The impact of innovative processing technologies and chemometric methods on virgin olive oil quality - a review

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Cukurova University, Faculty of Engineering, Department of Food Engineering, Adana, Turkey Virgin olive oil production and usage have increased in recent years. Only mechanical or other physical processes are used to extract olive oil from olive fruit. As an alternative to traditional methods, new processing strategies such as high pressure ultrasonic (HPU), microwave (MW), pulsed electric field (PEF), and ultrasounds (US) have been developed to improve the quality, physicochemical and nutritional properties of oils while reducing processing time and energy consumption. The top two olive oil qualities are extra-virgin and virgin olive oils that have unique physical, chemical, and sensory properties. Reference methods for determining quality parameters take a long time, utilise expensive and dangerous chemicals, require an analyst knowledge, require a sample preparation, and may have constraints that limit their use for a precise and thorough quality management of olive oil. Recently developed analytical processes include NIR, MIR, FT-NIR, Raman, NMR spectroscopy, mass spectrometry and chromatographic techniques with Chemometric analysis (PCA, PLS-R, PLS-DA, cluster) to assess the quality characteristics of olive oils quickly and accurately.

This review highlights the significant advancements in virgin olive oil quality assessment and emphasizes the valuable contributions of innovative processing technologies, analytical methods, and chemometric techniques in this field.

Keywords: Virgin olive oil, Innovative olive oil processing techniques, Chemometric, quality control

1. INTRODUCTION

Olive oil "obtained from the fruit of the olive tree (Olea europaea L.) only by washing, decantation, centrifugation and filtration, described as a fragrant, transparent, yellowish-green liquid by International Olive Council (IOC) [1]. The high yield olive oil production begins with harvesting the fruits at their optimum stage of ripening Traditional olive oil extraction process includes separating the leaves, washing and processing the fruits using mills. There are roughly 12 000 olive oil mills worldwide, with over 80% using centrifugation methods for olive oil extraction. The olives are ground and crushed to make the olive paste, which is then subjected to the malaxation process, and the oil will be extracted by using hydraulic presses, centrifugation (two or three-phase system). Then, olive oil is separated from the aqueous phase through centrifugation to be filtrated, stored and packaged [2]. Olive oil is probably an item that has been made and consumed since ancient times. An increase in interest in this oil has been observed and identified with the Mediterranean diet. Olive oil is widely popular in the Mediterranean region, and its consumption has increased globally as a result of its health benefits and sensory properties [2, 3]. World production of olive oil reach about 3 215 000 tones. Turkey is the world's fourth-largest olive oil-producing nation with 235 700 tons of olive oil produced in the 2021/22 crop year (IOC,

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2022). Contablsumption of olive oil in IOC member countries is 2 054 000 tones, while non-member countries consumed 1 071 000 tons of olive oil. Total consumption is expected at around 3 125 000 tones (-4.4%) [4] IOC for the year 2022. Thus, although only 5% of the oil produced in the world is olive oil, its consumption has doubled in the last two decades. Although Spain is the biggest oil producing country, Greece (20 L) is the biggest oil consuming country. Other countries are Spain (14.2), Italy (11.3), Portugal (8), Syria (4.9), Morocco (4.7), Tunisia (3.8) and Turkey (2.2) litres [4-6]. Olive oil quality and purity requirements, as well as safety issues, are specified by numerous legislation and agencies, such as the EU Regulations, as a natural product produced using "only mechanical means" from olive drupes. Legislation adequately describes the authenticity of olive oil. The top two olive oil qualities are extra-virgin and virgin olive oil, which have distinct physical, chemical, and sensory features. The IOC and European regulations are used to classify olive and olive pomace oil [2]. The quality of olive oil is determined by various factors, including geographical location, climate, crop season, tree age, field operations and treatments, and the time and activities used for olive harvesting, processing technology, process variables, and storage conditions. These variables influence olive oil properties such as fatty acid profile, phenolic contents, and sensory qualities. Controlling these variables precisely can result in an optimisation of olive oil quality, production yield, economics, and environmental impact [3], [7-9] grown in five olive orchards with different soil type (Sandy, Clay, Stony, Brown, Limestone and Gypsum.

