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Seed oils

Biochemical composition and antioxidant properties of Algerian date seed oils (*Phoenix dactylifera* L.)

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Date trees (*Phoenix dactylifera* L.) are widely cultivated in Algerian oases and are the main sources of income and the economic base of these areas; in fact, to date, more than 13 million plants and 940 varieties have been registered for a total production of approximately 1.13 million tons. Dates are fruits rich in essential nutrients, vitamins, minerals and dietary fiber with multiple health benefits [1, 2]. The main phenolic compounds of date fruits, in addition to cinnamic acid derivatives, are gallic acid, protocatechic acid, p-coumaric acid and ferulic acid [3], highly reactive bioactive compounds that act against free radicals and counteract the oxidation of proteins and lipids. The seeds, on the other hand, contain exceptionally high concentrations of phenolic compounds compared to fruits which could be explained by considering their fundamental importance for the survival of the plant itself [4-6]. Few studies have focused on the cultivation practices of date plants and on the chemical and biological composition of oil deriving from seeds [7-8]. The seeds can contain an amount of oil ranging from 5 to 13% and the studies found in the literature have shown that it is a rich source of important molecules that play a role in reducing the risk of many diseases [9-14]. However, there is little information that takes into account the different genotypes, growing areas and

cultivation practices of these plants [3, 7, 8]. Therefore, in this study we wanted to present the data obtained from the analysis on the chemical composition and antioxidant properties of seed oils from eight Algerian date cultivars (Arechti, Degla-Baida, Deglet-Nour, Ghars, Haloua, Itima, Mech-Degla and Tentbouchet). The results highlighted the potential of this oil as a beneficial food thanks to the presence in it of several important compounds with high antioxidant capacity. In particular, the analyses of fatty acids showed high percentages of oleic, lauric, myristic acids; triacylglycerols analysis showed the presence of 1-myristoyl 2-oleoyl, 3-linoleoyl glycerol, 1-linolenoyl 2-oleoyl 3-linoleoyl glycerol, 1-2-linolenoyl 3-linoleoyl glycerol and 1-linolenoyl-2-myristoyl-3-linoleoyl glycerol. Analysis of biophenols and tocopherols revealed the presence of many antioxidant compounds and vitamin E. From what has been observed, the production of date seed oil should be considered both as a new economic resource for the country and an alternative for the disposal of by-products derived from the processing of the fruits [15].

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Pressing seed oils: current situation under the regulatory standpoint

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As pressing seed oils, we mean those fatty substances obtained from seeds or fruits other than olive oil by mechanical pressing, for which only physical operations are allowed and which are marketed in their native state, unrefined.

This product category has long been included in the CODEX regulation (STAN 210-1999), it is quite widespread in other European markets, but in Italy it has never had an important development because of a particular existing local regulation (law of 27 January 1968, n. 35 otherwise known as “Salari Law”), that imposes the refining of seed oils, in order to make them suitable for the food market.

Furthermore, until a few years ago, the obligation for bleaching seed oils as a protective measure against olive oil was in force in Italy

During the last years of the last century, the CTG (Governmental Technical Committee) federated with UNI, the Italian body for Standardization, which was in place at the Experimental Institute for Oils and Fats (SSOG), set up and published a series of Standards to regulate a sector of potential interest for companies and consumers. For about 20 years, until the pronouncement of some courts and the stance of some Ministries, the national production and trade of these oils remained extremely marginal, due to obvious regulatory problems.

The abolition of the bleaching obligation, achieved thanks to the joint action of WG 18 "Oils, Animal and Vegetable Fats and their by-products" of the Agri-food Commission of UNI, of the Italian Society for the Study of Fatty Substances (SISSG), of ASSITOL with the contribution of the INNOVHUB-SSI-SSOG Researchers, resulted in the publication of the DEGREE-LAW 14 December 2018, n. 135 “Urgent provisions on support and simplification for businesses and the public administration”.

As a side fact, the refining process of "traditional" seed oils is facilitated, with undoubted advantages also from the point of view of the formation of any process contaminants. UNI activity led to the publication of new Standards for peanut, corn germ, rapeseed and sunflower oils, while those relating to hemp oil are actually in progress.

Assitol recently launched a series of official debates, in order to clearly define the regulatory frame of these oils, to allow sector operators to carry out their business in a clear regulatory context.

The presentation traces the regulatory evolution here discussed and goes into detail on the quality parameters established for this product category according to the different sources.

Durum wheat: a germ for sustainability

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Cereals are the main source of energy in the diets of populations all over the world, thanks to the prevalent presence of carbohydrates, and durum wheat (*Triticum turgidum* L. var *durum* Desf.) is the main ingredient used in the production of pasta, bulgur, couscous and many types of bread. The semolina production, obtained following the grinding of durum wheat after removing the germ, however, determines the production of by-products, among which the germ and bran are the most important quantitatively. These by-products, mostly intended for animal feed, contain a fair amount of lipids which, if properly recovered, could represent an added value not only to ensure the sustainability of the entire supply chain but also to increase the nutritional value of food.

The goal of the research was therefore to characterize the lipid fraction of the milling by-products of durum wheat, also evaluating the modifications during refining. Durum wheat oil, even at the end of the refining process, appears to be a source of PUFA and bioactive compounds (tocotrienols and tocopherols, carotenoids, phytosterols and policosanols) which, as well known, play a key role in ensure human health. These compounds can reduce the incidence of cardiovascular diseases and diabetes, as well as having anti-inflammatory and cancer-preventive properties.

Durum wheat oil is not yet available on the market although could represent an important opportunity for applying the circular economy to milling industries. The richness in tocopherols and tocotrienols could improve the nutritional and gastronomic value of foods, as well as prolong their shelf-life.

Lipid characterization of a medieval oil from Occitan valleys: the “marmot oil”

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The Brigantina apricot (*Prunus brigantina* Vill.) represents an endemic species of the Western Alps and from apricot stones it is possible to obtain a vegetable oil also known as "marmot oil", largely used in the past in the western Alpine region as substitute of olive oil. Today, marmot oil continues to be used in France while in Italy has drastically decreased. The present work represents the first study on lipid composition, in terms of free acidity, peroxide value, *p*-anisidine index, composition in total fatty acids and sterols, of Brigantina apricot stones oil collected in the Alpine valleys around the Cuneo area. The oil was characterized by a meaningful free acidity (12.5%) and oxidative profile since peroxide value and *p*-anisidine index was equal to 7.73 meq O₂/kg oil and 54.8; respectively. On the other hand, the composition in total fatty acids was mainly composed by monounsaturated fatty acids (~80% of the total fatty acids) and polyunsaturated fatty acids (~15%), while the saturated fatty acids were about 6% of the total fatty acids. Oleic acid (77.7%) was the main fatty acid followed by linoleic (14.4%), palmitic (4.2%) and stearic acid (1.3%). The sterol fraction consisted mainly of *B*-sitosterol (~70% of the total sterols), dehydrocholesterol (~14%), campesterol (~6%), sitostanol (~3%), Δ 5-avenasterol (~3%), citrostadienol (~1%); ergosterol (~1%) and cholesterol (~0.6%), while latosterol and campestanol in trace amounts were also determined. Furthermore, the presence of vitamin A and E has been observed. Based on these results, it is important to highlight that "marmot oil" could find application in various sectors (cosmetic, nutraceutical, nutri-cosmetic) going to expand the sources of bioactive lipids. However, further studies are needed to reduce the impact of the hydrolytic and oxidative phenomenon also evaluating the effect of soil and climatic conditions as well as harvesting, extraction and storage techniques as related to oil quality.

A multi-methodological approach for the hemp seed oils characterization

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Hemp seed oils are a cold-pressed oils with high nutritional value. Seeds and derived products are the only hemp part authorized for human consumption in Europe. Although in the last years the interest towards hemp food products is significantly increased, a specific regulation with quality analytical parameters is still lacking. In our work, a multi- methodological approach was carried out to study commercial hemp seed oils to provide information regarding the quality of the products present on the markets [1]. The proposed protocol is based on nuclear magnetic resonance (NMR) methodology as well as conventional analyses used for extra virgin olive oils (Regulation EU 2015/1830). In particular,untargeted NMR methodology was applied to identify and quantify simultaneously different classes of compounds namely fatty chains, β -sitosterol and aldehydes finding an ω -6: ω -3 ratio, in some cases, different from that reported on the bottle labels. The obtained results showed a great variability between the examined samples and the oil quality was in some cases poor although price was very high.

The results here reported, together with other recently studies, can constitute the starting point for the development, and drawing up, of harmonized guidelines with suitable quality and safety parameters specific for hemp seed oils. Furthermore, these studies might drive producers to standardize procedures of hemp seed oil production, guaranteeing the achievement of a good food objective, consumer safety, and the further expansion of the hemp food industry.

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Valuable oily seeds / cold pressed seed oil world production and market and risk of the contaminants during supply chain

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Turkey is known as the world's seventh largest agricultural producer, especially cereal crops and oil seeds. The majority of production of vegetable oils in Turkey is obtained from sunflower. In addition, cottonseed, soybean, rapeseed, safflower, olive and corn, Oilseed production in Turkey, which was 4.02 million tons in 2018, is expected to increase to 5.40 million tons in 2023. Only sunflowers account for 69% of vegetable oil production, 84% of total oil consumption, and 32% of total oil use. The demand for cold pressed oils has been increased in recent years due to their well-preserved nutrients and bioactives contents.

The global cold-pressed oil market size was valued at \$24.62 billion in 2018, and is expected to grow of 5.3% by 2026. Cold pressed oil market is classified into food industry, agriculture, and cosmetics & personal care industry by application. In 2018, based on region, North America is the leading segment in cold-pressed oil market owing to rise in health awareness among the population leading to healthy lifestyle. For example, cold pressed rapeseed oil is available across many temperate regions of the world including Northern Europe where its popularity is growing.

