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ORIGINAL ARTICLE

# Ontogenetic changes in the volatile composition of *Rosa damascena* Mill. flowers revealed by HS-SPME-GC/MS

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**Abstract:** Ontogenetic changes in the volatile composition of oil-bearing rose (*Rosa damascena* Mill.) flowers were investigated at eight developmental stages (HS1–HS8) under semiarid conditions in Mardin, Türkiye. Volatile profiles were analyzed using headspace solid-phase microextraction coupled with gas chromatography–mass spectrometry (HS-SPME-GC/MS). A total of 39 volatile constituents were identified, representing 96–99% of the total volatile profile. Distinct stage-dependent shifts were observed throughout flower development. Monoterpenes predominated at early developmental stages, whereas oxygenated monoterpenes increased markedly during flower maturation. Citronellol reached its highest level at HS8 (25.0%), while geraniol and nerol showed maximum accumulation at HS4 (5.7%) and HS4 (3.2%), respectively. Phenylethyl alcohol increased progressively during flower development and reached 18.7% at HS8. In contrast, sesquiterpenes declined continuously across maturity stages. Farnesol was detected only at mid to late developmental stages and reached its highest level at HS7 (0.4%). Principal component analysis (PCA) explained 81.97% of the total variance and clearly differentiated early, intermediate, and late developmental stages based on volatile composition. Detailed eigenvalues and major loading values are provided in the Supporting Information (Tables S1 and S2). HS7 emerged as the most suitable harvest stage with respect to volatile aroma composition and fragrance-related quality parameters due to its favorable balance of oxygenated monoterpenes and relatively low methyl eugenol levels, consistent with ISO 9842 rose oil specifications. These findings provide a chemical basis for optimizing harvest timing of *Rosa damascena* flowers cultivated under semiarid ecological conditions.

**Keywords:** *Rosa damascena*, volatile composition, HS-SPME-GC/MS, ontogenetic variation, flower development, semiarid conditions

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## 1 Introduction

The timing of flower harvest is a critical factor affecting the composition and quality of volatile compounds, which ultimately determines the commercial value of aromatic plant products. Volatile constituents derived from aromatic plants are widely used in perfumery, cosmetics, food flavoring, and therapeutic applications due to their complex chemical profiles and beneficial properties (Buchbauer & Başer, 2010). *Rosa damascena* Mill. is one of the most important aromatic plant species used for the production of rose oil and rose water, which are widely utilized in perfumery, cosmetics, and pharmaceutical preparations. The characteristic fragrance of rose oil is mainly determined by oxygenated monoterpenes such as citronellol, geraniol, and nerol together with phenylpropanoid derivatives. The chemical composition of rose volatiles is influenced by several factors, including genotype, environmental conditions, harvest timing, and flower developmental stage (Baydar et al., 2013; Sangwan et al., 2001; Pal et al., 2016; Izgi, 2022).

Volatile compounds in aromatic plants undergo significant changes during ontogenetic development. For example, in Salvia plebeia, the volatile profile of aerial parts shifts from sesquiterpene hydrocarbons toward oxygenated compounds during flower maturation (Singh et al., 2024). Similarly, *Spiranthera odoratissima* flowers exhibit developmental variations in volatile composition dominated by $\beta$-caryophyllene and spathulenol depending on maturity stage (Cabral et al., 2019). In addition, studies on Teucrium species demonstrated that volatile composition is strongly influenced by both developmental and environmental factors, highlighting the importance of ontogenetic regulation of secondary metabolite biosynthesis (Hanoğlu et al., 2023).

Despite extensive research in traditional rose oil production regions such as Isparta (Türkiye) and Kazanlak

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(Bulgaria), information on ontogenetic variation of volatile composition in *R. damascena* grown under semiarid ecological conditions remains limited, particularly for emerging production areas such as Mardin, Türkiye. Headspace solid-phase microextraction (HS-SPME) coupled with gas chromatography–mass spectrometry (GC–MS) provides a rapid, solvent-free, and sensitive technique for monitoring stage-dependent qualitative changes in floral volatile composition throughout flower development (Erbaş & Baydar, 2016; Dobreva, 2013). However, detailed information on stage-dependent shifts between monoterpene hydrocarbons, oxygenated monoterpenes, sesquiterpenes, and long-chain hydrocarbons in *R. damascena* flowers under semiarid conditions is still lacking.