2. ORGANIC OLIVE OIL PROCESSING

"Organic farming is an agricultural method that aims to produce food using natural substances and processes" [10, 11]. Organic olive oil should be obtained from organic crops, according to EU rules and regulations (EEC regulation 848/2018). According to the reference legislation, the general objectives of organic production are to maintain the health of soil, plants, water, and animals; to promote biological diversity; to use natural resources (energy, water, air, etc.); to obtain high quality extra virgin olive oil; and to satisfy the consumer's demand for organic olive oils that are better for their health and do not harm the surrounding environment. This law also prohibits the use of GMO products (from seeds to feed), and all products of chemical origin are prohibited in the organic olive oil production line. The logo can only be used on products that have been certified as natural by means of an authorised corporate enterprise or body. This is a useful tool for producers to realise certified organic products and for farmers wanting to market them throughout all EU countries. Although, organic farming is more expensive and more complex than conventional agriculture [12] since it offers several advantages, such as minimising all forms of pollution and producing food of high quality. Health is considered to be the competitive advantage of organic olive oil since It may include higher levels of polyphenols and squalene, but most significantly, it is grown and produced without the use of chemicals or GMO goods [3]. Thus, consumers pick organic foods, because they probably incorporate fewer agrochemicals, hormones, and artificial additives in contrast to conventional foods. However, PDO certification is preferred more for their higher perceived quality and consumer interest [13, 14]. In fact, Carzedda recently noticed that origin attributes like PDO or PGI have positive effect on a preference while the organic attribute is not highly valued by Italian consumers [11, 13] for extra virgin olive oil. The world's organic agricultural lands for olive cultivation are 20%. Europe and Africa provide 70% and 30% of the organic olive production, respectively. The organic olive production in Tunisia (254547), Italy (230671), Spain (194249), Turkey (81747) and Greece (50181) hectares. The organic field used in olive cultivation has increased almost three times [13]. Organic olive is a very important product both globally and in Turkey where 8% of olive orchards are kept organically and 2% of whole production is organic olive oil.

Organic farming in Turkey is validated by private Control and Certification agencies in accordance with the Organic Farming Regulation (No. 5262/2004). The Turkish Ministry of Food, Agriculture and Livestock accredited control and Certification private bodies. Organic olive tree production provides numerous advantages, both environmentally, socially, and economically with health and environmental protection, economic benefits for producers and consumers. Furthermore, both cooperatives have joined the organic olive and olive oil markets. Taris previously manufactured organic virgin olive oil, and Marmarabirlik entered the market with organic table olives in 2015 [12]. The organic olive oil production may significantly contribute to the nation's economy with a high added value if the proper safeguards are taken and knowledge is transferred to olive producers [15].

3. NEW EMERGING NON-THERMAL OLIVE OIL PROCESSING METHODS

Consumers want food with a better organoleptic character and more health and nutritional benefits. Simultaneously, there is an increasing awareness of the environmental sustainability of products and processes. They can shorten processing time, increase extraction yield (through rapid mass transfer), preserve sensory qualities (by non-thermal processing), and reduce or eliminate the usage of solvents, all while saving energy. Electro-technologies (pulsed electric field (PEF), high voltage electrical discharge (HVED), Ohmic heating, non-thermal plasma), electroma-

gnetic radiation technologies such as microwaves (MW), radiofrequency drying, pulsed light (PL), high pressure (HPP) or ultrasound (US) processing are the examples of these new technologies. Some of these technologies have the crucial attribute of being nonthermal procedures due to their capability to work at low-mild temperatures, which may be important for virgin olive oil because it is particularly sensitive to temperature during processing [16-18]. New emerging technologies low in energy usage and environmentally friendly processing strategies to reduce the negative effects of traditional olive processing methods, meet rising consumer demand for more natural products with fewer additives and preservatives, while also providing availability, freshness, and safety [16, 18], [25-28] mostly applied before malaxation [3]. They can be successfully implemented in EVOO processing to improve, shorten, or replace the malaxation step, or to eliminate the pre-heating of olive paste; however, they must be carefully designed to produce olive oils with the desired characteristics, as too much power and/or too long treatment times can affect the quality and stability of EVOO components [20]. Malaxation, which includes mixing crushed olive paste to facilitate oil drop coalescence, is critical during olive oil extraction process [16, 29]. Malaxation is the bottleneck in the continuous extraction process since the mechanical crusher and centrifugal separators are continuous devices but the malaxer is a batch equipment. Furthermore, due to a poor ratio between its large volume and tiny surface area, the malaxer has a low heat transfer coefficient and is an inefficient heat exchanger [17, 30]. Oil yield, along with oil quality, is an important component in the oil extraction process. Using current technology and despite ongoing improvement, the extractability ranges between 80% to 90%, as some of the oil is lost in the pomace and waste water, i.e. 10 to 20% [27]. Recent researches have been directed toward the development of malaxing equipment, capable of converting the batch malaxing step into continuous process thereby shortening the malaxation time, decreasing malaxation temperature while increasing both the yield and the quality (more flavour and aroma, less bitterness, a better yield, more antioxidants, and a longer shelf life) in obtained virgin olive oil [17, 18, 27, 31]. Utilisation of new emerging technology could allow for a decrease in malaxation temperature ranging from 26 to 15°C without affecting extraction yield, influencing the functional and nutritional value of the olive oil due to the low temperature of the olive paste, olive oil is produced rather than standard thermal processing methods [31]. Some of the selected studies have been given at Table I.