Traditional methods of oil extraction use excessive amounts of organic solvents and need high-energy input. Current environmental issues associated with organic solvent disposal demand alternative methods for the extraction of edible oils that are environmentally friendly and energy-efficient. Green technologies identified some alternative methods suitable for edible oil extraction. This has led to the improvement of more energy-efficient and eco-friendly green techniques that reduced the utilization of toxic organic solvents and enabled high-quality products to be developed. Edible oils can be produced as nonrefined (cold pressed and virgin) or as refined edible oils. In the production of edible non refined oils, cake is obtained as a by-product. From the cake after the production of crude oils, the remaining oil is extracted by solvent extraction and remains as a secondary product. Crude pressed oil and crude extracted oil are passed through a whole series of steps of the chemical or physical refining process, to obtain edible refined oil. Edible unrefined oils include cold pressed oils (CPO) and virgin oils. Unrefined oils are a category covered by technical regulations in the field of edible oils: "Cold pressed unrefined vegetable oil is produced without heating, precleaning, dehulling and milling mechanically.

Cold pressed unrefined oil can only be purified by washing with water, precipitating, filtrating or centrifuging." Cold pressing techniques are becoming an interesting substitute for traditional methods because of consumers' desire for safe and natural edible products. The advantages of this technology at an industrial level include lower energy consumption and lower investment cost. CPO are preferred to refined oils as they contain more antioxidants and bioactive substances like sterols, carotenoids, and phenolics. More natural biologically active substances such as phenolic compounds and tocols are present in CPO, which could improve oxidative stability. The main disadvantage of cold pressing techniques is the high capital or investment required compared to conventional methods. In addition, CPO have low efficiency and are not always of the same quality. Most CPO contain high amounts of polyunsaturated fatty acids (PUFA), which might be disadvantageous in terms of oxidative stability. CPO could also contain higher amounts of pro-oxidative compounds, so their shelf life might be shorter compared to refined oils. To increase the oil yield

from cold pressing, some pretreatments could be applied to seeds before pressing, such as enzyme application, microwave treatment, steaming, and roasting.

One of the important problems of the oil industry is the process contaminants, even though there is a lower risk in cold pressed oils compared to conventionally refined oils. Along the production chain, both MOH (MOSH, MOAH) and PAHs can enter the oil product through different ways as harvesting, manufacturing, packaging, transportation, storage process, and environmental pollution. Industrial, refined fats often show MOSH/MOAH levels only of higher molecular mass range >C24 or even >C35. A provisional ADI of 0.6 mg/kg was established for MOSH by EFSA, FAO and WHO. According to the Regulation No. 835/2011 by the European Union, the limits for PAHs, and the maximum value for BaP is 2.0 µg/kg and a limit for the total of 4 PAHs is 10.0 µg/kg for vegetable oils. There are very limited works on identification of PAHs contents in cold pressed oils.

In this study, oil seeds production values in Turkey will be reviewed and compared with the production of other countries. The world market place of cold pressed oils will be discussed as referring their nutritive properties. Oil extraction techniques is to be discussed. The extraction efficiency and oil qualities will be referred based on extraction techniques. Additionally, contaminants risks effects in cold pressed oils will be mentioned during production chain.

Extraction process of avocado oil

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On this occasion, I have the opportunity to present the plant manufacturer Amenduni Nicola S.p.A., of which I am the technical manager.

The Amenduni company is one of the largest manufacturers of olive pressing equipments; in recent years, the company has paid further attention, in addition to technological innovation, to the reduction of energy consumption and the use of renewable sources in the production of calories for heating the water used in the extraction process oil.

During these last years, given the increase in the production of some fruits (avocado, mango and papaya), from which it is possible to obtain their important oily fraction, Amenduni, always attentive to new operational dynamics, has diversified, creating a specific technological section to be used for new market demands, so as to be able to satisfy these new productions; also contributing to the improvement of the socio-economic conditions of the producing countries, as well as increasing the quality and also the quantity of the oils obtained from these fruits, compared to the previous artisanal and / or discontinuous activities, until recently used in those countries.

The report presented will mainly focus on the Avocado oil extraction process with the innovative milling and pressing technology of which Amenduni is recognized as one of the major players in this sector.

The presentation will be an opportunity to detail the function of each piece of equipment used in the production cycle of avocado oil, the fruit of which, as it is rich in fiber, it is necessary to adopt particular precautions, in order to obtain an efficient product yield. together with the good quality of the extracted avocado oil.

Furthermore, depending on the conditions of integrity, in which the fruit is found (lack of physical, mechanical damage and/or insect infestation, at the time of milling), the qualitative difference between virgin avocado oil will be presented and discussed (edible) and raw avocado oil, inedible for direct food consumption.

Obviously, obtaining directly from the extraction of the avocado fruit, oil ready for consumption, is of the utmost importance for the producer and more generally for the economy of the area and of the country in which this production activity has now reached an important activity of work, as well as social) sustenance.

Olive oil

Latest developments in EU marketing standards for olive oil

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The European Union is the leading producer, consumer and exporter of olive oil. In this context, olive oil marketing standards have been part of EU legislation since 1991, in order to ensure that the market is supplied with products of a standardised and satisfactory quality that meet consumers' expectations, to facilitate trade and to ensure a level playing field for EU producers. Legislation in the field comprises quality and purity parameters to be met by all categories of olive oils, but also rules for placing those oils on the market and for rules for quality control.

The European Commission is responsible for the EU legislation on marketing standards for olive oil and is constantly updating the regulations to keep them in line with international standards and consumer preferences.

The presentation will focus on the drivers for changing EU regulations for olive oil marketing standards, the procedures for updating them and the interrelation with international quality standards. While looking at the various policy initiatives that influence EU marketing standards top-down, the presentation will not neglect the bottom-up initiatives impacting those standards, and will show how stakeholder input is collected and taken into account along the way. Choices made when revising EU marketing standards will be clarified drawing on existing studies and consultations carried out in recent years. The presentation will also touch upon the inter-play with some of the horizontal food safety issues that might have an important impact on the marketing of olive oil to consumers. Finally, the changes in the latest revision of EU marketing standards, which is expected to be finalised at the end of the summer 2022, will be presented.

Sensory and instrumental dynamic methods to investigate perceived quality of Italian Extra Virgin Olive Oils

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Sensory perception during food eating is a dynamic process, involving both chemical-physical and cognitive factors. For this reason dynamic measuring methods appears to be more adequate to measure, monitor and understand such processes and how they interact.

Furthermore, the differences in sensory responses arise from the individual interaction between the product and the consumer, thus it is necessary to take into account inter-individual variability as well.

The present contribution describes the analytical approach we adopted in the frame of Violin project for the characterization and the valorization of the Italian extra virgin olive oils (EVOOs) and part of our recent results. We studied the variability of the products and food carriers and their interactions using time resolved sensory and analytical techniques.

Simultaneous sensory profiling by Temporal Check-all-that-apply (TCATA) [1] method and nose-space volatile compound analysis by PTR-ToF-MS [2] allows the investigation of how the volatile organic compounds (VOCs) may play a role in flavour perception during the tasting of EVOOs, consumed alone or in combination with other food carriers (bread or chickpeas). Products and carriers affected the release of EVOOs' VOCs in the oral cavity during tasting and specific VOCs resulted related with the perception of defined flavour attributes. Nose-space analysis differentiated the samples mainly on the base of EVOO typology, while TCATA analysis highlighted differences mainly on the base on food carriers. The multisensory integration that underlies flavor perception can be considered accountable for the presented discrepancy.

Differences in panel's sensibility for the oral sensations such as bitterness, pungency and astringency, known to be of primary importance for the acceptability of EVOOs [3], may explain observed differences in the interactions among monitored flavours.

Combination of time resolved sensory and instrumental measurements provides a new and powerful tool to shed light on the complex interactions occurring between physico-chemical food properties and sensory perceptions during eating.

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Flavored olive oils by co-milling of olives, black pepper and orange fruits or pomace: compositional characterization, sensory properties and sustainability aspects

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Olive oil is one of the most appreciated functional food in the Mediterranean diet, thanks to its unique flavor and health benefits that are mainly related, respectively, to the presence of volatile compounds and phenolic molecules. Moreover, the abundance of specific fatty acids esterified in the triacylglycerols, in particular oleic acid, has positive effects against cardiovascular diseases, cancer and diabetes. During the milling phase, the addition of other vegetable matrices to olives can enhance the intrinsic nutritional and sensory characteristics of the virgin olive oil, making it useful for specific food preparation, such as marinates and seasonings for meat, fish and shellfish, and salads. This kind of co-milling technique is one of the possible approaches applied to produce flavored oils.

The reuse of food by-products and waste during the milling phase can be considered as a valorization technique that meets the sustainability purpose as well as the transition toward a more circular economy, considering, as a case study, typical and local products of the Mediterranean area, and particularly Morocco.

In this context, this work aimed to produce flavored olive oils with the use of vegetable products and/or their by-products and waste, specifically by co-milling of olives and: i) entire orange fruit ii) orange pomace iii) black pepper and orange pomace. Control samples by milling the same batch of olives were also produced.

All the oils have been obtained by using an Abencor® lab-scale mill. Some compositional (volatile and phenolic profiles by SPME-GC-MS and HPLC-MS/MS, respectively), and sensory characteristics (through descriptive analysis) of the oils were investigated as well as the free acidity contents.

The herein flavored olive oils are potentially interesting for consumers with peculiar enogastronomic culture and attention to the environment. The obtained results are useful to evaluate quality and sustainability of the presented production chain.

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Characterization of new lipophenols in olive oil and olive oil by-products

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Olive oil represents the main source of fat in the Mediterranean diet and represents a valuable food product from both nutritional and economic point of view. The nutritional value and the health promoting effects of extra virgin olive oil (EVOO) rely on its favorable nutrient composition, including oleic acid as the most abundant fatty acid and fat-soluble vitamins and the presence of phenolic compounds [1–3]. The latter are recognized in contributing to the positive health effects related to the consumption of extra virgin olive oil [4, 5]. Noteworthy, the Regulation 432/2012 of the EU approved the claim “olive oil polyphenols contribute to the protection of blood lipids from oxidative stress” based on the scientific opinion of the European Food Safety Authority (EFSA) that “a daily intake of 20 g of olive oil, which contains at least 5 mg of hydroxytyrosol and its derivatives (e.g., oleuropein and tyrosol) provides the expected beneficial effects” [6, 7]. Tyrosol (Ty) and hydroxytyrosol (Hty) are among the main phenolic compounds found in olive and olive oil occurring in their free form as well as in the esterified forms, mainly as secoiridoid derivatives (ligstroside and oleuropein, respectively) [8, 9] or acylated, as in the case of hydroxytyrosyl acetate [10]. Hty has been reported to display a number of biological activities, including anticancer, antioxidant, and anti-inflammatory properties [11, 12]. Despite its potential health benefits, its uses in food and cosmetic

industries are limited by its hydrophilic character that eventually leads to low bioavailability.