Earlier studies conducted in Mardin demonstrated that seasonal variations and harvest timing significantly influence volatile composition of *R. damascena* flowers, particularly affecting oxygenated monoterpenes and phenylethyl alcohol content (Kataoka et al., 2000). Therefore, the present study aimed to characterize ontogenetic variation of volatile constituents in *R. damascena* flowers harvested at eight developmental stages (HS1–HS8) using HS-SPME-GC/MS and to evaluate stage-dependent changes in major compound classes in order to identify the optimal harvest stage for aroma quality in semiarid production conditions.

## 2 Materials and Methods

### 2.1 Plant Material and Study Site

The study was carried out in Yaylabaşı village (37°24′41″ N, 40°47′9″ E; 1120 m a.s.l.), Mardin Province, southeastern Türkiye. Oil-bearing rose (*Rosa damascena* Mill.) has been cultivated in this region for about seven years, representing a newly emerging production area. The plantation covers about 60 ha, and flower samples were collected during the 2024 flowering season (mid-May to mid-June). Flowers were harvested at eight developmental stages (HS1–HS8; harvest stages) representing different phenological phases of flower development. A voucher specimen (RD-2024) was deposited in the Herbarium of the Department of Field Crops, Faculty of Kızıltepe Agricultural Sciences and Technologies, Mardin Artuklu University, Türkiye.

Soil samples collected from 0–60 cm soil depth showed a loam texture with low organic matter content (1.68%), slightly alkaline pH (7.5–8.5), relatively high nitrogen levels (0.19–0.33%), and low concentrations of P, K, Ca, Mg, and micronutrients, reflecting typical semiarid soil conditions.

### 2.2 Experimental Design and Sampling

The experiment followed a randomized design with three replications. Each replicate consisted of a single plant row (12 m length, six shrubs). To avoid pseudoreplication, flowers were collected from different shrubs within each row. Sampling was conducted in the early morning (06:00) on 25 May 2024, when the average temperature and relative humidity were 10.8°C and 50%, respectively.

Flowers were harvested at eight distinct developmental stages (HS1–HS8), defined based on morphological characteristics:

- HS1: fully closed sepals
- HS2: slightly open sepals
- HS3: fully open sepals with tightly coiled petals
- HS4: petals beginning to open from the apex
- HS5: half-open petals with sepals parallel to the ground
- HS6: fully open petals, drooping sepals, visible stamens
- HS7: fully open flowers with slightly faded petals and yellow–brown stamens
- HS8: senescent flowers with decayed petals

### 2.3 Sample Preparation

Freshly harvested flowers were immediately transported to the laboratory in sealed thermos containers at 4°C. For each replicate, flowers collected from multiple shrubs were pooled (Kataoka et al., 2000; Snow & Slack, 2002). Approximately 5 g of fresh rose petals were placed in a 20 mL glass vial and sealed with a PTFE/silicone septum. The petals were gently crushed to facilitate volatile release prior to HS-SPME extraction.

### 2.4 HS-SPME–GC/MS Analysis

Volatile analysis was performed using a Shimadzu GC-2010 Plus gas chromatograph coupled with a Shimadzu GC–MS-QP2020 mass spectrometer. Separation was achieved on a DB-HeavyWax capillary column (60 m × 0.25 mm, 0.25 μM). HS-SPME extraction was conducted using a DVB/CAR/PDMS fiber (50/30 μM, 2 cm; Supelco, USA), exposed to the headspace at 45°C for 50 min. Relative percentages of volatile compounds were calculated from GC–MS total ion chromatogram peak areas without correction factors and therefore represent semi-quantitative estimates intended for comparative profiling of volatile composition across developmental stages. The present study was designed for comparative profiling of volatile composition across developmental stages rather than absolute quantification of distilled rose oil components.

