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Influence of calcium carbonate on extraction yield and quality of extra virgin oil from olive (*Olea europaea* L. cv. Coratina)

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ABSTRACT

The aim of the research was to evaluate the effect of calcium carbonate (1%, 2%, and 4% of addition) at two different particle sizes (2.7 μm and 5.7 μm), added at the beginning of the malaxation phase, on both the extraction yield and the quality of oil obtained from Coratina olives at different ripening index. The results showed that calcium carbonate significantly increased the extraction yield of olive oil, more than affecting chemical indices. In particular, for less ripened olives, 1–2% of larger particle size calcium carbonate addiction determined a significant increase of the extraction effectiveness, ranging from 4.0 to 4.9%, while more ripened olives required higher amounts of coadjuvant (2–4% when using the larger particle size and 4% when using the smaller one), with a significant increase of the extraction yield up to 5%. Moreover, an increase of pungent perception was observed in some cases when adding calcium carbonate to more ripened olives.

1. Introduction

Extra virgin olive oil is the most produced oil in the Mediterranean area and, due to its beneficial effects on humans' health, is currently being cultivated also in Australia, South Africa, Argentine, California, Chile, China, and more recently India (Kapellakis, Tsagarakis, & Crowther, 2008; Loumou & Giourga, 2003; Luchetti, 2002; Petruccelli et al., 2015; Úrbez-Torres, Peduto, Vossen, Krueger, & Gubler, 2013). The main nutritional and healthy features of extra virgin olive oil can be attributed to its peculiar composition, characterized by high content of monounsaturated fatty acids (oleic acid) as well as by several minor compounds such as phytosterols, carotenoids, tocopherols, and phenolic compounds (Huang & Sumpio, 2008; Perez-Jimenez et al., 2005; Stark & Madar, 2002), all contributing to the prevention of cardiovascular pathologies (Psaltopoulou et al., 2004; Visioli, Poli, & Galli, 2002).

According to current rules (Official Journal of the European Communities, 2001a), virgin olive oils can be produced exclusively by means of mechanical treatments of the starting olives aimed to separate the highest possible amount of the lipid fraction from the drupes. However, some oil remains into the drupe (Aguilera, Beltran, Sanchez-Villasclaras, Uceda, & Jimenez, 2010) due to several factors, not only imputable simply to eventual machinery inefficiencies (Di Giovacchino, 2010). In fact, the degree of ripening of the olives which, in turn, influences their chemical composition and their physical and structural features (Huisman, Schols, & Voragen, 1996; Vierhuis, Schols, Beldman, & Voragen, 2000), as well as the agronomic growing conditions (such as irrigation level and fertilization type), or the incidence of plant pathogens (Torres-Vila, Rodriguez-Molina, & Martínez, 2003), can strongly affect extraction efficiency. Moreover, an aliquot of the oil contained in the drupes is difficult to extract during processing: the so-called 'bound oil' remains into colloid systems arose in the olive paste, or is emulsified with the aqueous phase, or even stays trapped into the few cells remained intact (Espinola, Moya, Fernández, & Castro, 2009). The difficulty in freeing the bound oil is due to the action of a lypoproteic membrane that surrounds the dispersed or emulsified oil droplets and that stabilizes them (Petursson, Decker, & Mc Clements, 2004). Another important factor affecting the oil extractability is the presence of high molar mass polymeric substances derived from cell wall polysaccharides (Sadkaoui, Jiménez, Pacheco, & Beltrán, 2015). These phenomena are particularly evident in case of olive cultivars that are characterized by rather low ripening index at the moment of harvesting, such as Coratina cultivar, known to give 'difficult pastes' (Caponio et al., 2014a; Cruz, Yousfi, Pérez, Mariscal, & García, 2007; Di Giovacchino, 2010).

Several attempts have been made to improve the coalescence of oil droplets and to increase extraction efficiency: malaxation time prolongations, though it should not exceed 45 min to preserve the

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quality of extra virgin olive oil; temperature increases, though 30 °C should not be exceeded to guarantee the nutritional characteristics of the extra virgin olive oils; enzyme addition; coadjuvant addition, such as sodium chloride, micronized talc, or calcium carbonate (Caponio, Summo, Paradiso, & Pasqualone, 2014b; Cruz et al., 2007; Espinola et al., 2009; Moya et al., 2010; Ranalli, Pollastri, Contento, & Iannucci Elucera, 2003).

Among coadjuvants, calcium carbonate, in particular, does not involve any health risk for oil-mill operators that, instead, are related to the use of micronized talc (Ramanakumar, Parent, Latreille, & Siemiatycki, 2008), and is authorized by European Community (Official Journal of the European Communities, 2001b) under the E170 code. Moya et al. (2010), compared the effectiveness of different coadjuvants during olive processing and observed that calcium carbonate, in the same operative conditions, give better results than micronized talc. In particular, the highest oil yields were recorded by using 2.7-μm particle sized calcium carbonate at 0.3% w/w levels. At greater particle size, and at higher levels of calcium carbonate, the extraction yield decreased.