The traditional method for EVOO extraction includes a malaxation process, which boosts yield by around 5% when compared to non-malaxated olives, however the temperature and time of malaxation can affect the quality of olive oils. Innovative moderate appro-

aches have been presented to boost EVOO output without a negative impact on the quality parameters. Recently, Angeloni et al [18] found that the HPP treatment had no detrimental impact on olive oil quality, however HVED had a negative impact on olive oil quality. Amirante et al. [17] found that using a Sono Heat Exchanger (SHE) in conjunction with US enhanced oil extractability by 5% and polyphenol content by 12% when compared to EVOO samples extracted in the traditional way. Navarro et al. [19] discovered that up to 25% more oil may be extracted, providing enrichment of phenolic and volatile components while having no effect on the physicochemical parameters or tocopherol content and causing no faults or off-flavours of olive oil produced by PEF treatment. Nardella et al [20] HPU increased oil extractability by up to 20%, but the impact of HPU on olive oil quality and chemical composition is contradictory, particularly in the case of micro-components, such as polyphenols, tocopherols, and volatile compounds, due to the various transformations that occur during olive oil production and processing parameters, such as power, temperature, and time of treatment, as well as oxygen concentration in the headspace during malaxation, olive variety, and climate. Thus, an HPU treatment might be designed to increase antioxidants and volatile while limiting oxidative and thermal losses during extraction of olive oil. Amarillo et al., [21] Conditioning olive paste using microwaves and megasonics boosted olive oil extractability by up to 2.4% while having no negative influence on the extra virgin olive oil's sensory or chemical quality criteria (fermentative and oxidative). Servili et al. [24] When utilised at 3.5 bar power, low frequency high power ultrasound boosted oil extractability by up to 4.6% while causing no changes to the legal quality metrics and having a favourable impact on the phenolic composition of EVOO. Stilliatano et al. [25] assessed the efficiency of heating of paste before malaxation and a using vacuum decanter to avoid the final vertical centrifugation on olive oil yield, quality, and economic and environmental impacts (life cycle costing (LCC) and life cycle assessment (LCA). When the innovative system was used, oil quality improved with lower peroxide content and higher contents of chlorophylls, total polyphenols, and tocopherols, as well as antioxidant activity, but with lower oil yields than when the conventional system was used. The economic results revealed that the innovative system had the highest extraction cost as well as the lowest profitability, even though a positive return on investment feasibility can be achieved due to an increase in olive oil selling prices, which could be significant for the sustainability evaluation of innovations in the olive oil industry. US can be applied to olive paste to release oil quickly from vacuoles with a shorter malaxation time, low manufacturing cost, and high extraction yield, because of its mechanical effect on cell membranes [17, 27], [32-34]. Due to the combined effects of higher