Although flame ionization detection (FID) is commonly recommended for quantitative GC analyses, relative peak area normalization obtained from GC–MS total ion chromatograms is widely used in volatile profiling studies when the objective is comparative characterization rather than absolute quantification. This approach is commonly accepted for comparative profiling of plant volatiles when authentic reference standards are not available. In the present study, all samples were analyzed under identical experimental conditions and the results are therefore reported as relative peak area percentages intended for comparative evaluation of ontogenetic changes in volatile composition. This semi-quantitative approach is commonly applied in HS-SPME-GC/MS studies of plant volatiles and is suitable for monitoring relative variations in compound abundance across developmental stages.

The GC oven temperature program was as follows: initial temperature 40°C, increased at 3°C/min to 80°C (held for 1 min), then increased at 5°C/min to 240°C (held for 6 min).


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The injector temperature was set at 250°C, and helium was used as the carrier gas at a flow rate of 1.05 mL/min.

Compound identification was performed by comparison of calculated retention indices with literature values reported for polar columns. Retention indices were further verified using published reference values (Goodner, 2008; Kim et al., 2000; Wu et al., 2009). A representative GC chromatogram of the identified volatile compounds is provided in the Supplementary Material (Figure S1).

### 2.5 Statistical Analysis

Data were analyzed using one-way analysis of variance (ANOVA) with JMP® 14.0 software (SAS Institute, Cary, NC, USA). Normality and homogeneity of variance were evaluated using the Shapiro–Wilk test (Shapiro & Wilk, 1965) and Levene's test (Levene, 1960). Mean separations were performed using the LSD test at $p \leq 0.05$.

For multivariate analysis, principal component analysis (PCA) was conducted using Genstat (12th edition). Dataset suitability was confirmed using Bartlett's test (Bartlett, 1951) and the Kaiser–Meyer–Olkin measure (Kazaz et al., 2009). Prior to PCA, volatile compound data were autoscaled by mean-centering and scaling to unit variance in order to minimize the influence of differences in compound abundance.

## 3 Results and Discussion

### 3.1 Volatile Composition across Flower Development

Using HS-SPME-GC/MS, a total of 39 volatile constituents were identified in *Rosa damascena* Mill. flowers harvested at eight maturity stages (HS1–HS8) (Feng et al., 2010). Identified compounds were classified into monoterpenes (MT), oxygenated monoterpenes (OMT), sesquiterpenes (ST), oxygenated sesquiterpenes (OST), long-chain hydrocarbons (LCH), and other components (OC) (Table 1). The distribution of these compound classes across developmental stages is illustrated in Figure 1, while representative developmental stages are presented in Figure 2. The relative abundance of these chemical classes changed markedly throughout flower development, indicating strong ontogenetic regulation of volatile biosynthesis under semiarid conditions. Early developmental stages were dominated by monoterpene hydrocarbons and sesquiterpenes, whereas oxygenated monoterpenes progressively increased during flower maturation.

Early developmental stages (HS1–HS3) were characterized by a dominance of monoterpenes and sesquiterpenes, which are commonly associated with primary defensive and structural functions in young floral tissues. As flower maturation progressed, a pronounced shift toward oxygenated monoterpenes and long-chain hydrocarbons was observed, reflecting enhanced metabolic diversification and aroma specialization. The overall percentage of identified volatile compounds increased progressively from HS1 to HS6 and remained high through HS8, suggesting intensified secondary metabolic activity during advanced floral development.

### 3.2 Monoterpenes and Oxygenated Monoterpenes

Monoterpenes constituted the dominant fraction of the volatile profile at early developmental stages (HS1–HS2), accounting for nearly half of the total volatile content, but declined sharply with flower maturation. By HS8, monoterpenes represented less than one-quarter of the total volatile fraction. This decrease reflects progressive oxidative biotransformation of hydrocarbon monoterpenes into oxygenated derivatives during flower maturation

In contrast, oxygenated monoterpenes—particularly citronellol, geraniol, and nerol—showed a pronounced increase after HS3 and reached their maximum levels at HS7. These compounds are widely recognized as key contributors to rose fragrance quality and are used as primary markers in rose oil quality evaluation and standardization (Goodner, 2008). Their accumulation at later stages suggests increased activity of terpene-modifying enzymes, such as alcohol dehydrogenases, during advanced floral development. This transition from hydrocarbon monoterpenes to oxygenated monoterpenes reflects increased oxidative modification of terpene precursors during flower maturation, a key biochemical process contributing to the formation of characteristic rose fragrance compounds.