However, almost all the researches carried out until now about the effects of calcium carbonate on extraction yield and quality were carried out on Spanish olive cultivars. Few studies have regarded Italian olive cultivars. Coratina, that represents one of the most diffused Italian cultivars, gives a difficult paste because it has a rather low pigmentation index at the usual time of oil processing. Moreover, there is a lack of information about the effectiveness of calcium carbonate as a function of olive ripening index. In this framework, the aim of the present research was to evaluate the effect of calcium carbonate at two different particle sizes on both the extraction yield and the quality of oil obtained from Coratina olives at different ripening index.

2. Materials and methods

2.1. Experimental plan

Olive fruits (*Olea europaea* L. cv. Coratina) mechanically harvested in December 2013 (trial A) and in January 2014 (trial B) were transported, immediately after harvesting, to a local plant located in Bari territory (Apulia, Southern of Italy) where, after leaf-removal, were milled within 24 h.

Two lots of about 4500 kg of olives were considered for each sampling data. Each lot was divided into fourteen homogeneous batches of about 300 kg. In particular two batches of olives were submitted to extraction under each of the following processing conditions:

- without calcium carbonate addition (C, control),
- with 1% of Calcipur®2 (Ca2-1%),
- with 2% of Calcipur®2 (Ca2-2%),
- with 4% of Calcipur®2 (Ca2-4%),
- with 1% of Calcipur®5 (Ca5-1%),
- with 2% of Calcipur®5 (Ca5-2%),
- with 4% of Calcipur®5 (Ca5-4%).

For all the trials, calcium carbonate was added at the beginning of the malaxation phase. The olives after crushing (A40, Amenduni Nicola S.p.A., Modugno, Bari, Italy) were malaxed (6V830, Amenduni Nicola S.p.A., Modugno, Bari, Italy) at 25 °C for 45 min (including the malaxer loading and unloading time). Calcium carbonate, when provided, was added after the loading of malaxer. After malaxation the paste was pumped to the three-phase decanter (Ariete 902, Amenduni Nicola S.p.A., Modugno) – operating at 2970 rev min^{-1}, with a processing capacity of 1500 kg h^{-1} – and diluted with 380 L h^{-1} of water to separate the oil from pomace and waste water. Finally the oil was separated by the remnant water residues by means of a vertical axis centrifuge (A-3500, Amenduni Nicola S.p.A., Modugno, Bari, Italy) operating a 6400 rev min^{-1}. The oils were sampled in dark glass bottles having the capacity of 250 mL that were hermetically sealed. Every trial was sampled in duplicate.

2.2. Technological coadjuvants

Calcium carbonate was kindly furnished by Omya Spa (Milan, Italy). Two different mean particle sizes were considered: 2.7 μm (Calcipur®2) and 5.7 μm (Calcipur®5).

2.3. Pigmentation index and olive firmness

The pigmentation index (P_i) of the olive fruits was calculated according to Camposeo, Vivaldi, and Gattullo (2013) as follows:

$$P_i = \sum_{i=0}^{5} \frac{(i \times n_i)}{T}$$

where i is the group number, n_i is the number of fruits per group, T is the total number of fruits of the sample. The procedure consisted in distributing the sample of olives in six groups, according to the following characteristics: group 0, green skin; group 1, <50% black skin with white flesh; group 2, ⩾50% black skin with white flesh; group 3, 100% black skin with white flesh; group 4, 100% black skin with <50% purple flesh; group 5, 100% black skin and ⩾50% purple flesh ($0 \leqslant P_i \leqslant 5$).

The fruit firmness (FF, expressed as N) was measured by means of a FT 011 penetrometer (Turoni, Forlì, Italy) by a 2-mm diameter probe applied to the equatorial zone of the drupe (Camposeo et al., 2013).

2.4. Extraction efficiency

The extraction effectiveness was calculated as reported by Moya et al. (2010), i.e. as the percent ratio between the amount of oil recovered at the oil-mill to the real drupe oil content, the latter assessed by Soxhlet method.

2.5. Analytical determinations

The moisture content and the total oil content of olives (% w/w) were determined by drying the milled paste at 105 °C to constant weight and by Soxhlet extraction, respectively, as reported in a previous paper (Caponio et al., 2015).

The determination of the free fatty acids (FFA), peroxide value (PV), spectrophotometric constants phenolic compounds, chlorophylls, carotenoids, tocopherols, and antioxidant activity was carried our as reported in previous papers (Caponio, Bilancia, Pasqualone, Sikorska, & Gomes, 2005; Caponio et al., 2014a). For the determination of the volatile compounds, the oil samples (1 ± 0.005 g) were weighed into 20-mL vials, sealed with a screw top aluminium cap and pierceable butyl rubber septa, and submitted to the (SPME/GC–MS) in the conditions reported in Caponio et al. (2014b). The sensory evaluation of the virgin olive oils was performed as reported in Caponio et al. (2014b).

2.6. Statistical analysis

One-way and two-way analysis of variance (ANOVA), followed by Fisher LSD test for multiple comparisons, was carried out on the experimental data by the XLStat software (Addinsoft SARL, New York, NY, USA). Two-way ANOVA was performed considering the amount of coadjuvant added to olive paste during malaxation