Lipophilization has been suggested as a promising strategy to improve the properties of Hty as well as of other polar phenolics [13]. In order to expand the knowledge on the biological activities of Hty and Ty fatty esters as potential ingredients in functional foods with improved quality, the anti-inflammatory properties of a series of Hty and Ty esters with short (C2), medium (C12) and long (C16 and C18) acyl chains were evaluated. Furthermore, the potential occurrence of tyrosyl oleate (TyOle) and hydroxytyrosyl oleate (HtyOle) was investigated in EVOO, as well as in the by-products of olive oil industry, pomace and olive mill wastewaters. TyOle and HtyOle were found as novel lipophenols in olive oil and in by-products [14, 15]. Interestingly, TyOle was found in higher concentration in defective or low-quality oils than in EVOOs [16], while the identification of HtyOle in EVOO opens a new scenario for the rational use of olive oil in topical formulations. The research activities were partly supported by the project funds “Innovazioni tecnologiche nella filiera dell'olio d'oliva e dell'oliva da mensa - INNOLITEC - (D.M. 37067/7110/2018).

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NMR spectroscopy in extra virgin olive oil authentication

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Extra virgin olive oil (EVOO) is a high quality product with a high economic relevance, especially in Italy, that currently has 43 Protected Denomination of Origins (PDOs) and 4 Protected Geographical Indications (PGIs), the largest set of extra virgin olive oil with recognised Geographical Indications in Europe. In recent years, due to the high price that can be claimed, olive oil has been recognised as one of the foods mainly subjected to food frauds (annual report of the European Union Commission, 2020). EVOO can encounter different types of fraud, from adulteration with cheaper oils to mislabelling of the geographical or botanical origin and the assessment of its authenticity and traceability can be challenging. There are several officially recognized analytical methods based on chromatographic or other analytical techniques for its authentication, but no methods can unambiguously trace back the geographical and botanical origin of EVOOs. NMR (Nuclear Magnetic Resonance) spectroscopy can be a reliable and rapid tool to determine the olive oil metabolomic profile and the quantification of its constituents through both targeted and untargeted approaches. The different adulteration types identifiable with NMR can be divided into three main categories: the adulteration through the addition of oils of different botanical origin, the mislabelling of the cultivar and the mislabelling of the geographical origin. Here, recent progress in the fight against EVOO fraud based on NMR spectroscopy coupled with chemometric analysis are presented. This analytical approach could represent a reliable and robust tool for the traceability of this product.

A harmonized multi-analyte SPME GC-FID or GC-MS method for measuring volatile compounds in virgin olive oil: some evidence from the validation process

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The analysis of specifically selected volatile organic compounds (VOCs), followed by appropriate statistical elaborations, has been identified as a suitable approach to support the panel test in the classification of virgin olive oils (VOOs), according to the quality grade. For this reason, two collaborative actions were carried out during the EU H2020 OLEUM project to deliver a validated and harmonized method for determination of the selected VOCs in VOOs. One of the novelties of this approach is that the minimum number of diagnostic compounds responsible for the positive attribute (fruity) and sensory defects have been selected for the quantification and split into two standard mixtures (SMs) to effectively build the calibration curves by avoiding the need to prepare a single curve for each individual standard, thus abbreviating the analyses, in view of an everyday quality control. The method is based on the isolation and pre-concentration of volatiles molecules by solid-phase micro-extraction (SPME), subsequent separation of analytes by gas chromatography (GC) and quantification by mass spectrometry (MS) or flame ionization detector (FID). The results of the inter-laboratory validation study within select partners of OLEUM project formed the basis for an international validation trial that followed up, with the participation of twenty laboratories from Europe, UK, USA, China, and Japan. Both validation studies allowed to obtain additional information on the performance of the methods (both MS and FID), optimize its applications and having accurate knowledge of errors. Thus, the application of these methods will be fundamental for setting the limits and the concentration ranges expressing the variability of the selected VOCs (especially those related to sensory defects) to put them in relation with the different quality grades of VOOs. In fact, the establishment of limits and ranges for specific VOCs could be useful to

support the panel test and the instrumental analysis could be an effective tool for confirm or disconfirm the sensory classification in case of disagreement between different panels.

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Title of proposed presentation: “Analysis of Volatile Compounds: a potent multi-faced tool for EVOO quality evaluation”

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In recent years, analysis of Volatile Organic Compounds (VOCs) has become one of the most important tools for studying the quality of extra virgin olive oil (EVOO), including its change during storage. Indeed, VOCs strongly influence the sensory properties of the EVOO. They include several groups of molecules: i) those originated from the lipoxygenase pathway, which are usually associated to positive attributes in the literature; ii) several molecules linked to oxidative defects (i.e., rancid); iii) several molecules linked to microbiological defects (e.g., fusty, musty, winey); iv) a large group of volatile hydrocarbons. The study has been carried out thanks to a collaboration between the University of Florence and Carapelli Firenze S.p.A. This presentation is aimed at showing the results of the analysis of VOCs of EVOO and the VOCs related to by-products (i.e., the olive pomace) toward the quality and conservation of the product. The volatile fraction of a large group of virgin olive oils of different geographic and botanical origin has been analyzed using a validated HS-SPME-GC-MS method involving the Multiple Internal Standard Normalization (MISN) quantitative approach.

As for oxidative defects, a number of selected samples was analyzed over storage under different non-accelerated oxidative conditions.

Finally, concerning the defects of microbiological origin, the volatile fraction of olive pomace stored in different oxygen-temperature conditions was analyzed. The obtained results gave important information on:

- The definition of suitable molecular markers of the main sensory defects
- The authentication of the geographical and botanical origin of EVOO
- The conservation of extra virgin olive oils
- The management of by-products for their re-use as a nutraceutical ingredient

The results of our experiment, carried out analyzing oils with very different fatty acid composition (and therefore with different susceptibility to oxidation), indicated that oxidation during storage in different conditions (e.g., in the dark, under light exposure, under light exposure in the presence of high amounts of oxygen) triggers the increase of different groups of VOCs, which in turn can be used as markers to follow such oxidation processes during the storage of EVOO from production to shelf. At the same time, the VOCs originated during storage of olive pomace in different conditions allowed identifying markers related to defects from microbiological origin (e.g., the fusty) in virgin olive oils.

Our results also showed that data from HS-SPME-GC-MS analysis of VOCs in combination with suitable statistics, allows the authentication of the geographic origin of EVOOs from some of the main worldwide

producing countries (i.e., Spain, Italy, Greece, Tunisia, Portugal), and a first characterization of EVOOs of some spread cultivars. Overall, our studies demonstrated the importance of volatile compounds in getting important information on EVOO quality and conservation, as well as their importance for the authentication of EVOO origin. At the same time, the study of olive pomace VOCs allowed getting important information for a suitable managing of this by-product, towards its re-use as a nutraceutical ingredient for human diet.

Med-Index - a food product labelling system to promote adherence to the Mediterranean diet encouraging producers to make healthier and more sustainable food products- the case study of extra virgin olive oil

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Consumers are increasingly demanding transparency in food labelling as they want more and better information about what they are eating and where their food comes from. Several food indexes have been developed in the last decades to promote healthy eating with the aim to reduce certain diseases such as obesity, cancer, diabetes. The Mediterranean diet is known to be one of the healthiest dietary patterns and it is associated with a lower incidence of mortality from all-causes, and it is also related to lower incidence of cardiovascular diseases, type 2 diabetes, certain types of cancer, and neurodegenerative diseases, but a comprehensive index that quantifies the Mediterraneanness of foods is still missing. The real European challenge is to identify a uniform labelling system for the whole of Europe which promotes a healthy lifestyle. A new label system named Mediterranean Index (MI) has been developed with the aim to accurately measure the degree of food Mediterraneanness. The MI simultaneously integrates nutritional and sustainability characteristics of foods. The MI may provide an objective basis for the use of the “Mediterraneanness” label on food products, which can ultimately promote adherence to the Mediterranean diet encouraging producers to make healthier and more sustainable food products.

According to EFSA, the extra virgin olive oil which is promoted for consumption as part of the Mediterranean diet for its beneficial effects against diseases of the cardiovascular system is a product rich in oleic acid, polyphenols and tocopherols. This condition depends on the latitude of the production area, the variety of olives and the agronomic and technological practices, therefore a nutritional label should allow the consumer to recognize with certainty a standard extra virgin oil, that performs a mere function of condiment, from the functional product, effective in reduce the risk of pathologies.

The Med-Index responds to this need in a complete and effective way and is a real food education tool that breaks the information asymmetry and allows to recognize a prize of price for the best extra virgin olive oil.

Study on the time validity of the health claim of polyphenols in extra virgin olive oil.

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Prince of healthy foods since ancient times, extra virgin olive oil was defined by Hippocrates as "the great healer". Recent studies have confirmed that olive oil polyphenols contribute to the protection of blood lipids from oxidative stress, a mechanism of physiological cellular damage in the organism, which can cause, through DNA mutations, the cancer and lead to heart and blood diseases, diabetes and neurodegenerative diseases. Thanks to the accreditation of many scientific studies, the various virtues and qualities attributed to olive oil, have been legally recognized through the definition of specific healthy claims that are allowed to be used on the label, included by the European Commission in Regulation (EU) N. 432/2012 (May 16, 2012) establishing the European Register of nutrition and health claims on food. To date, for extra virgin olive oil it is possible to use 4 claims between nutritional and healthy claims but the most important one refers to the content of polyphenols, among the most valuable components able to determine the fruity aroma, the spicy and bitter taste. The most important property of these molecules, however, is not only organoleptic but also chemical, related to the high antioxidant power that translates into the ability to bind free radicals in our body and resist the oxidation of fat with a consequent increase in the shelf life of the oils. According to the European regulation the benefits of polyphenols become tangible with a daily intake of 20 g of olive oil containing at least 5 mg of hydroxytyrosol and its derivatives. For the operators of the supply chain the knowledge of the polyphenol's content in an oil is essential because allows to properly manage the processing stages in order to maintain a high concentration than preserving the antioxidant capacity. The oil that aspires to carry the claim on the label must have, at the time of bottling, a sufficiently high amount of biophenols to ensure, without risk of non-compliance, the validity of the statement until the expiry date of the product. The claim of polyphenols, although potentially a powerful and innovative marketing tool for the olive oil sector, is still little used by producers and therefore little known among consumers, especially for the lack of knowledge about the evolution of the biophenolic content during the life of extra virgin olive oil. The specific objective of the research is therefore, through chemical analysis, to monitor and evaluate quantitatively and qualitatively the evolution of polyphenols in extra virgin olive oil samples during storage and packaging. The new information acquired will therefore contribute to develop systems to preserve the amount of substances with proven health action in all stages of the production / marketing process and consequently promote the use of the claim for the commercial valorization of oils rich in polyphenols.