Similar ontogenetic trends have been reported in *Salvia pinnata*, where early-stage oils were dominated by monoterpenes (e.g., $\alpha$-pinene), followed by increased accumulation of oxygenated monoterpenes at later stages (Singh et al., 2024). The present findings confirm that flower maturity plays a decisive role in shaping the balance between hydrocarbon and oxygenated terpenes in *R. damascena*.

The ontogenetic trends observed in the present HS-SPME-GC/MS study are generally consistent with previously published hydrodistillation studies on *Rosa damascena*. Kazaz et al. (2009) reported significant developmental changes in rose oil composition, particularly involving citronellol, geraniol, and nerol, which increased during flower maturation. Similarly, Erbaş and Baydar (2016) demonstrated that although HS-SPME and hydrodistillation differ in extraction principles and therefore may yield different quantitative proportions, both approaches consistently identify oxygenated monoterpenes as the principal fragrance-related constituents of mature flowers. The increasing accumulation of citronellol, geraniol, and nerol observed from HS4 onwards in the present study therefore agrees with previously reported hydrodistillation-based findings and supports the biological significance of the identified developmental trends.

### 3.3 Sesquiterpenes and Oxygenated Sesquiterpenes

Sesquiterpenes, including $\beta$-bourbonene, germacrene D, and $\alpha$-guaiene, were abundant during early flowering stages but declined progressively with flower maturation. At HS1, sesquiterpene levels were substantially higher than those observed at HS8. Such early-stage dominance of sesquiterpenes may be linked to their ecological roles in plant defense and stress tolerance.

**Table 1.** Volatile composition of *Rosa damascena* Mill. flowers at eight developmental stages (HS1–HS8) determined by HS-SPME-GC/MS