Technology	Objectives	Parameters	Results	References
HE and US	Sono heat exchanger combined with ultrasound applied malaxation	Olive oil quality, yield, oil extractability, phenolics compounds, fluid dynamic analysis	Increase in oil extraction (5%), polyphenols (12 %) at 23–27°C	[17]
HPU, HVED	High pressure ultrasound, high voltage electrical discharge applied before malaxation	Olive oil quality (FFA, PV, K ₂₃₂ , K ₂₇₀ , ΔK), phenolic compounds, volatiles	HVED caused increase in anisidine value (50 %), fall in biophenol concentration (5 to 10 %), 15 % in volatiles at 19 °C	[18]
PEF	Pulsed electric field applied before malaxation/malaxation time and temperature	Olive oil quality, yield, oxidative stability, phenolic compounds, volatiles, tocopherols	PEF increased oil yield up to 25%, 10– 17% higher oleacin and oleocanthal and hex-2-enal content, unaltered physicochemical parameters, tocopherols below 20 °C	[19]
HPU	Ultrasound applied before malaxation/frequency, malaxation time	Olive oil quality, yield, phenolic compounds, volatiles, consumer perception	HPU increase the oil extractability up to 20 %, the effect on olive oil quality and chemical composition contradictory	[20]
MW and MSW	Microwave and megasonic wave applied malaxation/ malaxation time	Olive oil quality, yield, oxidative stability, ethyl esters and wax content, polyphenols, tocopherols	MW caused increase in oil extractability 2.4 %, does not negatively impact on the sensory chemical quality	[21]
US	Ultrasound assisted PDO malaxation	Consumer perception, sensory properties	Extra-virgin olive oil extracted through ultrasounds may be, generally, accepted by consumers.	[22]
US	Ultrasound assisted malaxation	Volatiles, phenolic compounds, antioxidant activity	Increase of the yield has not impaired any difference in chemical composition, sensory characteristics, the nutraceutical properties of EVOO produced by US and heat exchanger	[23]
US	Low frequency high power ultrasound applied malaxation / different levels of pressure	Olive oil quality, yield, oil extractability, phenolic compounds, volatile compounds	Ultrasound technology İncreased oil extractability to 4.6 %, no alterations to quality parameters, showed a positive impact to the phenolic composition at 3.5 bar	[24]
-	Low Oxygen Pressure milling, vacuum malaxation	Quality, and olive oil extraction yield	Innovative plant resulted in olive oil with a significant increase in quality, with lowest in extractability, unfavourable in life cycle costing and life cycle assessment	[25]
Microwave, Megasaound	Microwave and megasound applied malaxation/ malaxation time	Olive oil quality, oil extractability, phenolic compounds, volatile compounds	Combined continuous MW and megasonic conditioning technology to fasten the olive oil extraction, to enhance yields, and total phenolic content	[26]
US and PEF	US and PEF technologies in an industrial olive oil mechanical extraction plant	Olive oil Yield and Rheological Characteristics of olive paste	US and PEF system before malaxation increased oil extractability of up to 3.7 % and oil yield up to 0.54 %. Caused a slight decrease in viscosity	[27]

Table I - Some Innovative Non-Thermal Processing Technologies for Obtaining Virgin Olive Oil

processing temperatures and cavitation, HPU processing, particularly to aid malaxation, tends to greatly improve oil production and increase oil extractability [20]. PEF is effective for reversible or irreversible cell membrane permeability by subjecting olive paste to an electric field, which causes pores in cell membranes. The potential of PEF and HPP applications is to increase extraction yield and lower malaxation time and getting olive oil with a high phytonutrient content, including bioactive and antioxidant components, as well as health-promoting characteristics [27, 28, 35] since they have enhancing ability for mass transport thus improving the extractability of intracellular bioactive compounds from olive paste. These technologies are also effective at increasing the oxidative stability of olive oils without any negative impact on their flavour, colour, and consistency. These technologies have a significant effect on oil quality parameters, nutritional and sensory features such as no changes in the fatty acid content and volatile components of the virgin olive oil, but they resulted in an increase in tocopherol, chlorophyll, and carotenoid contents, yield, extractability while reduced in malaxation time [17]. Romaniello et al. [27] found that the application of the US and PEF systems prior to malaxation boosted oil extractability to 3.6 to 3.7% and yield to 0.5 to 0.4%, respectively. Microwaves are non-ionising electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz that can be used to shorten the period of malaxation while also enhancing oil release [30]. It is an innovative extraction technology applied during the extraction of olive oil, that causes disruption of cell wall materials facilitating the release of high-quality oil with low energy requirement, and has significant environmental impact and low financial costs, due to heating [18, 26, 35].

Leone et al. [26] discovered that new conditioning of olive paste employing MWs, MS, and their combination at an industrial scale had no significant effect on extra virgin olive oil quality and related chemical and sensory descriptors. A combination of continuous MW and megasonic conditioning technology allows for faster olive oil conditioning, higher yields, and oils with a higher overall phenolic content.