The modern oil mill: extraction efficiency, olive oil quality, diversification and sustainability.

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In the last two decades, oil mill modified its structures assuming a more industrial aspect, anyway assuring good yields, olive oil quality and an economic and sustainable utilization of the by-products. The modern oil mill adopts the continue centrifugation system, at two or three phases, and the olive crushing is carried out by the metallic crushers, having different characteristics able to influence oil yield and the organoleptic quality of oil.

The following operation of olive paste malaxation, if carried out for rational time and temperature, favors the coalescence phenomenon, makes “free” most of the oil contained in the cell vacuole and allows to extract up to 85-86% of the oil content of olive fruits, without any negative effect on olive oil quality.

Today, the separation of oil from other phases is obtained by undergoing the olive paste to the centrifugal force produced by the rotation of the decanter at two or three phases. These machines, if loaded with olive paste at 60-75% of their theoretical capacity, allow to obtain the mentioned oil yields. The same decanters have a loading capacity variable between 1 and 15 t/h (and also more) and, therefore, are used either in the medium-small size oil mill either in the big, private or cooperative oil mill at high loading capacity. In these last oil mills, if the double centrifugation is carried out, it is possible to reach an oil yield equal to 87-88% of oil contained in the olives.

The by-products of oil mill, equipped with the centrifugal decanter, are the wastewater and/or the wet olive pomace. In Italy, due to the reduction of the pomace oil prices, the activity of the related industry is difficult for the high operative costs, for the regulations on industrial safety and on the environment protection and for possible residue of new formed chemical compound into the oil. Because of the mentioned reasons, the wet olive pomace is used for:

- the separation of the wooden stone fragments to use as fuel (calorific value > 4000 kcal/kg), with positive effect on the environment because they are obtained from a renewable source of energy;
- the direct burning, after a suitable drying, in the industrial boilers and also in the domestic ones;
- the controlled spreading on olive grove or on cultivated soil;
- mixed with other agricultural residues and treated in the plants for the depuration by the anaerobic digestion, producing biogas also.

Oil mill wastewater, instead, is effectively used by the controlled spreading on cultivated soil, as allowed by the Italian law n. 574 of 1996. In that way, a great amount of mineral substances, having fertilizer properties, and organic matter, useful for the micro-organisms, is supplied to the agricultural soil, avoiding the use of chemical fertilizers and causing a virtuous recycle representing an effective example of sustainable agriculture and circular economy.

The definition of analytical markers of the geographical origin of virgin olive oils based on the evaluation of minor constituents with particular reference to volatile compounds

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Extra virgin olive oil (EVOO) is one of the food products whose origin influences the organoleptic characteristics of the finished product, as well as the appreciation, the perceived quality and the purchase choices of consumers. For these reasons, the Italian legislation (DM n. 8077 of 10.11.2009) and the EU legislation (EU Reg. n. 29/2012 and EU Reg. n. 1151/2012) prescribe the declaration of origin, both for conventional EVOOs and for those subject to specific EU quality schemes, based on forms of documentary traceability according to EU regulation n. 178/2002.

However, this type of traceability is restrictive and excessively conditioned by the degree of accuracy and integrity of those who implement it. Therefore, it would be desirable to implement forms of analytical traceability that are able to corroborate the regulatory requirements.

To this purpose, this experimentation aims to achieve an authentication of the geographical origin of EVOOs based on the headspace volatile compounds. It's well known that the profile of the molecules responsible for the flavour of EVOOs is also affected by the soil and climatic conditions of the place of cultivation, so these substances can serve as markers able to discriminate EVOOs according to their different origins.

The research activity was carried out on 77 EVOO samples, belonging to the milling campaign 2020/2021 and selected in order to provide an overall view of the different origins of the EVOOs available on the market. The analysis of volatile compounds was carried out, in comparison, by HS-SPME-GC-MS (headspace - solid phase microextraction - gas chromatography - mass spectrometry), a well-known, proven and solvent-free technique, and by ion mobility spectrometry (HS-GC-IMS), using a *Flavourspec*[®] instrument. Ion mobility spectrometry (IMS), combined with gas chromatography (GC), is an innovative and fast analytical technique requiring little or no sample preparation, which can provide a fingerprint of volatile compounds from the EVOOs headspace. The result of the analysis is a three-dimensional chromatogram (GC retention time vs. IMS drift time), in which each individual spot corresponds to a volatile compound, the concentration of which is proportional to the intensity of the red colour of the spot itself.

The data collected were processed through the techniques of multivariate statistical analysis, which allowed the development of several models, where all the different spots of each of the 3D chromatograms, relating to the samples under analysis, were taken into consideration.

The results obtained from the *Flavourspec*[®] allowed discriminating samples according to their different geographical origin; the data derived from HS-SPME-GC-MS substantially confirmed the results of HS-GC-IMS.

The *Flavourspec*[®] has proven to be an easy-to-use instrument, able to quickly provide accurate results. These strengths could become interesting for private companies that need to perform screening analysis to verify the geographical origin of their EVOOs.

FlavourSpec® & Machine learning: le nuove frontiere nel supporto strumentale all'analisi sensoriale degli oli vergini di oliva

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The FlavourSpec® is an analytical instrument of G.A.S. Dortmund, it uses gas chromatography and an ion mobility spectrometer (IMS) as a detector. The separation and identification of odorous molecules occurs directly from the headspace without the use of any adsorbent fibers, as occurs in the solid phase extraction technique (SPME).

IMS is a chemical-physical separation technique in which the odorous molecules suitably ionized are separated in a neutral gas phase (pure nitrogen) at atmospheric pressure.

The separation of the ions occurs under the action of an electric field and is the result of the different mobility of the ions in the drift cell: The drift time is the time required by the ions to travel through the drift cell and is related to the applied electric field, to the mass of the ion and its coefficient of friction, as well as other factors. Gas chromatography and ion mobility detection are separation methods based on totally different principles and therefore, they are perfectly orthogonal, their coupling gives rise to an analytical technique (GC-IMS) capable of being used both for research and for the management of quality control processes in the industrial sector.

The purpose of this report is to present the applicative potential of the technique in a olive oil supply chain through the digital control of production.

The analytical result of the FlavourSpec® consists of three-dimensional "images" ("raw data") that give an immediate understanding of the characteristics of a sample of virgin olive oil; in the operative practice two-dimensional chromatograms with "pseudocolors" are then used to identify the different signal intensities.

The approach can be twofold: "targeted", if the individual odorous molecules corresponding to the three-dimensional peaks of the extracted image are identified; or "untargeted" if the raw matrices are processed as if they were undifferentiated fingerprints of the virgin olive oil sample analyzed.

Both classical chemometric techniques and machine learning with deep neural network techniques were applied to the two sets of data thus extracted for the purpose of verification and comparison.

The samples of virgin olive oil analyzed belong to the three product classes defined in the E.U., this classification was decided by the application of sensory evaluation according to the COI / T20 / Doc method. 15 Rev.10. In order to verify the correspondence between the classification derived from the application of the sensory methodology and that, instead, determined by the statistical processing of the data derived from the FlavourSpec®, three different classification approaches were applied simultaneously:

the supervised one, the unsupervised one and the semi-supervised one. The different approaches were compared directly in order to analyze and understand the three fundamental factors:

- type of data approach: non-targeted vs targeted;
- type of data analysis: chemometric/statistical vs machine learning;
- type of approach to classification: supervised, unsupervised, semi-supervised.

Shelf-life predictive model study of bottled extra virgin olive oil based on head space aromatic molecules analysis

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Sampling

The bottles of Mediterranean blend extra virgin olive oil have been selected from different production batches and stored in closed cartons of 6 bottles each in a room with temperatures ranging from 64.4-71.6°F for 20 months. Every 4 months they were sent to the analytical laboratory (Coteca S.r.l. Acc.1944). Also, a strong point, periodically, according to availability, samples of bottles deriving from monitoring from shelf (MFS) carried out in the United States were added.

Instrumental and sensory analysis.

In the chemical laboratory the odorous molecules of the headspace were determined according to the consolidated HS-SPME-GC/MS technique, while the sensory profile and the product classification were determined by the panels (COI recognized).

Assessment

The data obtained were pre-processed and evaluated in Python according to the untargeted modality, using different machine learning and deep neural network techniques according to the classic sequence: training (70%), test (30%), evaluation (MFS samples) in order to fine tuning of the model parameters. By applying, for example, Random forest Regression together with SMOTE (Synthetic Minority Oversampling Technique), an accuracy of over 95% was achieved (R Square equal to 0.97 in_train and 0.84 in_test with MAE of 0.6).