| Compound | RI (calc) | RI (lit) | Identification | HS1 | HS2 | HS3 | HS4 | HS5 | HS6 | HS7 | HS8 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| α-Pinene | 1120 | 1035[13] | MS, RI | 24.3 | 30.3 | 18.8 | 7.3 | 7.3 | 8.7 | 7.0 | 3.0 |
| β-Pinene | 1203 | 1118[13] | MS, RI | 4.1 | 4.4 | 2.3 | 1.1 | 1.1 | 1.2 | 1.6 | 2.2 |
| β-Phellandrene | 1219 | 1245[13] | MS, RI | 2.4 | 2.4 | 0.5 | 0.7 | 0.7 | 0.9 | 1.1 | 1.7 |
| β-Myrcene | 1264 | 1158[13] | MS, RI | 9.3 | 8.6 | 1.5 | 2.1 | 1.9 | 2.3 | 4.1 | 4.3 |
| α-Terpinene | 1279 | 1184[13] | MS, RI | 0.4 | 0.4 | nd | nd | nd | nd | 0.1 | 0.1 |
| Limonene | 1298 | 1208[13] | MS, RI | 0.8 | 0.6 | 0.1 | 0.1 | 0.1 | 0.2 | 0.2 | 0.3 |
| Trans-Sabinene hydrate | 1308 | 1308[13] | MS, RI | 0.1 | 0.3 | nd | 0.1 | 0.1 | nd | 0.1 | 0.1 |
| γ-Terpinene | 1348 | 1249[13] | MS, RI | 0.8 | 0.7 | nd | 0.1 | nd | 0.1 | 0.2 | 0.2 |
| β-cis-Ocimene | 1355 | 1355[13] | MS, RI | 0.5 | 0.4 | nd | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 |
| α-Terpinolene | 1385 | 1290[13] | MS, RI | 0.2 | 0.2 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Trans-Rose oxide | 1457 | 1370[14] | MS, RI | nd | nd | nd | 0.2 | 0.3 | 0.2 | 0.2 | 0.2 |
| cis-Rose oxide | 1472 | 1356[14] | MS, RI | nd | nd | nd | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Rosefuran | 1507 | 1507[14] | MS, RI | 0.1 | 0.2 | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 | 0.2 |
| Citronellal | 1587 | 1492[13] | MS, RI | 0.1 | 0.1 | 0.3 | nd | 0.1 | 0.1 | nd | 0.1 |
| β-Bourbonene | 1631 | 1624[13] | MS, RI | 1.1 | 1.1 | 0.9 | 0.4 | 0.2 | 0.1 | 0.3 | 0.1 |
| Linalool | 1648 | 1544[14] | MS, RI | 0.5 | 0.3 | 0.5 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 |
| Hexadecane | 1698 | 1600[13] | MS, RI | 0.4 | 0.3 | nd | 0.7 | 0.6 | 0.7 | 0.4 | 0.1 |
| Heptadecane | 1700 | 1700[13] | MS, RI | nd | 3.0 | 5.8 | 3.6 | 2.7 | 3.2 | 3.7 | 0.5 |
| α-Guaiene | 1703 | 1702[13] | MS, RI | 8.8 | 5.7 | nd | 2.0 | 1.4 | 0.3 | 1.2 | 0.4 |
| Citronellol acetate | 1770 | 1639[13] | MS, RI | 0.1 | 0.1 | 0.2 | 2.3 | 2.2 | 0.9 | 0.8 | 1.1 |
| Humulene | 1790 | 1787[13] | MS, RI | 4.8 | 3.2 | 2.5 | 1.4 | 0.9 | nd | 0.8 | 0.3 |
| cis-3-Heptadecene | 1821 | 1821[15] | MS, RI | 1.4 | 1.5 | 14.7 | 12.5 | 11.4 | 16.4 | 9.2 | 4.9 |
| Germacrene-D | 1829 | 1827[13] | MS, RI | 18.8 | 14.2 | 11.9 | 5.0 | 3.9 | 0.9 | 2.5 | 1.2 |
| α-Bulnesene | 1834 | 1831[13] | MS, RI | 4.6 | 3.1 | nd | 1.5 | 0.8 | 0.3 | 0.8 | 0.3 |
| Citral (geranial + neral) | 1843 | 1840[13] | MS, RI | 0.5 | 0.2 | 0.3 | 0.7 | 0.2 | 0.4 | 0.3 | 0.2 |
| α-Farnesene | 1855 | 1856[13] | MS, RI | 1.1 | 0.5 | 0.6 | 0.3 | 0.4 | 0.3 | 0.2 | 0.1 |
| Citronellol | 1865 | 1768[14] | MS, RI | 0.1 | nd | nd | 17.5 | 20.6 | 23.6 | 18.1 | 25.0 |
| Octadecane | 1899 | 1800[13] | MS, RI | 0.1 | 0.1 | 1.2 | 0.6 | 0.5 | 0.6 | 0.4 | 0.3 |
| Nerol | 1902 | 1792[14] | MS, RI | nd | 0.1 | 0.2 | 3.2 | 0.4 | 2.6 | 1.5 | 1.2 |
| Phenethyl acetate | 1933 | 1785[13] | MS, RI | nd | nd | nd | 0.4 | 0.2 | 0.1 | 0.1 | 0.2 |
| Geraniol | 1947 | 1852[14] | MS, RI | nd | 0.1 | nd | 5.7 | 1.5 | 4.6 | 5.0 | 2.7 |
| Heneicosane | 2001 | 1995[13] | MS, RI | 0.1 | 1.1 | 23.7 | 20.3 | 15.2 | 21.6 | 18.0 | 17.6 |
| Phenylethyl alcohol | 2022 | 1955[13] | MS, RI | 0.3 | 3.5 | 1.4 | 0.1 | 7.8 | 0.2 | 8.1 | 18.7 |
| Nonadecene | 2036 | 2014[13] | MS, RI | nd | nd | 0.7 | 0.1 | 8.4 | 0.2 | 2.6 | nd |
| Methyl eugenol | 2119 | 2035[13] | MS, RI | nd | nd | 0.3 | 0.7 | 1.6 | 1.1 | 1.8 | 1.9 |
| Eicosane | 2200 | 2000[13] | MS, RI | nd | nd | 2.1 | 2.3 | 2.0 | 2.4 | 1.4 | 3.6 |
| Eugenol | 2285 | 2164[13] | MS, RI | nd | 0.3 | 0.4 | 1.0 | 1.2 | 0.5 | nd | 1.8 |
| Hexatriacontane | 2400 | 2400[13] | MS, RI | 0.1 | 0.2 | 1.6 | 0.2 | 0.3 | 0.3 | 0.1 | 0.4 |
| Farnesol | 2460 | 2452[13] | MS, RI | nd | nd | nd | 0.2 | 0.2 | 0.2 | 0.4 | 0.1 |