By-products of the Olea europaea L. processing industry are rich in various bioactive compounds such as polyphenols, anthocyanins, tannins, flavonoids, and dietary fibre (pectin) can be recovered and reused for a variety of applications. Traditional extraction procedures such as the use of organic solvents and filtration processes (membrane) Soxhlet, hvdro-distillation, and solvent extraction methods can be used to separate these bioactive compounds from olive by-products [36, 37] but applied at considerably high temperatures. However, green extraction technologies like high-hydrostatic pressure, and ultrasoundassisted extraction, microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE), pulsed electric field, Radiofrequency drying, high voltage electrical discharge, and super-heating are all methods of low temperature extractions. SFE and PLE (critical fluid extraction and pressurised liquid extraction) may also be used to concentrate these valuable by-products [36, 38]. Based on the results of economic and environmental studies, the most efficient and profitable extraction method was found to be the innovative green technologies despite their high extraction cost and their low or zero negative environmental impacts [25].

4. QUALITY CONTROL OF VIRGIN OLIVE OIL WITH CHEMOMETRIC METHODS

Virgin Olive oil is a special food with a high price. Adulteration, mislabelling, mischaracterisation, and fake origin are all examples of variables that affect authenticity. Therefore, quality and safety as well as the trade in vegetable oil products, depend greatly on the detection of quality and fraud [39]. Previous studies are concentrated on classification, monitoring of adulteration, characterisation of cultivars and determination of the geographic origin of olive oil. Even if virgin olive oil analysis remains a cornerstone in terms of diagnosing possible fraud, there is a need for the development of new procedures for assessing quality, authenticity and also determining geographical and botanical origin [39, 40]. Traceability is another important quality that must be secured throughout the full virgin olive oil acquisition process, encompassing the four fundamental stages (harvesting, milling, storage, and packaging) to ensure the product's identification and manufacturing chain [41].

The free acidity (FA), peroxide value (PV), and specific extinctions (k232, k270, and k) obtained according to the official methods under the codes COI/T.20/ Doc/N°34, N°35, and N°19, respectively, are wellknown chemical parameters used to determine the quality and authenticity of OO and to classify VOO grade [47]. IOC and EC have both approved that chemical parameters along with sensory properties (fruity attributes, defects) are the most common parameters that define the olive oil guality. International Olive Council establishes moisture and volatile matter, insoluble impurities in light petroleum, flash point, trace metals (for iron and copper), fatty acid ethyl esters and biophenol contents as set as additional quality criteria. Moreover, oxidative stability, chlorophyll and carotenoid content, and the bitterness index [1, 42] and contaminants (such as pesticides or mycotoxins, among others) are also included for the evaluation of the quality and minimising associated risks of olive oil [1, 3, 5]. There is growing interest in novel, robust, quick, and cost-effective analytical methods for investigating olive oil quality for authentication and traceability. Because several of the official analytical procedures used for the certification of virgin olive oils have limitations in certain aspects, a number of alternative analytical methods and approaches have been proposed over the last decade [1, 5]. The analytical procedures (including sample preparation, analysis, data acquisition and processing) have been developed and proposed to quality control of virgin olive oil are; Vibrational spectroscopic techniques (Near-infrared (NIR) Visible/ Near Infrared (Vis/NIR), Fourier transform infrared (FT-IR) and Fourier transform-Raman (FT-Raman): Mass spectrometry (electrospray ionization (ESI), Atmospheric pressure photoionisation ion (APPI), Matrix-assisted laser desorption/ionisation (MALDI), fingerprinting method based on MALDI-TOF MS); Chromatographic techniques (GC and HPLC) and other analytical approaches (voltammetry, DSC and e-sense techniques) [42] have been used. These techniques have been used for the determination of acidity, peroxide value, iodine value, anisidine value, malondialdehyde, soap contents within a single measurement to determine olive oil quality with significantly good results in a very short time [43-48]. These new analytical procedures could determine and predict several parameters because they offer important advantages such as no need of reagents, rapid measurements and fast data acquisition, relatively low cost, easy samples handling because of their non-destructive nature (analysis is performed directly on intact samples or with only minimal sample preparation) [49]. The produced analytical data (spectroscopic, chromatographic, isotopic, sensorial, etc.) are often multivariate data matrices which demand appropriate Chemometric analysis that allow to be used as rapid screening techniques compared to the standard reference methods for determining the quality and authenticity of olive oil [44, 47]. Thus, various procedures have been created and proposed for the quality control of virgin olive oil, as well as discrimination and classification are shown at Table II.

