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Original Article

Aroma transition from rosemary leaves during aromatization of olive oil

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ABSTRACT

The aroma profile of aromatized olive oil was determined in this study. The primary objective was to investigate the transition of major aroma compounds from rosemary and olive fruit during the kneading step of olive oil production by response surface methodology. For this purpose, temperature, time, and amount of rosemary leaves were determined as independent variables. The results indicated that temperature and time did not affect the transition of target compounds, but rosemary leaves addition had a strong influence on transition, especially for characteristic aroma compounds of this herb. Adequacies of developed models were found to be high enough to predict each aromatic component of interest.

1. Introduction

Olive fruit and its related products, especially extra-virgin olive oil, are popular products in the Mediterranean countries because of their delicious taste, pleasant aroma, and nutritional benefits [1–3]. These products have their own characteristic aroma and taste, which differentiate them from other similar products. Thus, the aroma profile of any olive product plays a significant role in its quality evaluation and product characterization. The main aroma compounds that migrate from olive fruits to oil are *trans*-2-hexenal, hexanal, and *cis*-3-hexenal [4,5]. In recent times, aromatized olive oil has been gaining increasing attention in the olive oil industry, because the main objective of aromatization is to produce alternative tastes for consumers. Aromatized olive oil is generally produced by small scale producers (boutique manufacturers). Herbs and aromatic plants are extensively used in aromatization due to their strong aromas. Rosemary (*Rosmarinus officinalis* L.; Family: Lamiaceae) is one of the popular plants used in the aromatization of olive oil due to its beneficial effect on health and significant nutritional potential with

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high antimicrobial, antidiabetic, and antioxidant effects [6–10].

α-Pinene, 1,8-cineole, camphene, camphor, *p*-cymene, myrcene, limonene, and β-caryophyllene have been reported as the main volatile compounds responsible for the strong aroma of rosemary [7,11]. Different techniques are used to induce transition of compounds of interest from aromatic plants and herbs into olive oil. These, in most cases, involve mixing their extracts with oil or adding these herbs/plants to the oil. However, these methods are reported to have some disadvantages such as turbidity, overdosage [12], and coextraction of undesirable constituents (waxes and bitters) [1]. By contrast, some aromatization techniques involve direct addition of ground and/or whole-plant materials into olive or olive paste during the crushing and malaxation steps, respectively. However, these methods also cause some problems, which should be resolved prior to obtaining standard aromatized olive oil. For example, in the crushing step, it is not easy to adjust the concentration of aromatic plant added due to the nonhomogenous distribution of leaves, woody parts, and limited time available for transition. In the malaxation step, kneading parameters have a significant effect on transition of target compounds from natural source to olive oil [4,13,14]. Previous studies have indicated that temperature and time are important variables affecting the malaxation step, and thus both should be considered and well adjusted [4,14,15]. Although there are studies on aromatized olive oils, to the best of our knowledge none of these studies has examined the influence of malaxation parameters and herb amount on the aroma profile of aromatized olive oil.

The main objective of this study was to evaluate the transition of aroma compounds from rosemary and olive fruit to the final oil under the influence of malaxation parameters and amount of herb.

2. Material and methods

2.1. *Study material*

Gemlik olive, a commercial cultivar, was used as the raw material in this study. The aromatic plant rosemary (*R. officinalis*) was cultivated in the research and application fields of Agricultural Faculty of Süleyman Demirel University, Isparta, Turkey. Rosemary was ground and sieved using a 1-mm sieve. Samples were stored in a sealed plastic bag at 4°C until further use. Analytic standards (α-pinene, myrcene, *p*-cymene, camphor, 1,8-cineole, and camphene) were purchased from Sigma-Aldrich Co. Ltd. (St Louis, MO, USA), limonene was purchased from Fluka (Steinheim, Germany), and hexanal and *trans*-2-hexenal were purchased from Merck (Darmstadt, Germany).

2.2. *Methods*

2.2.1. *Experimental design*

A central composite design was chosen to model the variation in compounds of interest in the aroma profile as a function of malaxation conditions for each of the following: temperature, time, and rosemary amount at five levels with 18 runs including four central points. Independent variables were temperature (X_1), time (X_2), and rosemary amount (X_3). The area of each major aroma compound (α-pinene, 1,8-cineole, camphene, camphor, *p*-cymene, myrcene, and limonene) was the dependent variable in this study. The range and levels of independent process variables with coded values and corresponding responses, which are experimentally obtained, are presented in Tables 1 and 2. Response surface methodology was used to evaluate the effects of process parameters and to produce the corresponding models. Experimental data were analyzed using Minitab Software (Minitab version 16.1.1; Minitab, Inc., State College PA, USA). Full quadratic second-order regression model including the linear, quadratic, and two-factor interaction effects was used for the prediction of process conditions towards targets (Equation 1).

$$Z = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ij} X_i X_j \qquad (1)$$

where:

Z is the dependent variable;
X is independent variables;
β_0 is the constant coefficient;
β_i is the linear coefficient (main effect);
β_{ii} is the quadratic coefficient; and
β_{ij} is the two-factor interaction coefficient.

Response surfaces of predicted values obtained using proposed models were plotted in the studied variable ranges using the Sigma Plot Software (SPSS Inc., Chicago, IL, USA). Model adequacy was evaluated by considering parameters of R^2 value and lack-of-fit test.

2.3. *Extraction of olive oil*

Olive oil was extracted according to the experimental design of the malaxation process (Table 1) using the Abencor method [16]. The aromatized oil obtained was filtered using cotton and anhydrous sodium sulfate. The filtered oils were stored in amber glass bottles at 4°C without headspace until further analysis.

2.4. *Determination of aroma profile of aromatized olive oil by solid-phase microextraction–gas chromatography/mass spectrometry*

A 2-g sample was weighed in a 15-mL vial closed by a silicone septum. The sample was placed on a heating block at 45°C and held for 15 minutes to achieve temperature equilibrium. A Carboxen/polydimethylsiloxane manual solid-phase microextraction (SPME) fiber (75-μm Fused Silica, Supelco Ltd., Bellefonte, PA, USA) was inserted into the vial and kept for 30 minutes at 45°C to absorb volatile compounds from olive oil. The fiber was then inserted into the injection port of gas chromatograph for 5 minutes at 250°C for the desorption of aroma compounds.

Gas extraction/mass spectrometry (GC/MS) analyses were performed using a Shimadzu GC-2010 gas chromatograph equipped with an MS-QP2010 plus a mass spectrometer