Note: Values represent mean relative percentages (n = 3) of total peak area obtained from three replicate analyses. Relative percentages were calculated from GC–MS total ion chromatogram peak areas and represent semi-quantitative data. Identification of compounds was based on comparison of mass spectra with NIST libraries and retention indices relative to n-alkanes (C8–C40) with literature values reported for polar stationary phases. RI (calc): retention indices experimentally calculated relative to n-alkanes on a DB-HeavyWax column. RI (lit): retention indices reported in the literature; superscript numbers indicate reference sources. HS: flower harvest maturity stages (HS1–HS8). nd: not detected. Reference sources for RI(lit): [13]–Goodner (2008); [14]–Kim et al. (2000); [15]–Wu et al. (2009).

This pattern is consistent with previous observations in Teucrium species, where sesquiterpene-rich profiles were primarily associated with early flowering phases (Hanoğlu et al., 2023). The gradual decline of sesquiterpenes with maturation indicates a metabolic reallocation toward compounds with greater sensory relevance.

Oxygenated sesquiterpenes were represented exclusively by farnesol, which was detected only from HS4 onward and reached its highest concentration at HS7 (0.36%). Farnesol is of particular interest due to its documented anti-inflammatory and anticancer activities (Jung et al., 2021). Its selective accumulation at mid to late developmental


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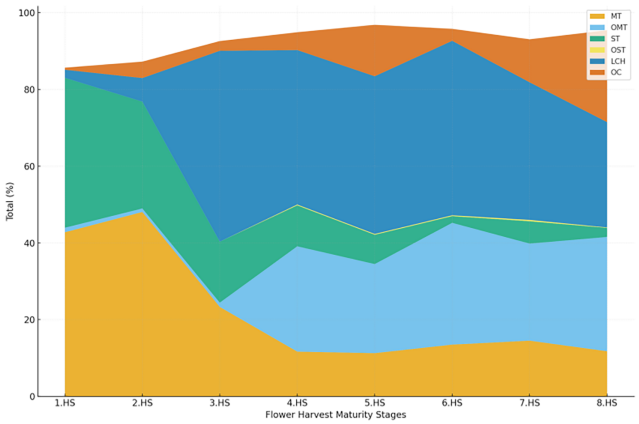


**Figure 1.** Stacked area chart showing the relative distribution of volatile compound classes (MT, OMT, ST, OST, LCH, and OC) across flower harvest maturity stages (HS1–HS8) in *Rosa damascena*. Percentages represent the relative contribution of each compound class based on identified compounds listed in Table 1


**Figure 2.** Developmental stages of *Rosa damascena* flowers (HS1–HS8), ranging from fully closed sepals (HS1) to senescent petals (HS8)

stages suggests that rose oil harvested at HS7 may offer additional functional relevance beyond fragrance attributes.

### 3.4 Long-Chain Hydrocarbons and Other Components

Long-chain hydrocarbons exhibited variable accumulation patterns across maturity stages. Compounds such as heptadecane and heneicosane increased notably during intermediate stages (HS3–HS6), likely reflecting changes in cuticular wax composition and adaptive responses to semiarid environmental conditions.

