Available online at www.sciencedirect.com
ScienceDirect
C. R. Chimie 12 (2009) 895–904
Comptes Rendus Chimie
http://france.elsevier.com/direct/CRAS2C/

Full paper / Mémoire

Chemical changes in virgin olive oils as a function of crushing systems: Stone mill and hammer crusher

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Received 3 June 2008; accepted after revision 19 January 2009
Available online 21 March 2009

Abstract

Olive oils were tested for their chemical composition in polyphenols, free fatty acids and volatile compounds as a function of the crushing systems, i.e. the stone mill and hammer crusher. The qualitative and quantitative HPLC/DAD analyses of the olive oils showed that luteolin and tyrosol were the most abundant identified phenolic compounds. The olive oil obtained by the hammer crusher had the highest concentration of phenolic compounds and ultimately the strongest antioxidant activity. Olives treated by the two crushing systems were observed by scanning electronic microscopy. Micrographs provide more evidence of the better cell cuts of olive fruits treated by hammer crushing, in contrast to stone mill where olive cell layers have been broken and damaged. ***To cite this article: S. Veillet et al., C. R. Chimie 12 (2009).***

Résumé

Des huiles d'olive issues de deux procédés de broyage (meule de granit ou broyeur à marteaux) ont été comparées en terme de composés polyphénoliques, composés volatiles et acidité libre. Les analyses qualitatives et quantitatives par CLHP avec une détection à barrette de photodiodes des huiles montrent que la lutéoline et le tyrosol sont les composés biophénoliques identifiés majoritaires. L'huile d'olive issue du broyeur à marteaux a des concentrations plus fortes en composés biophénoliques et montre une plus forte activité antioxydante. Les olives broyées par les deux systèmes de broyage ont également été observées par microscopie électronique à balayage. Les photographies mettent en évidence une meilleure découpe des cellules de fragments d'olives issues du broyeur à marteaux, par opposition à la meule de granit pour laquelle les cellules ont été simplement cassées et endommagées. ***Pour citer cet article : S. Veillet et al., C. R. Chimie 12 (2009).***

Keywords: Olive oil; Stone mill; Hammer crusher system; Scanning electronic microscopy; HPLC/DAD; GC/MS

Mots-clés : Huile d'olive ; Moulin a meule ; Moulin à marteaux ; Microscopie électronique à balayage ; CLHP/DAD ; CG/SM

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doi:10.1016/j.crci.2009.01.003

1. Introduction

Virgin olive oils play an important role in the food industry and are important in human nutrition for a variety of reasons. Lipids are the main sources of energy for the human body compared with their weight. Moreover, olive oils are becoming increasingly important as suppliers of fat-soluble vitamins and polyphenols as antioxidants. They are also an important source of essential unsaturated fatty acids, which cannot be synthesized by the human body. Virgin olive oils are extracted from olive fruits by using only physical methods, which include crushing of olives, malaxation of resulting pastes and separation of the oily phase. Because of its location in mesocarp of cells and the use of purely mechanical pieces of apparatus for its extraction, virgin olive oil does not require further treatment before its consumption. Therefore, it preserves a great number of volatile compounds, responsible for the aroma and also the presence of antioxidant compounds which inhibit the lipid autoxidation (nonenzymatic oxidation by dioxygen).

Provence, the most important area of France for olive oil production, is part of the Mediterranean basin where the climate and soil encourage production of olive fruits and consequently virgin olive oil at high quality with lots of volatile and non-volatile compounds of interest. The extraction procedure is of great importance to produce olive oil rich in secondary metabolites [1–4]. The choice of the technique is the result of a compromise between efficiency and reproducibility of extraction, ease of procedure, together with considerations of cost, time, degree of automation and safety.

The objective of this work was to investigate the effects of the crushing systems, stone mill and hammer crusher, on the chemical changes of virgin olive oil considering quality parameters such as: free acidity, peroxide value, conjugated dienes concentration, fatty acid composition, volatile compounds, and polyphenolic compounds with regard to their antioxidant potentialities. The chemical analysis studies were supplemented by scanning electron micrographs to shed light on the physical action of the two crushing systems.

2. Materials and methods

2.1. Sample collection and reagents

In these investigations, oil was extracted from olives *Aglandau* (Vaucluse, France) collected from local fields (Domus Claudia, Hameau de st Veran, France) during the harvesting period for oil production (1st and 2nd of December 2007). 1000 kg of olives were harvested and processed in a small scale mill (Le petit Moulin, Travaillan, France) on the 3rd of December 2007.

Acetic acid, chloroform, sodium thiosulfate, ethanol 96%, phenolphthalein, BF_3–methanol and *n*-heptane used were of analytical grade; *n*-hexane and Folin-Ciocalteu reagent were of UV-grade; and methanol, water and *n*-hexane used for HPLC were of HPLC grade. All these solvents were supplied by VWR International (Darmstadt, Germany).

Standards of gallic acid, vanillic acid, p-coumaric acid, ferulic acid and *trans*-cinnamic acid were from Alfa-Aesar (Karlsruhe, Germany) and those of tyrosol, luteolin and apigenin were from Extrasynthèse (Genay, France).

2.2. Oil processing

2.2.1. The stone mill

The stone mill is still the most common way of crushing olives in the south of France even if only small scale mills can use it due to the longer time of grinding compared to more modern crushers (Fig. 1). In the mill of this study the two granite stones used to grind the olives were 23 cm wide and turned at 17 rpm. The maximum charge at one time was 250 kg of olive and the crushing time was about 35 min at room temperature (20 °C).

2.2.2. The hammer crusher

Hammer crushing is used in almost all the large scale mills. The olives were introduced into a metal container where a six arm hammer was rotating at a speed of 2890 rpm (Fig. 1). Once properly crushed at room temperature, the ground olives fell through a 0.7 cm grid before going into the malaxation compartment.

2.2.3. Other steps

Malaxation was performed in a metal container with a no-end screw without any heating of the olive paste. This step lasted for 25 min until a good coalescence of the olive oil droplets was achieved. Then the olive paste was placed on fiber disks which are stacked on top of each other and further it was placed into the press where a pressure of 400 bar was applied. This allows the liquid/solid separation and the liquid fraction goes through a centrifuge at a speed of almost 7000 rpm for 10 min. Olive oil obtained was immediately frozen and kept at −18 °C prior to analyses.