, 2013; Leone, Romaniello, Zagaria, & Tamborrino, 2015):

- 3-phase decanters, able to separate oil (5–10% of humidity) from dry pomace (50–55% of humidity) and waste water. In three-phase sedimentation, 10–30% of warm water is added to the inlet olive paste;
- 2-phase decanters, able to separate oil (5–10% of humidity) from the wet pomace (65–70% humidity). In two-phase sedimentation, no water is added to the inlet olive paste;
- 2.5-phase decanters, able to separate oil (5–10% humidity) from dry pomace rich in heavy solids at 50–55% humidity and to produce a new by-product called "paté," which contains wastewater enriched by soft solids without pit fragments and having a semi-solid consistency.

An important evaluation parameter for a decanter's performance is the oil extractability. This parameter is calculated by dividing the oil extracted by 100 kg and the oil contained in those 100 kg of olives. The decanter's extractability can vary according to (i) the type of decanter, (ii) the wear/maintenance, (iii) the cultivar and ripeness of the olives processed, (iv) the particle size and rheology of olive paste and finally on (v) the decanter adjustments. Extractability is in the range 80–90% (Tamborrino, Leone, Romaniello, Catalano, & Bianchi, 2015); the oil that is not extracted remains in the unbroken cells or is trapped in the cytoplasm tissues, or is emulsified into the aqueous phase. The extractability reflects the decanter's correct functioning, and it is very important to know it. The quantification of residual oil in the pomace is considered a crucial control for the qualitative optimisation of the olive oil extraction plant.

To obtain on-line information on the oil content in input olives and in the output by-products allows managers to react to imperfections in the process by corrective actions or by redefining/reinforcing preventive actions. Nowadays the traditional or rapid Soxhlet method is used to analyse the oil content in olives, pomace and paté, requiring a time-consuming drying step, followed by an extraction using solvent. This method is now often substituted in routine analyses by nuclear magnetic resonance (NMR) spectroscopy, but this technique is also not very rapid due to water interference. For this reason, the olive pomace sample must be completely dry. Consequently, this method is unsuitable for an on-line application during process control.

The investigation of the characteristics of the olives entering the milling process and of the features of by-products during milling could allow operators to control the quality of the process. A better monitoring of the oil production process also depends on controlling production of the paste, the pomace and the paté, the intermediate products between the olives entering into the process and the oil outlet from the mill, to establish correlations among olives, paste, pomace, paté and oil. Hence, in the olive oil industry, quick and easy-to-use technologies are required to (i) assess olive ripening and the characteristics of the by-products, (ii) for early detection of possible failures, (iii) to monitor in a lasting way the production process during its crucial steps in order to control the oil quality and yield. The sector could be helped by optical non-destructive and rapid applications for the olive oil production chain optimisation.

Over the last 30 years, on/in-line NIR spectroscopy has gained much success by placing on the market efficient and advanced tools for continuous product quality monitoring in the food processing industry, such as for fruit, vegetables, meat, grain, dairy products and beverages (Huang, Yu, Xu, & Ying, 2008; Porep, Kammerer, & Carle, 2015). Regarding the oil sector, several studies have highlighted the enormous opportunities offered by NIR spectroscopy in terms of applications for quality control during the process, performing on/in/at-line measurements on olive fruits, on pastes, and on oils (Armenta, Moros, Garrigues, & Guardia, 2010). Researchers tend to focus attention on the on-line applications of non-invasive technologies in order to reduce the gap between laboratory scale experimentation and the olive milling industry (Ortega, Gila, Puerto, García, & Ortega, 2016). A number of studies applying different vibrational techniques in the olive oil chain can be found in the literature, mainly with the aim of standardising the procedure for an application as official control of the end product (Nenadis & Tsimidou, 2017). For this purpose, it is crucial to evaluate the optimal spectral range to be used and the chemometric methods to be performed to obtain robust predictive models for the estimated parameters. On intact olives, Giovenzana, Beghi, Civelli, Marai, and Guidetti (2015) and Beghi, Giovenzana, Civelli,