Azazian et al. [45] created FT-NIR calibration models to assess virgin olive oil quality by evaluating thirteen parameters, including five major FAs and DAG and FFA concentrations. Alamprese et al. [50] Image analysis was used to create FT-NIR PLS-DA calibration models for determining the olive ripening degree. PLS-DA models developed independently for olive origin yielded prediction sensitivity and specificity values greater than 81%. As a green, non-destructive, extremely dependable technology for optimising virgin and extra virgin olive oil quality, such a tool can be used for sorting olives right at the mill's entry or even

Table II – Recent Analytical Methods used with Chemometric Techniques for Virgin Olive Oli Quality Contr

Methods	Objectives	Parameters and Data Processing	Results	References
FT-NIR	Quality and Authencity of EVOO	Volatile aldehydes and ketones, triacylglycerol, diacylglycerols, free fatty acids, phenolics, and water, PLS	FT-NIR calibration models allow assess the authenticity and freshness of EVOOs, the linoleic acid composition (15 to 21 %) of the oil, and establish the volatile and water content	[45]
FT-NIR	Quality of olives	Olive ripening degree, PLS-DA	FT-NIR PLS-DA calibration models confirmed by common visual evaluation of maturity index up to 81 %.	[50]
NIR	Quality	Moisture content, PLS	FT-NIR with PLS calibration models correlated well with KF reference values up to 95 %	[51]
HPLC-ESI-MS	Characterization	Phenolic and tocopherol compounds, pigments, oxidative stability, triacylglycerol, and fatty acid compositions, PCA and HCA	PCA and HCA can explain the variability of the oil composition according to the cultivar	[52]
HS-SPME-GC- MS	Authentication	Volatile compounds, LDA, PLS-DA	LDA and PLS-DA Chemometric models can be used to discriminate monocultivar virgin olive oils based on their volatiles up to 94 % confidence level.	[53]
HS-SPME-GC- MS	Quality assessment	Volatile compounds, PCA, HCA, LDA, kNN	PCA and HCA were applied for clustering virgin olive oils and LDA, kNN, and SVM predictive models correctly classified the oils up to 88.1%.	[54]
UHPLC-UV/Vis	Authentication	Polar fraction of olive oils, PLS-DA, SVM, SIMCA	UHPLC-UV/Vis spectrums are transformed to instrument-agnostic fingerprints by using SIMCA, PLS-DA and SVM models to discriminate virgin olive oils	[55]
Flash GC	Quality grades	Volatile fraction fingerprints of virgin olive oils, PLS-DA	Volatile fraction of virgin olive oils analysed by flash gas chromatography to predict the commercial category of olive oils by using PLSDA models classifying up to 85 %	[56]
Flourescence Spectroscopy	Quality	Acidity, peroxide value, K232, 270, ΔK, tocopherols, PLSR	PLSR models with 0.9 R ² values were found at excitation at 350 nm, for correctly measuring physiochemical properties and tocopherol contents of virgin olive oils	[57]
HPLC-DAD	Variety Origin	Fingerprints of phenolic fraction, PCA, PLS-DA, SIMCA, kNN	Virgin olive oils accurately classified between 92.23 and 94.17%, best results were obtained with SIMCA models	[58]

*PCA: Principal Component Analysis, PLS: Partial Least Square, DA: Discriminant Analysis, R: Regression, HCA: Hierarchical Clustering Analysis, LDA: Linear Discriminant Analysis, kNN: k-nearest neighbour, SIMCA: Soft independent modelling of class analogy,

