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Flavour quality critical production steps from fruit to extra-virgin olive oil at consumption

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ABSTRACT

The production of olive oil comprises a number of production steps where the quality of the oil may be controlled through an understanding of how the production step influences key compounds such as volatiles and biophenols. In this study, critical production steps and significant inter-relationships between isolated production steps from fruit to oil-at-consumption were identified with the application of multivariate statistics. Having identified the key steps and relationships, sensory attributes associated with volatile and compounds may be enhanced and maintained during production and through to consumption. Our study showed that flavour compounds (e.g. oleuropein and derivates) could be controlled through olive fruit properties whereas transfer of the best sensory attributes from the fruit (e.g. arising from C5 and C6 volatiles) was critically controlled during oil extraction. Once olive oil was produced, maintenance of quality was critically controlled through storage conditions. Consequently, quality attributes from phenolic and volatile compounds could be targeted for maximum transfer from the olive fruit to oil while taking into account the impact on aroma, bitterness, and pungency of fresh oils and the subsequent loss in flavour quality during storage and consumption.

1. Introduction

The production of virgin olive oil commences in the grove where characteristics of the raw material, the olive fruit, determine the market value (Garcia, Magalhães, Fregapane, Salvador, & Paiva-Martins, 2012; Rotondi et al., 2004). Market value and classification of olive oil quality are dictated by flavour and its changes (Cavalli, Fernandez, Lizzani-Cuvelier, & Loiseau, 2004; Kalua, Bedgood, Bishop, & Prenzler, 2006b). For instance, premium quality, fresh, extra-virgin virgin olive oil is characterised by a fruity aroma and a peppery finish. For such oil, it is common for consumers to pay high prices. In contrast, the lower grades of olive oil, which retail at low prices, are distinctly "flat" in flavour.

The distinctive flavours of premium quality virgin olive oil are due to volatile compounds while the pepperiness (more correctly pungency and bitterness) is attributable to phenolic compounds (Andrewes, Busch, de Joode, Groenewegen, & Alexandre, 2003; Dierkes et al., 2012; Servili et al., 2004). Interestingly, most flavour compounds are not present in significant quantities in fresh olives. They are formed during virgin olive oil production and might be altered by the time olive oil reaches the consumer. In particular, it is at the oil extraction step where most significant fruity aroma development occurs—to be lost thereafter during the distribution/retail supply chain and consumption.

Quality in the virgin olive oil industry is assessed with emphasis on the olive fruit and virgin olive oil in terms of "potential" and "real" quality, respectively (Pardo, Cuesta, & Alvarruiz, 2007). Real quality is that measured on olive oils sampled randomly from storage tanks (Pardo et al., 2007)—common and standard for the olive oil industry. Evaluation of the sensory quality of virgin olive oils, usually assessed after oil extraction (real quality), involves perception of both favourable and unfavourable sensory attributes, with sensory defects used to classify oils into various grades (IOOC, 2003). Classification of olive oils based on potential quality is rare despite its importance in determining the real quality. Potential quality of olive oil is reached when healthy and clean olive fruits are selected at optimal maturity; processed at optimal conditions with quick separation of residues and by-products. Once the potential and real quality are assessed, stability is evaluated to determine the commercial quality of the oils at the end of the maximum possible time of storage from bottling and distribution to supermarkets or retail outlets (Pardo et al., 2007). Our previous experience (Kalua et al., 2006b) has shown that virgin olive oil quality deteriorates rapidly once extracted and the oil further loses its flavour at supermarkets or retailers prior to consumption. This loss of flavour quality calls for a paradigm shift to consider the entire virgin olive oil production and supply chain to enhance and maintain quality during consumption.

The diversity and inter-relationships of factors affecting the status of virgin olive oil quality make it tremendously difficult to carry out a complete quality investigation along the entire virgin olive oil production and supply chain without multivariate statistical techniques (Aparicio & Luna, 2002). Through a proper statistical process control,

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critical production steps can be identified and flavour quality attributes associated with volatile compounds and phenolic compounds transferred from olive fruit and maintained in the oil until consumption. "Critical production steps" are processes or operations that must be maintained under strict control to ensure the production and maintenance of a premium quality product (FAO, 1990). At these critical production steps, olive oil flavour quality attributes—aroma, taste, and pungency—can be controlled. The objective of this study was to identify critical production steps for virgin olive oil that could be controlled to enhance and maintain flavour quality attributes associated with volatile compounds and phenolic compounds. The concept of critical production steps is novel to the olive oil industry although it is common in process engineering—statistical process control—and in food technology to ensure microbial safety with "Hazard Analysis and Critical Control Points (HACCP)".

2. Materials and methods

2.1. Selection of samples and virgin olive oil production steps

Oil used to identify critical production steps was harvested in the same year (2005–06 season) from olive fruits of similar maturity indices (Table 1) that were not significantly different and the fruit was similarly processed without fruit storage apart from the work on fruit storage. The olive fruits used were also from the same *Frantoio* family; *Corregiola* and *Paragon* (Kailis & Considine, 2002), which have been reported to have similar volatile and phenolic profiles (Kalua, Allen, Bedgood, Bishop, & Prenzler, 2005).