Among other components, phenylethyl alcohol showed a steady increase throughout flower development and reached its highest concentration at HS8. This compound is a major contributor to the characteristic rose scent and is particularly valued in perfumery applications. In contrast, methyl eugenol remained at moderate levels during commercially relevant stages (HS5–HS7), which is advantageous given existing regulatory restrictions on this compound in food and fragrance products (European Commission Health & Consumer Protection Directorate-General, 2001).

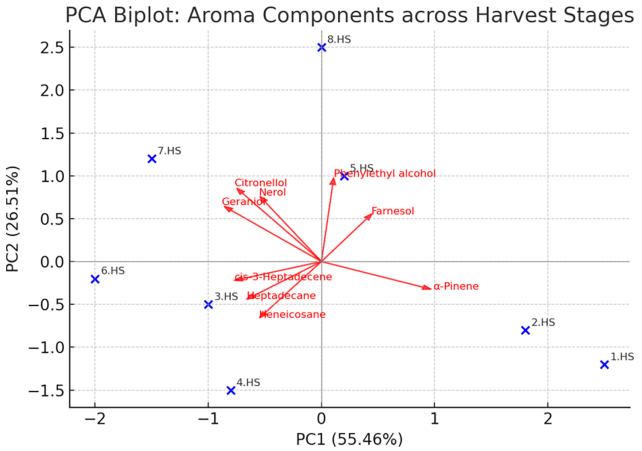


**Figure 3.** PCA biplot showing relationships between volatile compounds and harvest stages (HS1–HS8)

### 3.5 Multivariate Analysis and Optimal Harvest Stage

Principal component analysis (PCA) explained 81.97% of the total variance, with PC1 and PC2 accounting for 55.46% and 26.51% of the variance, respectively (Figure 3). PC1 was mainly associated with oxygenated monoterpenes, particularly citronellol, geraniol, nerol, and phenylethyl alcohol, whereas α-pinene, germacrene-D, and other sesquiterpenes contributed negatively to PC1. PC2 was primarily influenced by long-chain hydrocarbons, including heneicosane and cis-3-heptadecene. PCA clearly separated early developmental stages (HS1–HS3), intermediate stages (HS4–HS6), and late stages (HS7–HS8) according to differences in volatile composition. Prior to PCA, data were autoscaled by mean-centering and scaling to unit variance.

HS7 showed strong positive loadings with oxygenated monoterpenes and farnesol, indicating an optimal combination of aromatic richness and biochemical diversity. Notably, this stage also aligned closely with ISO 9842 rose oil quality standards, particularly ISO 9842 standards regarding citronellol and phenylethyl alcohol contents (International Organization for Standardization, 2003; Staikov et al., 1975). Comparable stage-dependent optimization of essential oil quality has been reported for *Spiranthera odoratissima*, where specific flowering stages were identified as optimal harvest points based on volatile composition (Cabral et al., 2019).

### 3.6 Optimal Harvest Stage Determination

Taken together, these results demonstrate that flower maturity exerts a decisive influence on the volatile composition of *R. damascena*. Under semiarid conditions in Mardin, HS7—characterized by fully open petals and yellow–brown stamens—emerged as the most suitable harvest stage with respect to volatile aroma composition and fragrance-related quality characteristics. A comparative evaluation of major volatile compounds at HS7 relative to ISO 9842 specifications and literature values is presented in Figure 4. Harvesting at this stage maximizes the concentration of quality-defining oxygenated monoterpenes while maintaining relatively low levels of methyl eugenol.