Organic and new processing technologies and quality control analysis of extra virgin olive oil with multivariate approach

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The production and consumption of olive oil has been increasing in recent years. The competitiveness of olive oil industry depends not only to olive production, interest in the processing industry and commerce but also quality and health benefits of the oil. Turkey is the world's fourth-largest olive oil-producing nation will yield 235,700 tons of olive oil production for 2021/22 crop year. Organic olive oils are usually extra virgin or virgin olive oil, products with exceptional taste and high nutritional value protecting consumers health and consumers to be convinced that organically grown foods are safer and provide greater health benefits than conventional counterparts. Olive oil is preferred by consumers because of being rich in bioactive components, beneficial effect for health and high nutritional value. The production process of extra virgin and virgin olive oil, obtained only by mechanical or other physical methods from olive fruit. New processing strategies have been developed to remove the negative effects of conventional olive oil processing methods that includes converting the traditional malaxation batch process into a continuous one in order to reduce preheating phase and time and optimize the phenolic and volatile compounds related to EVOO healthy and sensory properties, different technological solutions can be adopted during olive paste conditioning: microwave energy (MW), pulsed electric field (PEF), ultrasounds (US). Thus new technologies will improve the quality, physico-chemical and nutritional properties of oils and reduce the processing time and energy consumed during extraction compared to traditional methods toward greater economic growth in the olive oil industry. The top two qualities of olive oil are the extra-virgin and the virgin olive oil on their unique physical, chemical and sensorial parameters. Reference methods that are used to determine fatty acids, tocopherols, carotenoid etc. takes long time, uses expensive and sometimes toxic chemicals, needs analyst expertise, needs sample preparation, can not be applied on-line, at-line and off-line and they may have certain limitations, restricting its use for accurate and complete quality control purposes. New analytical procedures with chemometric analysis including vibrational spectroscopic techniques (NIR, MIR, FT-NIR (Attenuated Total Reflection Fourier Transfer Infrared (ATR-FTIR) and Raman scattering), Nuclear magnetic resonance (NMR) spectroscopy and mass spectrometry with chromatographic techniques (HPLC, GC) have been developed recently. They have become popular since they provide many practical advantages, such as providing rapid, accurate, non-destructive and simultaneous analysis and allows online, off-line and at-line detection of quality parameters and widely applied in the quality control of virgin olive oils. Multivariate analysis, such as partial least squares regression, discriminant analysis (PLS-R, PLS-DA) and principal component analysis (PCA) methods have been frequently developed for rapid and online detection systems for mainly virgin olive oil quality, safety and assurance. Thus chemical and sensorial properties, authenticity identification, geographic origin, adulteration detection, quality parameters detection of extra virgin and virgin olive oils can be done rapidly, accurate, and due to its non-invasive and simple sample preparation make it a very valuable analytical method. On-line spectroscopic detection is practical for monitoring the quality of industrial virgin olive oil production line.

Keywords: Extra virgin olive oil, Conventional, Organic and Non-thermal olive oil processing Techniques, high yield, quality, new technologies, new analytical procedures, FT-NIR, NMR, Raman Scattering, HPLC-MS, quality control, safety, in, on and off-line quality monitoring, Chemometric Analysis.

HS-GC-IMS and SPME-GC-FID: screening and targeted methods to classify virgin olive oils, in support to the panel test, by exploiting the volatile fraction

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In virgin olive oil, the volatile compounds (VOCs) are responsible for positive (fruity and peculiar aromatic notes) and negative attributes (main defects and others), that are identified and quantified by sensory evaluation; the results obtained from the application of this methodology, together to some titrimetric and instrumental ones, permit to define the belonging of a sample to a specific quality grade (extra virgin EVOOs, virgin VOOs, and lampante olive oils LOOs) according to the European Union regulation. The official sensory analysis of virgin olive oils (Panel test) has some weaknesses concerning the limited number of samples that can be evaluated by assessors in each tasting session and possible disputes due to disagreement among panels. In this context, the research aims at developing diagnostic strategies to combine sensory and instrumental results, i.e. obtained through qualitative and quantitative analysis of volatile compounds (VOCs). In this work two analytical methodologies for VOCs analysis have been considered: i) gas chromatography coupled with ion mobility spectrometry (HS-GC-IMS), as a rapid "semi-targeted" screening tool based on selected volatile compounds to support the Panel test in prioritizing/pre-classifying samples; ii) solid phase micro-extraction (SPME) and subsequent gas chromatographic (GC) analysis with flame ion detector (FID), aimed at the identification and quantification of selected highly diagnostic volatile compounds, as related to the fruitiness and the main sensory defects (targeted analysis), for purposing limits and ranges as quality indexes.

Sixty virgin olive oil samples were selected and collected in a balanced number according to their quality grade. For each sample, 4 aliquots of 500 mL were prepared and shipped to 4 officially recognized panels of public entities (ICQRF – Laboratorio di Perugia, Alma Mater Studiorum - Università di Bologna, Agenzia delle Dogane e dei Monopoli di Roma and Agenzia delle Dogane e dei Monopoli di Bari). The sensory data obtained by each panel were processed by applying the "decision tree" developed within the H2020 OLEUM project, which is based on the agreement of more than 50% of the involved panels (3 out of 4 in this case), both on the intensity of the most perceived defect and/or fruitiness, to reach a shared and reliable quality classification.

In parallel with the official sensory evaluation, the headspace of the 60 selected samples was analysed by HS-GC-IMS (Flavourspec) and SPME-GC-FID. By processing the heat maps (3D chromatograms) obtained by HS-GC-IMS analysis and the results of the robust sensory classification, it was possible to build a PLS-DA classification model. The results obtained were satisfactory in terms of percentage of correctly classified samples: 83.3% for EVOOs, 84.2% for VOOs and 100% for LOOs.

Finally, for each sample the predicted commercial category by HS-GC-IMS was compared with that coming from SPME-GC-FID results in which 18 particularly relevant volatile compounds were identified and

quantified, then considered according to limits and ranges previously established on a wider set of samples (in the H2020 EU OLEUM project).

The dosage of volatile compounds by SPME-GC-FID can be, therefore, enhanced as a complementary analytical method, useful and supportive to the Panel test especially in case of disagreement between panels in the evaluation of a sample. Keeping in mind this purpose, it is essential to study in-depth limits and ranges for the selected volatile compounds by analysing more samples. HS-GC-IMS can be exploited as a rapid screening method for the prioritization of virgin olive oil samples to be sensory analysed by Panel test.

Development of rapid and non-destructive methods for determining the chemical composition and label compliance of extra virgin olive oil

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Monitoring food quality requires accurate methods of analysis, that can be extensively applied with the opportunity to analyze a large number of samples in a short time. Methods that are accurate fast and at a reasonable cost can be effectively implemented for preserving the integrity of the products and for quickly detecting fraud on the nutritional composition.

In this regard, the Near Infrared Reflectance Spectroscopy (NIRS) technology possesses all the listed characteristics and can be used for both qualitative and quantitative chemical analysis of EVOO (Extra Virgin Olive Oils) with possibility of simultaneously quantifying the concentration of different parameters without the use of chemical reagents (which are nowadays required to conduct reference analysis on EVOO).

The goal of this project is the development of calibration and methods for NIRS analysis for quantitative determination of the main quality parameters of EVOOs within one spectroscopic analysis at the same time. The quality parameters chosen for the first stage of the project are: acidity, peroxide value, spectroscopic indexes (K232 / K268).

EVO oils are analyzed in special vials for the spectroscopic analysis of liquids without any pretreatment. To ensure better analytical performance and standardization each sample was heated at 45°C for 4 minutes, placed in the instrument scanning chamber before spectra acquisition.

Fourier transform spectrophotometer FT-NIR Multi-Purpose Analyzer (Bruker Corporation, Billerica, Massachusetts) was used together *OPUS* software for data acquisition, spectra processing and development and validation of calibration models.

The validation of the calibrations takes place through the evaluation of the prediction model statistical quality indexes for each parameter, using laboratory uncertainty as target for the prediction error.

Preliminary results of calibration performances for predicting acidity and peroxide value, yielded fairly high R² coefficients (0.98 for acidity; 0.95 for peroxide value).

Future work will be focused on the development of proper methods for the evaluation of other important olive oil characteristics such as fatty acid composition, and other minor components (sterols, stigmastadienes, etc.).

Geographical characterization of virgin olive oils by headspace volatile compounds analysis (HS-SPME-GC-MS).

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Given the growing demand by consumers of high quality and authenticity extra virgin olive oils (EVOOs) and considering the fact that the European Union also pays particular attention to this problem, the determination of the geographical origin of EVOOs is a very important factor that could guide consumers' purchasing choices and help ensure the consumption of high quality EVOO; for this reason, it is necessary to develop analytical methods that are able to establish their origin.

In recent years, various methods have been developed in this regard and those inherent to the evaluation of volatile compounds appear to be among the most promising ones. However, a unique solution has not yet been identified, also given the great variability that these compounds show due to genetic, agronomic, pedoclimatic, technological, and conservation factors.

In the present study the headspaces, including both the volatile compounds deriving from the lipoxygenase pathway and the terpenes and hydrocarbons, of 221 Italian EVOOs and 84 foreign EVOOs of two different years were analysed.

From the results obtained by means of multivariate statistical analysis (using the PLS-DA, OPLS-DA methods and the SIMCA class modelling) it can be seen that the analysis of the volatile component is able to give excellent results as it is possible to reclassify in a correct way almost all the olive oil samples in the respective production areas even when simultaneously samples of EVOO from different years are considered. Therefore, the study of volatile compounds could be a valid tool for the discrimination of the geographical origin of olive oils, potentially able to overcome even the annual variability. Another consideration that can be made based on the results obtained is that by evaluating only the terpene compounds the results are more than satisfactory, however when the compounds originating from the lipoxygenase pathway and the hydrocarbons are also included in the modelling, the separation between the production areas improves significantly as well as the correct reclassification.

Considering the models with all the samples of the two years and all the variables, 94% of correct attribution was obtained according to the geographical origin in the model in which the oil samples were grouped according to the origin area and over 98% of the exact reclassification was achieved between the Italian EVOOs and those of foreign origin in the model in which these samples were divided into two classes (Italian and foreign).

Correlation between the volatile compounds and the organoleptic characteristics of Extra Virgin Olive Oils

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At the end of 2018, the Italian Association of the Oil Industry (ASSITOL) promoted a Task Force, between associated companies and chemical laboratories, to start a study on the correlation between the content of volatile compounds present in virgin olive oils and the analysis of panel test, due to the pandemic, in 2020 the work was interrupted and resumed in 2021 to improve the first results obtained.