The optimum conditions (Table 1) for the production and maintenance of premium quality virgin olive oil as identified earlier (Kalua, Bedgood, Bishop, & Prenzler, 2006a; Kalua, Bedgood, Bishop, & Prenzler, 2008; Kalua et al., 2005, 2006b), have been used to compare the relative changes in quality attributes from fruit to oil at consumer level. Although postharvest olive fruit storage is not a normal production operation, it was included in the process of identification of critical production steps (Table 1) as its likelihood to be part of the normal virgin olive oil production process increases when fruit supply exceeds processing capacity. The choice of fruit maturity and fruit storage represents changes in potential oil quality (Pardo et al., 2007). Real quality (Pardo et al., 2007) was represented through changes in quality just after oil extraction whereas changes during the distribution and supply chain were represented with oil storage in the absence of oxygen. Storage of oil in presence of oxygen simulates virgin olive oil changes that might occur during consumer use. Dark oil storage conditions (Table 1) were chosen since such conditions are recommended for the preservation of virgin olive oil quality (IOOC, 1990). This consideration therefore covers a likely oil production process from olive fruit to oil at consumer level.

2.2. Qualitative and quantitative analysis of phenolic compounds

Qualitative and quantitative analysis of the phenolic compounds in Table 2 was performed using liquid chromatography-electrospray ionisation-mass spectrometry (LC-ESI-MS) and high-performance liquid chromatography-diode array detector (HPLC-DAD) respectively, as described in our earlier paper (Kalua et al., 2005). Phenolic compounds were extracted with 50 + 50 (v/v) methanol + water solutions (3 × 1 mL) from virgin olive oil (15 g) dissolved in hexane (15 mL). Gallic acid (0.5 mL, 100 μg/g) was added to the oil as an internal standard. The hexane/methanolic mixture was vigorously shaken in an extraction flask (25 mL) and the lower methanolic layer drained out after each extraction. The combined methanolic extracts, from the oil, were washed with hexane and filtered through 0.45 μm plastic non-sterile filter prior to qualitative and quantitative analysis.

2.3. Qualitative and quantitative analysis of volatile compounds

Qualitative and quantitative analysis of the volatile compounds in Table 2 was performed using solid phase microextraction–gas chromatography–mass spectrometry (SPME–GC–MS) and solid phase microextraction–gas chromatography–flame ionisation detection (SPME–GC–FID) respectively, as described in earlier our papers (Kalua, Bedgood, & Prenzler, 2006) with a DVB-CAR-PDMS, 50/30 μm fibre.

2.4. Statistical data analysis

Stepwise linear discriminant analysis (SLDA) was applied on nineteen volatile compounds and twenty-four phenolic compounds to select discriminating variables that characterised particular production steps (Table 2) as described earlier (Kalua et al., 2005). Volatile and phenolic compounds concentrations were of different magnitudes and to necessitate comparison on a similar reference scale, standardised normal variables (statistical z-scores) were generated for each production step (Table 1). A cumulative statistical z-score was re-calculated for the entire virgin olive oil production process (CP1 to CP5, Table 1). Cumulative z-scores were plotted (Figs. 2–4) with Sigma Plot 10.0 (SPSS Inc., Chicago, USA) to compare the relative changes of volatile and phenolic compounds and identify critical production steps from olive fruit to extra-virgin olive oil at consumption. Significant differences ($p < 0.05$) between production steps were determined using one-way ANOVA post hoc multiple comparison tests using Duncan's test with SPSS 15.0 (SPSS Inc., Chicago, USA).

2.5. Identification of critical production steps and their indicators

Critical production steps were qualified with significantly different ($p < 0.05$) statistical z-scores from zero, otherwise the production steps were under control for particular volatile and phenolic compounds. The different signs and magnitudes of z-scores (Figs. 2–4) showed the different influences of virgin olive oil production steps (Table 1). Positive signs implied that optimising such steps maximise the compounds whereas negative z-scores indicate minimisation of the compounds at particular production steps. Minimal changes in z-scores (Figs. 2–4) showed controlled processes with minimal impact from a compound. The process of identifying either maximum

Table 1
Production conditions and samples used in the identification of critical production steps.

Control point (CP)	Production steps	Optimum conditions	Olive cultivar	Olive fruit colour	Reference
CP1	Fruit maturity	Oil extracted from spotted olive fruits	*Corregiola*	Spotted (MI = 3.06 ± 0.68)	Kalua et al. (2005)
CP2	Fruit storage	Oil extracted from olive fruit stored at low temperatures (4 ± 2 °C) for 2 weeks	*Frantoio*	Spotted (MI = 2.92 ± 0.06)	Kalua et al. (2008)
CP3	Oil extraction	Malaxation at 30 °C for 60 min	*Corregiola*	Spotted and Green (MI = 2.4 ± 0.1)	Kalua et al. (2006a)
CP4	Oil storage (without headspace)	Oil stored at room temperature (24 ± 3 °C) in the dark for 4 months	*Paragon*	Spotted (MI = 3.06 ± 0.68)	Kalua et al. (2006b)
CP5	Oil storage (with headspace)	Oil stored at room temperature (24 ± 3 °C) in the dark for 2 months	*Paragon*	Spotted (MI = 3.06 ± 0.68)	Kalua et al. (2006b)

"MI" represents maturity index of the olive fruit.