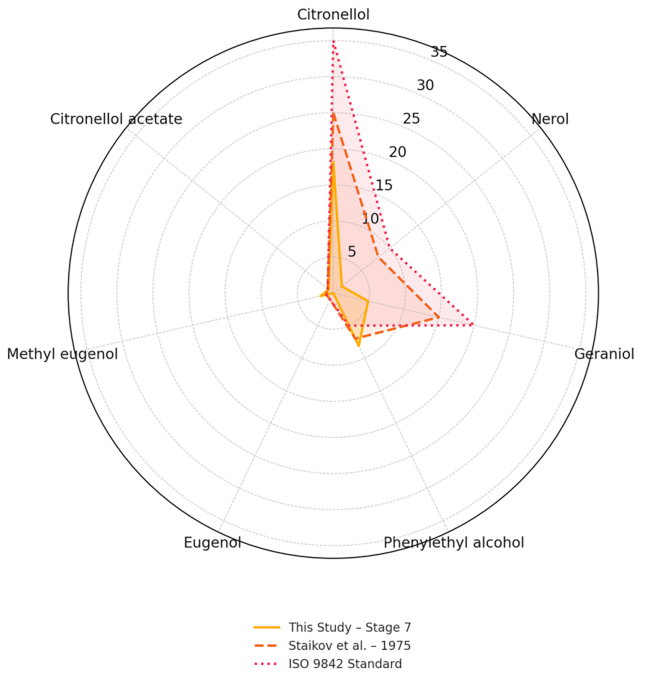


**Figure 4.** Comparative profile of seven major volatile compounds in *Rosa damascena* at HS7 (this study) compared with Staikov et al. (1975) and ISO 9842 standards

The ontogenetic trends observed in the present HS-SPME-GC/MS study are generally consistent with previously published hydrodistillation studies on *Rosa damascena*. Earlier investigations reported increasing proportions of oxygenated monoterpenes, particularly citronellol, geraniol, and nerol, during flower maturation, accompanied by changes in the relative abundance of other volatile groups. Although HS-SPME-GC/MS and hydrodistillation differ in extraction principles and therefore may yield different quantitative proportions, both approaches consistently identify oxygenated monoterpenes as key contributors to rose fragrance quality. The marked increase in citronellol, geraniol, and nerol observed after HS3 and their maximum accumulation at HS7 therefore support the biological and practical relevance of this developmental stage.

From the perspective of volatile aroma quality, targeting HS7 may provide growers with a useful guideline for improving fragrance characteristics and maintaining compliance with international rose oil quality standards in emerging semiarid cultivation regions. However, the designation of HS7 as the most suitable harvest stage in the present study is based exclusively on volatile aroma composition and fragrance-related quality parameters obtained by HS-SPME-GC/MS analysis.

It should be emphasized that the designation of HS7 as the most suitable harvest stage in the present study is based exclusively on volatile aroma composition and fragrance-related quality parameters obtained by HS-SPME-GC/MS analysis. Additional investigations involving hydrodistilled essential oil yield, rose oil recovery, and economic performance would be required to establish a comprehensive recommendation for commercial harvest optimization.

## 4 Conclusion

This study demonstrated pronounced ontogenetic variation in the volatile composition of *Rosa damascena* Mill. flowers cultivated under semiarid conditions in Mardin, Türkiye. HS-SPME-GC/MS analysis revealed significant stage-dependent shifts among major volatile compound classes during flower development. Early developmental stages were characterized by high levels of monoterpenes and sesquiterpenes, whereas oxygenated monoterpenes, particularly citronellol, geraniol, and nerol, increased markedly during flower maturation. The selective accumulation of farnesol and the progressive increase of phenylethyl alcohol further highlighted substantial biochemical differentiation among developmental stages. Multivariate analysis clearly separated developmental stages according to volatile composition and identified HS7 as the most suitable harvest stage with respect to volatile aroma composition and fragrance-related quality characteristics, due to its favorable balance of oxygenated monoterpenes and relatively low methyl eugenol levels. These findings provide practical guidance for optimizing harvest timing of *R. damascena* flowers cultivated under semiarid ecological conditions and contribute to a better understanding of ontogenetic regulation of floral volatile biosynthesis.

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## Author Contributions

The author solely contributed to the conception, design, experimental work, data analysis, and writing of the manuscript.

## Availability of Data and Materials

The authors declare that the data supporting the findings of this study are available within the paper and its Supplementary Information files. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request. Source data are provided with this paper.

## Conflict of Interest

The authors declare that they do not have any conflict of interest.

## Supporting Information

Supporting Information accompanies this paper on http://www.acgpubs.org/journals/records-of-natural-products.

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