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Effect of roasting conditions on pigment composition and some quality parameters of pistachio oil

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ABSTRACT

Pistachio roasting before oil extraction increases consumer preference but may cause changes in the oil composition. In this work, the effect of different roasting conditions on the physical parameters, oxidative stability, and pigment composition of pistachio oil extracted by pressure was studied. Density value of pistachio oil was reduced with severe roasting conditions (125 °C), while viscosity increased slightly. This adverse effect was compensated by a significant increase in both oxidative stability and, especially, in the content of chlorophyll and carotenoid pigments. Pistachio roasting temperature had a clear impact on the color of the pistachio oils, changing from yellow in oils from raw or minimally roasted pistachios (50–75 °C) to brilliant green in oils from pistachios subjected to higher temperature treatments (100–125 °C). An increase in temperature favored the pigment transfer to the oil. The green oils had a total pigment content between 2.3 and 4 times higher than the yellow oils.

1. Introduction

Pistachio nut (*Pistacia vera* L.) is one of the most popular nuts in the world. It is widely cultivated across arid zones in the world due to the ability of pistachio tree to grow in hot and dry areas. The production of pistachio worldwide has increased from 440.000 tons in 2004 to exceed 850.000 in 2014. Iran, the United States and Turkey are the major pistachio producers (FAO, 2016). The escalation of pistachio production has been the cause of an important increase of pistachio demand due to the health-promoting compounds of nuts in general and pistachio particularly.

Pistachios have a high nutritional value, becoming a good source of vegetable proteins, fiber with positive effects on gut microbiota associated to its consumption (Ukhanova, et al., 2014) and phytosterols linked to the reduction of total plasma cholesterol and low-density lipoprotein cholesterol (Rabadán, Álvarez-Ortí, Pardo, et al., 2017). In addition, pistachios contain between 50 and 62% of oil with healthy fatty acid profile, composed mainly by unsaturated fatty acids, mainly oleic (52–81%) and linoleic (8–31%) (Catalán, et al., 2017; Hojjati, Noguera-Artiaga, Wojdyło, & Carbonell-Barrachina, 2015). Due to its high lipid content, the extraction of pistachio oil appears a viable procedure (Rabadán, Álvarez-Ortí, Pardo, et al., 2017). Within oil extraction methods, cold pressed extraction has been widely studied as it allows the production of high quality oils (Álvarez-Ortí, Quintanilla, Sena, Alvarruiz, & Pardo, 2012). This virgin oil obtained can be consumed directly without the need to apply a refining process. Therefore, its healthy properties are not only due to an adequate composition of fatty acids but also to the presence in the virgin oil of other fundamental minor components that contribute to the specific characteristics of these oils, such as oxidative stability, and special flavor and color.

Pistachios are particularly appreciated for its emerald-green color, originated by the pigments present in the kernel. Chlorophylls *a* and *b*, and lutein are the major pigments found in raw pistachios (Bellomo & Fallico, 2007; Pumilia et al., 2014). Pigment composition of pistachio nut may vary depending on several factors such as variety, growing region, ripeness degree (Bellomo & Fallico, 2007), storage conditions (Bellomo, Fallico & Muratore, 2009) and processing techniques (Pumilia et al., 2014). Chlorophylls *a* and *b*, may reach 150 mg/kg dry matter in raw pistachio (Bellomo & Fallico, 2007). Extractable chlorophylls and lutein are higher in pistachio kernels slightly roasted than in the raw kernel, although this process degrades chlorophylls to pheophytins, pyropheophytins and pyrochlorophylls (Pumilia et al., 2014). Beyond temperature, pistachio long term storage also causes the formation of pheophytins (Bellomo et al., 2009). Regarding lutein, this is a xanthophyll carotenoid and its content in pistachios has been estimated to range from 18 to 52 mg/kg dry matter depending on the

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pistachio origin and the kernel degree of ripeness (Bellomo & Fallico, 2007; Bellomo et al., 2009). In contrast with chlorophylls, lutein seems to be more resistant to high temperatures (Khachik et al., 1992). In addition, lutein dissolved in oil is more resistant to thermal processing than other carotenoids (Henry, Catignani, & Schwartz, 1998), with studies even reporting an increase in the content of lutein after thermal processing of food products due to inactivation of the enzymes able to oxidize the carotenoids (Kirk & Tilnet-Basset, 1978).

Pistachios are usually sold and consumed roasted, so roasting process is responsible for the characteristic aroma and taste of pistachios. Pistachio aroma after roasting has been identified as determinant for consumer acceptance. However, the appearance and evolution of main components of sensory odour and flavour aromas with roasting also differ depending on the pistachio cultivar considered.

Roasting increases the concentration of volatiles and originates changes in the color of pistachios as a result of Maillard reaction (Hojjati, Calín-Sánchez, Razavi, & Carbonell-Barrachina, 2013; Ling, Yang, Li, & Wang, 2015). The Maillard reaction causes the decrease of sugars and can lead to the formation of aroma compounds that cause consumer preference. The antioxidant activity of Maillard reaction compounds is considered responsible of the increase of oxidative stability values in oils obtained from thermally treated foods (Rabadán, Pardo, et al., 2017). Pistachio roasting could even be able to inactivate enzymes responsible for pistachio rancidity (Pumilia et al., 2014), increasing pistachio quality and shelf-life.