Five companies have participated in the Task Force (Carapelli Firenze Spa, Colavita Spa, Costa d'Oro Spa, Oleificio RM Srl and Salov Spa), the three professional panels of ASSITOL (c / o: Costa d'Oro Spa, Carapelli Spa and Agridè Srl) and initially four chemical laboratories (Carapelli Company Lab, Chemiservice Srl, Coteca Srl and Soremartec Company Lab) one of which interrupted the collaboration after the first phase of the study.

Taking inspiration from what was present in literature and especially from the work of M. Fortini, M. Migliorini, C. Cherubini, L. Cecchi, L. Calamai; "Multiple internal standard normalization for improving HS-SPME-GC-MS quantitation in Virgin Olive Oil Volatile Organic Compounds (VOO-VOCs) profile" [Talanta 165 (2017) 641-652], an analytical system has been developed in SPME / GC / MS, for the quantification of volatile compounds in virgin olive oils.

The analysis in SPME can have some issues, due to the competitions between the various analytes absorbed on the fiber, in function of the quantity present in the oils and their different polarity, so is necessary that the laboratories use the same conditioning system of the fiber to obtain a acceptable reproducibility.

Initially, we have chosen 12 markers representative of the positive organoleptic characteristics and 26 markers for the 4 predominant defects of virgin olive: Vinegary (Av), Rancid (Ra), Fusty/Muddy (Ri) and Musty (Mu) using 9 internal standards for the quantification of the markers.

Subsequently, after the analysis of Extra Virgin Olive Oils (EVOO) and Virgin Olive Oils (VOO), the markers were reduced to 27, of which 6 for positive attributes and 21 for the defects, using 3 internal standards (4-Methyl-pentan-2-ol; Octan-3-one, 3,4-Dimethyl-phenol).

A total of 46 samples were analyzed, of which 31 declared EVOO and 15 VOO, for the panel test we have used the three ASSITOL professional panel and the internal panel of the Task Force companies.

Eliminated the results don't congruent between the volatile and the panel analysis and using EVOO without defects it was possible to establish a limit threshold for the defects associated with the markers.

Using oils with defects we have found a linear regression between the median obtained with the panel test and the ratio between the content of the markers and their threshold.

The system studied of the volatile analysis on blind samples showed a greater reproducibility than the results obtained with the professional and official panels.

An ¹H NMR-Chemometric model for the classification of Italian extra virgin olive oils

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Italian EVOOs are “peculiar” and “prestigious” products that have to be protected against the constant risk of adulteration or mislabeling. Although EU Regulation 182/2011 requires mandatory labelling reporting the geographical origin of EVOOs, the actual official quality control methods are unable to verify their real geographical origin. NMR spectroscopy, combined with multivariate statistical analysis, has shown to be an important tool in the geographical olive oil characterization.

In this study, extra-virgin olive oils (383 samples; EVOOs) of three consecutive harvesting years from nine Italian regions were collected and submitted to an ¹H NMR-chemometric protocol to characterize the samples according to their origin (geographical area and variety). A more complete assignment of the olive oil ¹H spectrum in CDCl₃ and DMSO-d₆ was reported identifying 24-methylencycloartanol [1]. A single classification model provided the discrimination of EVOOs among the three geographical macro-areas (North, Islands, Center-South), whereas a hierarchical approach based on breaking the overall classification problem into a series of smaller linear discriminant analysis (LDA) sub-models was tested to differentiate olive oils according to their geographical regions. Specific compounds responsible for olive oil characterization were identified.

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High vacuum technology applied to mechanical extraction process of virgin olive oil.

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The high vacuum technology was applied to the virgin olive oil (VOO) mechanical extraction process to evaluate its performance as a new system of assisted extraction process. The new extraction method, used during the malaxation step, was preliminary tested with a lab-scale plant to assess the chemico-physical characteristics of olive paste and VOO. Successively, the studies concerned the application of high vacuum technology at industrial scale to evaluate the impact on oil extractability and quality characteristics responsible of the main VOO health and sensory properties. Lab-scale trials showed significant changes of the cellular structure of the olive paste, improving the coalescence of the oil droplets evaluated by cryo-scanning electron microscopy (Cryo-SEM). The treatment under vacuum condition determined an increase of phenolic concentration and a stripping phenomenon of volatile compounds that was influenced by malaxation temperature. Industrial-scale trials, which were carried out by different extraction plants with a working capacity ranged from 2.5 and 4 ton/h, showed a significant effect on oil yield, also confirming a positive impact on phenolic fraction. The use of lower temperatures and selected pressures were also able to reduce the losses of volatile compounds. The decrease of volatile fraction did not concern only the molecules responsible of VOO flavor but also other compounds with a low molecular weight such as ethanol, 3-methyl-1-butanol, ethyl acetate and acetic acid. The stripping process of these molecules determined an improvement of VOO sensory notes, reducing potential alterations of oil quality characteristics during storage and shelf-life.

Analytical

Automation of sample preparation in olive oil analysis

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The commodity characterization of olive oils, mandatory at EU level, involves the evaluation of numerous parameters aimed at determining the class to which a given oil belongs.

Many of the required analyses involve a long sample preparation characterized by the use of large volumes of solvents and consumables, as well as massive operator intervention.

SRA instruments has developed a range of automations, based on the concept of green chemistry, aimed at determining Alkyl esters, Waxes, Sterols, Alcohols and Stigmastadienes. Such automations allow to:

- Drastically reduce, in some cases up to a tenth, the consumption of organic solvents
- Almost completely eliminate the use of consumables
- Hugely limit the operator's intervention
- Obtain a drastic reduction in analysis times
- Guarantee greater reliability of the analytical data

Comparison of three different methodologies for the quantification of hydroxytyrosol and tyrosol in olive oils, in relation to the health claim

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Hydroxytyrosol (HYTY) and tyrosol (TY) are phenyl ethyl alcohols naturally present in *Olea Europaea* L. fruits, leaves and oil as free, but especially as bounded, derivatives belonging to the chemical class of secoiridoids (e.g. oleuropein and ligstroside). These compounds are particularly interesting because they have the property "to act as an inhibitor of the oxidative damage of LDL". Their determination in olive oil, in particular the quantification of total HYTY and TY is important to support the health claim for 'olive oil polyphenols', according to EU regulation 432/2012, in which it is requested to quantify "hydroxytyrosol and its derivatives (e.g. oleuropein complex and tyrosol)" to verify their presence in a concentration of at least 5 mg per 20 g of oil.

There is a great interest by all actors (producers, bottlers, distributors and consumers) of the olive oil supply chain to report, when applicable, this claim on the labels to highlight the olive oil healthy properties. Despite the several analytical approaches proposed in literature, at now a method to be used for verifying the compliance with the labelled abovementioned health claim has not been officially adopted yet. The availability of an international standardized method is therefore of utmost interest.

In this sense, this research work is focused on the comparison of three different analytical procedures, already published in the scientific literature, validated or approved in the ISO framework, for the determination of HYTY and TY according to the EU regulation 432/2012.

To carry out this study, six different olive oil samples, characterized by different content of phenolic compounds, have been selected and analysed, applying the three methodologies cited before.

These protocols are based on a chromatographic separation of the fraction of interest (HPLC or UHPLC) but differ for the treatment of the oil sample, specifically: in one case an acidic hydrolytic step was applied directly to the oil, in order to obtain a fraction containing HYTY and TY (resulting by the hydrolysis of the secoiridoids), in the other two cases the phenolic fraction was previously extracted, applying a liquid-liquid procedure, before the acidic hydrolysis. For the identification and quantification, standards of the compounds of interest were used. The obtained results highlight the need of using suitable correction factors for a proper quantification

of the polyphenols, when free HYTY and TY are quantified, to correctly answer to the specific requirement of the health claim.

The main advantages and limitations of these methods, also in terms of time of analysis and use of solvents, have been considered and discussed.

Solvent-saving sample preparation for high-sensitivity determination of MOSH and MOAH in vegetable oils

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Mineral oils (MOH) are complex mixtures of hydrocarbons (thousands of isomers) of petrogenic origin which can contaminate edible oils through different routes. MOH can be divided into two main classes: mineral oil saturated (MOSH) and aromatic hydrocarbons (MOAH), of different toxicological concern.

Although a precise definition of their toxicity is lacking, for the MOAH the request for a limit of 0.5 mg/kg has been repeatedly expressed through draft ordinances or benchmark levels, as often requested by the large-scale retail trade. This limit cannot be reached by direct analysis, which involves the use of liquid chromatography (LC) coupled with gas chromatography (GC) equipped with flame ionization detection (FID), but only after proper sample pre-treatment. For the MOSH the required limits are generally higher (around 2 ppm), but the evaluation of background levels implies in some cases the need to increase the analytical sensitivity also for them.

Specifically, in vegetable oils the sensitivity is limited by two main factors: the triglycerides, which limit to 20 mg the amount of sample which can be injected into the LC column, and endogenous interferents covering and/or overloading the signal of interest (i.e. olefins for the MOAH and n-alkanes for the MOSH). Triglycerides are commonly removed by saponification, while olefins are generally removed by epoxidation. Differently, interference by biogenic n-alkanes does not always exclude the possibility to perform a reliable quantification of MOSH,

e.g. signal overload can be avoided reducing the amount of sample injected, to reach the right compromise between sensitivity and resolution. However, when this is not possible, as in the case of oils particularly rich in endogenous n-alkanes and/or showing low level contaminations, they need to be removed by adsorption on activated aluminium oxide (Alox). The choice of the analyst cannot be made a priori, but needs to be made after a first evaluation of the sample, in order to avoid unnecessary steps that only increase the uncertainty related to the results.

With this contribution we report the development and optimization of an intra-lab validated protocol involving microwave assisted saponification (MAS) followed by epoxidation for MOAH determination in vegetable oils (at LOQ around 0.5 mg/kg). The importance to check recovery during the saponification step and to opportunely choose the internal standard for quantification, will be discussed. Concerning the MOSH, different Alox protocols performed directly on the oil or on the sample after MAS and epoxidation, will be compared, and advantages and disadvantages will be discussed to better guide the analyst towards a conscious choice aimed at minimizing sample handling and solvent consumption.