in the field. Moisture and volatile matter are olive oil guality characteristics, according to CODEX, and the creation of a quick screening method for moisture assessment is critical [51]. Karunathilaka et al. [51] The NIR method presents a clear time and cost saving alternative to the KF method and would be appropriate for routine screening applications provided calibration is effectively achieved, as FT-NIR PLS calibration models gave a sensitivity up to 95% when compared to the reference laboratory Karl-Fischer method. Baccori et al. [52] investigated the selection of novel feral olive cultivars for oil production with high oil quality. The results show that the chemical properties of the oils under consideration vary greatly. The statistical analysis (PCA and HCA) can explain the variation in oil composition by cultivar. They determined that feral olive oils are a new edible oil source that is high in natural bioactive components. Cecchi et al. [53] found that volatiles that have a considerable influence on customer preferences influenced by the varietal origin of virgin Olive Oil (VOO). The LDA and PLS-DA algorithms properly classified 94% of the oils into three clusters based on their volatile components that will help against fraud and to increase the value of monocultivar extra virgin olive oils. Gerhardt et al. [54] used HS-GC-IMS to analyse non-targeted volatile organic compound (VOC) profiles in order to differentiate between virgin olive oils of different classification. PCA and HCA were used to cluster virgin olive oils. while LDA, kNN, and SVM were employed to develop predictive models predicting the oils volatiles correctly up to 88.1%. Perez Beltrean et al. [55] The polar fractions of several olive oil samples analysed by (NP) UHPLC-UV/Vis are translated to instrument-agnostic fingerprints using PLS-DA and SVM models obtained before and after signal instrumentation. This will enable the development of multivariate classification models that can be exported between laboratories and generally deployed in routine laboratories to readily authenticate olive oil. Barbierei et al. [56] Using PLSDA models, the volatile fraction of virgin olive oils was analysed by flash gas chromatography to predict the commercial category of olive oils, and 331 olive oil samples were accurately classified ranging from 72 to 85%. Baltazar et al. [57] set multivariate models to evaluate the physicochemical properties and antioxidant content (tocopherols) of extra virgin olive oils from fluorescence spectra obtained at 326 nm, 350 nm, and 365 nm excitation wavelengths. In evaluating the physiochemical characteristics and antioxidant content of virgin olive oils, PLSR prediction models were created reaching 0.9 R² values excitation at 350 nm. Bajoub et al. [58] investigated the phenolic constituents of 140 extra-VOO samples from seven olive fruits and the results statistically processed for varietal authentication purposes using PCA, PLS-DA, SIM-CA, and k-NN. Overall classification accuracy for the authentication of virgin olive oil samples was between 92.23 and 94.17%, with the best results achieved by

using SIMCA models for classification. Furthermore, computer vision, machine olfaction technology, electronic tongues and dielectric spectroscopy, differential scanning calorimetry, and voltammetry are used to determine parameters such as acidity, peroxide indexes, ripening indexes, organoleptic properties, fatty acids, volatiles, the melting point, odours, flavour and minor components in olive fruit, olive slurry, and olive oil to determine quality, adulteration, freshness stage and to predict sensory attributes of olive oil [47, 48, 59].

5. CONCLUSIONS

In recent years, the quality assessment of virgin olive oil has received increasing attention due to its nutritional and health benefits, as well as its economic importance. Olive oil authentication is vital not solely for consumers, but additionally for suppliers, retailers, regulatory agencies, and administrative authorities. The production process of extra virgin olive oil is conducted, as known, only by means of mechanical-physical methods applied to the treatment of the olive fruit. Various emerging processing Technologies suc as US, PEF, and MW have been developed and applied to enhance the quality of virgin olive oil during or instead of malaxation. These technologies can improve the yield, purity, stability, and preserve the natural compounds present in the virgin olive oil. The quality and safety of olive oils are regulated by governing organisations such as the IOC and the EU using physicochemical characteristics and sensory attributes. Analytical methods play a crucial role in assessing the quality parameters of virgin olive oil. Recent advancements in spectroscopic (NIR, MIR, FT-NIR, Raman), techniques have enabled the identification and quantification of minor constituents, volatile compounds, and sensory attributes with greater accuracy and sensitivity. These comprehensive analysis allows for a more detailed understanding of the composition and quality of virgin olive oil. Chemometric techniques have emerged as powerful tools to analyze and interpret the vast amount of data generated from innovative processing technologies and advanced analytical methods. These techniques, including PCA, PLS-R, and cluster analysis, can identify patterns, correlations, and outliers in the data, providing valuable insights in optimizing processing conditions, predicting sensory attributes, and determining the authenticity and geographical origin and quality of the virgin olive oil.

Systems for process monitoring, quality control, quality prediction of virgin olive oil can be utilised for relevant parameters in both industry 4.0 and 5.0. Overall, by investigating the impact and effectiveness of innovative processing technologies, analytical methods, and chemometric techniques, contributes to the understanding of how these advancements can enhance the quality of the virgin olive oil.

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