The preference of consumers for roasting has been also described in pistachio oil when roasted pistachios are used for oil extraction (Rabadán, Álvarez-Ortí, Gómez, et al., 2017). The drying treatment of pistachio may originate changes in the oil characteristics and pigment concentration when the temperature is high (Sena-Moreno et al., 2015). In recent years there has been growing interest in pistachio oil due to its value in the food and cosmetic industries (Catalán et al., 2017). Chlorophyll and carotenoid pigments are among the components of interest present in pistachio oils. They are responsible for the oil color, which has also a marked influence on consumer preference and acceptance. In addition, a wide range of studies have reported important roles that chlorophyll and carotenoid compounds play in health as bioactive components, what make them even more attractive (Mínguez-Mosquera, Gandul-Rojas, Gallardo-Guerrero, Roca, & Jarén-Galán, 2008; Mínguez-Mosquera, Hornero-Méndez, et al., 2008). Special attention has been drawn to the potential health benefits of a carotenoids-rich diet, due to their role as antioxidants (Landrum & Bone, 2001).

A global estimate of the chlorophyll and carotenoid fractions may be directly obtained from the absorption spectra of the oil dissolved in cyclohexane (Mínguez-Mosquera, Rejano-Navarro, et al., 1991). Recent researches have determined in that way the pigment contents in pistachio oils from different cultivars (Ojeda-Amador, Fregapane, & Salvador, 2018) or from raw and roasted pistachios (Ling, et al., 2015; Martínez et al., 2016). Therefore, all these works have been limited to quantify the total chlorophyll and carotenoid contents, and possible differences in the individual pigment composition, which could modulate the functional properties of these components in the pistachio oil, have not been considered. The complexity of food systems due to the co-oxidant or protective effect that the numerous compounds can show, encourages the study of the evolution of pigments in every oil matrix individually (Henry et al., 1998). The aim of this study was to characterize for the first time the pigment profile of pistachio oil, as well as evaluate the effect of different roasting conditions of pistachio on the chlorophylls and carotenoids composition of the corresponding oils, obtained by cold pressed extraction. In addition, changes on physical parameters of oil (color, density and viscosity) and oxidative stability were also analyzed. Our major interest was focused in the changes in pigment composition by effect of different roasting conditions, and their relationships with oil quality and functional properties.

2. Materials and methods

2.1. Chemicals and standards

Ammonium acetate was supplied by Fluka (Zwijndrecht, The Netherlands). Solvents used for chromatography were HPLC grade (Prolabo, VWR International Eurolab, Barcelona, Spain). Analysis grade solvents were supplied by Scharlau (Microdur, Sevilla, Spain). The deionized water was obtained from a Milli-Q® 50 system (Millipore Corporation, Milford, MA). For all purposes, analytical grade (American Chemical Society) reagents were used (Merck, Madrid, Spain).

Standards of chlorophylls *a* and *b*, lutein and β-carotene were supplied by Sigma Chemical Co. (St. Louis, MO). Standards of pheophytin *a* and pyropheophytin *a* were provided by Wako chemicals Gmbh (Neuss, Germany). Standard of pheophorbide *a* was purchased from Frontier Scientific Europe Ltd. (Carnforth, Lancashire U.K.). All other chlorophyll derivatives were prepared in the laboratory from the related chlorophyll or pheophytin (*a* or *b*) (Chen, Ríos, Pérez-Gálvez, & Roca, 2015; Mínguez-Mosquera, Gandul-Rojas, et al., 2008; Vergara-Domínguez, Gandul-Rojas, & Roca, 2011). Phytol-free derivatives were obtained by enzymatic de-esterification, and Mg-free derivatives by acidification with 5 M HCl. Pyro-derivatives were formed by heat treatment in pyridine at 80–100 °C with reflux, while 13^2-hydroxy- and 15^2- hydroxy-lactone derivatives were prepared in a similar way but including selenium dioxide to produce the oxidation of the isocyclic ring.

Violaxanthin, neoxanthin, and antheraxanthin were obtained from a saponified extract of pigments from fresh spinach and separated by TLC with silicagel GF_{254}, using petroleum ether (65–95 °C)/acetone/diethylamine (10:4:1). β-cryptoxanthin was obtained from papaya. Luteoxanthin and auroxanthin were prepared from violaxanthin by acidification and subsequent separation by TLC. Similarly, neochrome and mutatoxanthin were prepared from neoxanthin and antheraxanthin, respectively (Mínguez-Mosquera, Hornero-Méndez, et al., 2008).

2.2. Pistachio samples and oil extraction

Pistachios of the Kerman variety were collected manually and selected according to its sanitary state from orchards of the Instituto Técnico Agronómico Provincial de Albacete (ITAP), in Albacete (Spain). Pistachios were immediately shelled in controlled conditions before drying at room temperature for three days until a humidity percentage lower than 5% was reached. Dried pistachios were vacuum packed and refrigerated until processing.

Before oil extraction, pistachios were subjected to a roasting step. Eight homogeneous pistachio batches of 1 kg were subjected to roasting under different conditions of temperature (50, 75, 100 and 125 °C) and time (15 and 30 min). For the roasting process, pistachios were distributed in trays in a monolayer, and introduced in a forced air oven (Heraeus UT6, Hanau, Germany) controlling the temperature and time as previously described. Additionally, oil from a batch of unroasted pistachio was also obtained.

To extract the oil, a hydraulic press was used (Mecamaq DEVF 80, Vila Sana, Lleida, Spain). This press allows the oil extraction at room temperature, avoiding any other additional effect of heat on oil characteristics. Samples of 250 g of each batch were ground and subjected to a pressure of 200 bar for 15 min. The resultant oil was centrifuged at 12000 rpm for 5 min to eliminate residual solid particles.

2.3. Physical characteristics and oxidative stability

Oil samples were filtered and the color was measured using a spectrophotometer UV/Vis Jasco V-530 (Jasco Analytical, Madrid, Spain). *N*-hexane was used as blank reference. The values obtained were used to calculate the CIELAB chromatic coordinates: L*