Analysis of triglycerides, cholesterol, wax esters and sterol esters in foods by means of Restek 65-TG HT capillary column and analysis of fatty acids as phenethyl esters by means of HPLC

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In the food sector, for quality control, fraud detection and authentication of butter fat and other oils and fats, three methods have been developed, one for triglyceride analysis and two for fatty acids analysis. The various analytical methods, based on high resolution chromatographic techniques, have been applied to the study of the composition of different food matrices with one composition mainly of triglycerides (butter, olive oil, lard) [1]. In addition, it is also analyzed the minor lipid component contained in other types of food, such as flour and semolina. In these foods wax and sterol esters were analyzed to detect their lipid composition. For the determination of the composition of fatty acids in food matrices with composition mainly of triglycerides (fats and oils), two innovative methods of transesterification of triglycerides in the pentyl and phenethyl esters of fatty acids were set up. Conversion to pentyl esters of fatty acids avoids the loss of butyric acid compared to when sampling fatty acids are derivatized as methyl esters of fatty acids. Finally, in the HPLC analysis the conversion of fatty acids in the phenethyl esters allowed to separate the unsaturated fatty acids without thermal degradation compared to GC analysis. Also, using the same capillary (RTX 65-TG) with 65% phenyl-35% methyl silicone stationary phase, a method is presented based on gas chromatographic analyzes for the quantification of cholesterol content in eggs [2] and the determination of cholesterol content in egg-pasta to establish total number of eggs added to semolina [3]. Wax esters and sterol esters were determined in olive oil; this procedure was proposed as an alternative method to the procedure proposed by EEC Regulation 183/93 and by using RTX 65-TG capillary column separation of analytes was improved in respect of capillary column 5% phenyl methylsilicone [4].

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Advancing MOSH/MOAH analysis towards speciation and contaminants identification

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Food contamination attributed to mineral oil (MO), revealed by the presence of saturated (MOSH) and aromatic (MOAH) hydrocarbons in various food products, has been for years at the center of attention for its potential impact on consumers' health. In particular the presence of MOAH is correlated to increased risks in terms of toxicity due to their suspect carcinogenicity and genotoxicity, especially when species with 3 or more aromatic rings and low alkylation degree are present.

Current instrumental platforms, based on the LC-GC-FID hyphenation, allow individual quantification of the aliphatic and aromatic contaminant fractions. Nevertheless, the task is often challenging due to matrix complexity and the presence of interferences. Moreover, FID detection does not permit to obtain qualitative information about the type of MOSH or MOAH present, the occurrence of synthetic hydrocarbons such as polyolefins (POH) and polyalphaolefins (PAO) or hydrocarbons of endogenous origin (terpenes, olefins residues, ecc.).

Therefore laboratories tasked with performing MOSH/MOAH analysis need, for samples positive to contamination, access to advanced, more insightful investigation tools for improved characterization of both fractions.

This contribution presents the development and optimization of a platform for MOSH/MOAH analysis based on a preliminary HPLC separation followed by two-dimensional comprehensive gas chromatography (GC×GC) in combination with high resolution mass spectrometry. This solution significantly increases the capacity to characterize the two fractions for a more detailed classification of the hydrocarbon profiles. A real-life case study is shown to highlight the value added by this technique for a confident identification of contamination origin in extra-virgin olive oil.

Phenolic analysis and *in vitro* biological activity of pomace and grape seeds oil derived from *Vitis vinifera* L. cv. Montepulciano d'Abruzzo

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Grape pomace is commonly considered a waste product of monovarietal red wine production. Methods: HPLC-DAD analysis was performed to determine the polyphenol and flavonoid contents of all the extracts obtained from Montepulciano d'Abruzzo red wine and grape skins whereas, GC-MS was applied to the determination of fatty acid composition in grape seeds oil. Biological characterization involves antioxidant and antimicrobial assays for all the extracts and seeds oil; Their ability to inhibit α -glucosidase, α -amylase, α -tyrosinase, and ChE enzymes was also detected, together with anti-inflammatory activity on wine, grape skin extracts, and seeds oil by lipoxigenase (5-LOX) and LPS-stimulated macrophage release assays. Data indicate significant polyphenols content (199.31 ± 7.21 mgGAE/g), antioxidant (CUPRAC assay (1036.98 mgTE/g)), enzymatic inhibition (α -tyrosinase: 151.30 ± 1.20 mgKAE/g) and anti-inflammatory activities for wine-organic extract 2, while the antimicrobial activity of grape skin decoction is higher than those reported by wine extracts on three bacterial strains. Interestingly only dealcoholized wine and wine-aqueous extract exerts inhibitory effects on α -glucosidase (20.62 ± 0.23 mmolACAE/g and 19.81 ± 0.03 mmolACAE/g, respectively), while seeds oil is rich in oleic and linoleic acids. These results confirm the strong antioxidant properties of Montepulciano d'Abruzzo grape pomace, suggesting the potential use of this waste product as functional food supplements in the human diet and in cosmeceutics.

Technology

Riduction of 3-MCPD and GE contaminants present in edible oils

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Many refined vegetable oils, especially palm and palm kernel oils, contain a relatively high amount of 3-MCPD (3-monochloropropanediol) and GE (fatty acid glycidyl esters). These contaminants are commonly defined as process contaminants as their formation occurs mainly during the Refining phase of the vegetable raw material, in particular when temperatures are above 200 ° C.

EFSA, the European Food Safety Authority, has established through various scientific research that these substances can be harmful to health, especially in the case of newborns fed with high doses of infant formula. The European Union has therefore determined, with subsequent interventions, stringent limits regarding the amount of 3-MCPD and GE allowed in vegetable oils and derived food products, and set the entry into force of these limits in January 2021. The aim of this presentation is to illustrate the changes and new technologies that will have to be applied to existing and future refining plants in order to ensure the minimum levels of 3-MCPD and GE required by the EU.

3-MCPD esters and GE have different chemical and physical characteristics and do not have the same mechanism of formation. Hence, different mitigation strategies are required to achieve required low levels in refined food oils.

GE are mainly formed from diglycerides at high temperature (>220°C). This explains the high GE content in standard refined palm oil.

3-MCPD esters can be formed by reaction of triglycerides (TAG) with chlorine (precursors) at temperatures >140°C. Hence, removing the chlorine precursors and/or avoiding acidic conditions during the refining process are the most effective mitigation strategies.

TECHNOIOLOGY has successfully overcome the technical problems related to the reduction of 3-MCPD and GE by developing cutting-edge technology - currently under patent reviewing - which involves the installation of some machinery within new plants or existing plants.

It is worthy to note that although 3-MCPD and GE are primarily produced during Deodorizing, mitigation measures can be applied across the edible oil production chain, from agricultural practices (such as cultivation, harvesting, transporting and storing of oil fruits and seeds), to oil milling and refining (crude oil production and treatment, Degumming/Bleaching and Deodorizing), as well as to post-refining measures (additional Bleaching and Deodorizing and the use of activated bleaching earth).

The impact of polyphenol content and gelator type on the structure of extra virgin olive oil-based oleogels

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Extra virgin olive oil (EVOO) is well known to provide several health benefits, due to its balanced fatty acid profile and to the presence of minor bioactive components, such as polyphenols and tocopherols. EVOO application as a functional ingredient in food formulations would be thus particularly interesting not only to replace high-saturated plastic fats, but also to further enhance the health-promoting properties of food by delivering bioactive components.

However, the direct addition of EVOO into food formulation is challenging due to its liquid state at ambient temperature. Thus, its conversion through oleogelation into a solid-like material to mimic the functionalities of plastic fats could enlarge the possible applications of EVOO.

Oleogelation has been increasingly applied in recent years to modify the physical properties of oils without involving any chemical modification. It relies on the ability of certain molecules, called oleogelators, to self-assemble into a network able to physically entrap the liquid oil. Up to now, different oleogelators and oleogelation strategies have been proposed in the literature. However, the oil type and in particular its composition could greatly impact the final oleogel structure, not only because of its fatty acid profile, but also due to the presence of minor components.

The aim of this research was to study the ability of different gelators to turn liquid EVOO into an oleogel, while understanding the role of polyphenols on the oleogel structure.

To this purpose, three EVOO samples with increasing polyphenol content were obtained by selectively removing polyphenols from a freshly produced EVOO. Four well-known oleogelators, i.e., monoglycerides (MG), rice waxes (RW), sunflower waxes (SW) and a

mixture of α -sitosterol and γ -oryzanol (PS) were then used at 10% (w/w) concentration to gel the EVOOs. The structural characteristics of oleogels were assessed by different methodologies, including visual appearance, rheology, polarized light microscopy, calorimetry and FTIR.

Independently from the polyphenol content, all oleogelators demonstrated a good capacity to gel EVOO, resulting in self-standing materials with a pseudo-plastic behavior. However, the oleogelator type significantly affected the oleogel strength. In particular, the PS-based oleogels presented the highest rheological parameters followed by SW, RW and MG ones. This behavior has been attributed to the different network features: PS mixture assembled into hollow tubes embedding oil, whereas MG, SW and RW formed crystalline networks characterized by peculiar crystal morphologies depending on the molecular properties of the oleogelator.

The presence of polyphenols differently affected oleogel characteristics depending on the network features. In particular, the structure of the PS-based oleogels was not influenced by the level of these minor components, whereas their presence resulted in a significant reinforcement of the crystalline networks (MG, SW and RW). It is likely that polyphenols contributed to network structure by inducing additive interactions among crystals, as confirmed by FTIR analysis.

In conclusion, turning EVOO into oleogels could represent a profitable strategy to obtain novel functional ingredients also characterized by different and tunable technological features. Interestingly, the polyphenol content played a key role in oleogelation, by steering the crystalline interactions and finally the oleogel structure.

Functional oils through enrichment of olive oils and refined seed oils treated with vegetation water

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One of the main strengths of extra virgin olive oil (EVO), compared to other edible oils, is its content in polar phenolic substances, which also strongly contribute to its oxidative stability, health properties and sensory profile. The vegetation water, a by-product in the production of EVO, is very rich in these substances which therefore make this “waste” a precious source of bioactive substances. The idea of this study is to use the vegetation water to enrich refined seed and olive oils in phenolic substances, thus developing functional foods. Refined peanut oil, sunflower oil and olive oil were treated with vegetation water obtained from the production of an EVO, also taken into consideration in the study (reference EVO) and showing high quality, both considering sensory characteristics and chemical parameters. Two treatments of the oils with vegetation water were compared. In one case, the oils were shaken with vegetation water (2 min for ten times, with 10 min intervals). In the other one, the oils with vegetation water were sonicated for 20 min, in a laboratory ultrasonic bath (Sonomatic S0475, 40 KHz). The ratio oil/vegetation water was 1/1 (vol/vol), in both the treatments. After the treatment, the oils were separated from vegetation water by centrifuge (5000 rpm, 10 min). Analyses were performed before and after the treatment to verify the variation in the quantity and composition of the polar phenolic substances, the total phenolic content (TPC), as well as other important components in the definition of the quality of an oil, such as volatile substances. The polar phenolic substances were analyzed through high performance liquid chromatography coupled with a diode array detector and mass spectrometer (ion trap) [1,2], the TPC by the Folin Ciocalteu method, and the volatile substances through solid phase microextraction followed by gas chromatography coupled to mass spectrometry. The results showed a significant enrichment of phenolic substances in the oils. Their level before the shaking treatment was 12.0 mg gallic acid equivalents (GAE) /kg in olive oil, 0.8 mg GAE/kg in sunflower oil and not detectable in

peanut oil, while after the treatment with vegetation water they were 323.5, 234.8, and 243.5 mg GAE/kg in olive, peanut and sunflower oil respectively, thus 44.3-61.0% of the content found in the reference EVO (530.6 mg GAE/kg). The increase was slightly higher in the treatment with ultrasounds, after which the oils contained 46.8-63.2% of the TCP found in the reference EVO. There was also a transfer of volatile substances from the vegetation waters to the oils taken into consideration, for example 2-*E*-hexenal increases its content by 3.5 times in olive oil when treated with vegetation water by shaking, and by 2.7 times when treated by ultrasounds. The enrichment effects obtained will be shown in detail. The study shows how the content of substances that generally correlate positively with the oil healthy and sensory quality, such as polar phenolic substances, can be significantly increased in refined seed oils and olive oils, through a simple treatment with a by-product of the oil industry.

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Basics of efficient and environmentally friendly vacuum systems

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The corner stone for this now worldwide operating family establishment was laid when founding the company in 1871.

Supported by its subsidiary companies, Körting Hannover GmbH has placed its focus on technical engineering competence and flexibility in the respective application fields.

Constant and consequent further development, own research and development as well as the ongoing interchanging of ideas with customers and operators have resulted in the extensive product mix which Körting Hannover GmbH is able to offer today.

In practically all important process stages for the production of edible oils, Körting steam jet vacuum pumps and multi-stage steam jet vacuum systems are traditionally a guarantee for an energy - efficient and reliable vacuum creation.

To meet today's increasing demands for energy savings and environmental protection Körting now supplies besides the conventional barometric vacuum systems also different specially developed systems with closed water circuits, cold-water systems as well as ice (dry)-condensation vacuum systems for efficient edible oil processing.

Valorization of olive mill wastewaters through the production of phenol-enriched extracts: utilization for the formulation of low-nitrite meat products

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During the mechanical extraction of virgin olive oils, a large volume of wastewater (OMWW) is produced, which have a significant environmental impact due to a high BOD (35-100 g/L) and COD (40-195 g/L). However, OMWW represents a great source of phenolic compounds (about 48% of total phenols in olives), so if suitably treated, OMWW biophenols can be extracted, purified and used for the formulation of innovative food products. Due to the high radical scavenging capacity of these compounds, phenol-rich extracts can help reducing the use of synthetic antioxidants in food, such as nitrate/nitrite salts in processed meats, whose utilization is discouraged as they have been correlated to the onset of colorectal cancer. The aim of this study was to valorize by-products of olive oil food chain by testing the efficacy of an extract rich in phenols (PE), obtained from the purification of OMWW, to limit lipid oxidation in low-nitrite meat products. To pursue this aim, two meat products (cooked ham and wüstel) were formulated with different levels of PE and nitrites, followed by 30-day chilled storage. During the cold storage of cooked ham, more than 42% of the added phenols were still retained in all formulations (S1, S2 and S3 with PE 200 mg/kg + NO₂ 150/35/0 mg/kg, respectively) after 30 days of storage. Sample S1 showed the best oxidative stability, with a thiobarbituric acid reactive substances (TBARs) value close to 1.0 mg MDA/kg of meat, while the control sample (NO₂ 150 mg/kg) had significantly higher TBARs values (< 3.80 mg MDA/kg of meat). S2 and S3 displayed a similar oxidative trend, with TBARs values below 1.45 mg MDA/kg of meat. However, no significative differences on cholesterol oxidation products (COPs) and its oxidation ratio (COR%) were found between PE and control cooked ham samples. Therefore, it is possible to hypothesize that the antioxidant activity in both S2 and S3 was mainly due to phenols, while nitrites in S1 were more involved in the development of color and in the microbial stabilization of the product.

In the case of steam cooked wüstels, more than 64% of the added phenols were still retained in all formulations (W1, W2 and W3 with PE 200 mg/kg + NO₂ 35/150/0 mg/kg, respectively) after 30 days of storage. On the other hand, cooking caused a more drastic reduction of phenolic compounds in all grilled samples; 3,4-DHPEA-EDA, in fact, completely disappeared, while a severe loss of hydroxytyrosol was observed as well. Regarding oxidation, control sample (NO₂ 150 mg/kg) showed an increase in both conjugated dienes and trienes during the cold storage, while these parameters remained almost constant in ungrilled PE samples. After grilling, all samples exhibited the classical bell-shape behavior of primary oxidation products. Regarding secondary lipid oxidation, C and PE grilled samples had TBARs values that were about 2 times higher than those of steam-cooked wüstels, confirming the pro-oxidant effect of grilling. However, surprisingly, TBARs in both control steam-cooked and grilled wüstels were lower than those of the PE samples during storage. This behavior might be partly due to the thermolysis and hydrolysis of

phenols during the cooking treatments; moreover, during grilling at 220°C, phenols might have been involved in the Strecker reaction, preventing them from exerting their antioxidant role. Regarding COPs and COR%, no significative differences were found between PE and control steam-cooked wüistel samples.

This study confirms that the use of PE obtained from OMWW could be a successful way to valorize by-products of olive oil food chain and improve its sustainability, by efficiently stabilizing lipid oxidation in low-nitrate cooked meat products to promote the development of healthier meat products.

Innovation in the filtration of vegetable oils

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In the oil sector, especially in the EVO segment, ITALPROGETTI offers filtration technology with continuous flooding filter press, which offers numerous advantages both in terms of efficiency and process effectiveness. The two main innovative technologies that distinguish our filtration are:

- 1) running the filtration cycle, which can work up to 12 bar in the case of particularly difficult oils;
- 2) the squeezing system of the adjuvants at the end of the cycle at a pressure of 40 bar.

Filtration up to 12 bar and above all squeezing at 40 bar are made possible by an important filter structure, welded with a latest generation robot with high welding penetration performance.

The possibility of reaching 12 Bar of pressure and therefore a high Delta P between the two sides of the filter media guarantees, in accordance with Darcy's law, to be able to balance the increase in resistance of the filter media and the filter cake, thus being able to keep the filtration flow rate as constant as possible during the entire cycle.

In addition, the high Delta P achievable guarantees effectiveness even with very viscous oils such as oils at low temperatures or oils rich in waxes, such as Greek or refined oils. It is possible to reach the degree of polished with a single filtration cycle, even with difficult or freshly produced oils, thus avoiding the management of settling grounds.

The squeezing system, on the other hand, is responsible for the almost total recovery of the oily matrix from the exhausted adjuvants and guarantees optimal cleaning of the cloths, given the high level of compaction of these waste lands.

In fact, there is talk of a loss of less than 1 kg per ton of filtered oil and a time of about 30 minutes to restart with a new cycle, always with a clean filter.

In the Evo oil sector this system is already enjoying numerous successes and is entering the world of refined oils, avocado oil and winterization, in fact the possibility of filtering at low temperatures, even oils rich in waxes, has allowed, in some companies where there were problems with the detection of winterized oils, to reach the total brightening of the oily matrix.

Olive mill pomace valorisation: sustainable methods to extract phenolic compounds

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Nowadays one of the main worldwide challenges is the achievement of the 17 ONU sustainable development goals. Among these, the target 12.3 focuses on the reduction of food waste and losses along the food production and supply chains by 2030. In addition, by promotion of sustainable actions, one of the Europe targets is to become the first continent with zero impact by 2050. Olive oil production is an agro-industrial activity which has a relevant environmental impact in the Mediterranean area, since it generates annually about 30 million tons of waste, being olive mill pomace one of the major by-products. Olive mill pomace contains high load of organic compounds which is phytotoxic. Despite this, some of the organic compounds, such as phenolic compounds, are also widely recognised for their beneficial properties for human health. For this reason, the valorisation of olive mill pomace to recover functional high added value products, potentially useful in different sectors such as cosmetic and nutraceutical, represents an attractive strategy. The aim of this work is to develop a sustainable method for the recovery of phenolic compounds from olive mill pomace. The extraction procedure was tested on two types of raw materials, with and without added wastewater, collected directly from an Italian oil mill that uses a two-outlet decanter. Solid-liquid extraction protocols by using different solvents, such as ethanol (food grade, with low environment and lab operators toxicities) and other approaches which requires low amounts of solvents, were tested. In particular, the herein used solvent mixtures were methanol/water and ethanol/water, in different ratios and volumes. In brief, the mixture composed by the olive mill pomace and the solvent mixture was homogenised by a mixer, then introduced in an ultrasonic bath to enhance the extraction of the compounds of interest, and finally centrifugated. The supernatant was analysed by HPLC to study the phenolic fraction. An aliquot of the obtained polar fraction was used to analyse the content of hydroxytyrosol and tyrosol. With this purpose, sulphuric acid was added to promote the hydrolysis of the extracted fraction. Moreover, a mechanical approach based on a screw press was tested on olive mill pomace to obtain other extracts rich in phenolic compounds with a reduced use of solvents, making the process more sustainable. Ongoing studies are aimed to: i) improve the stability of the extracts, ii) reduce the need of water removal (e.g. through the addition of excipients such as corn starch), and iii) investigate their lifespan to identify potential practical uses.

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