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# Chemometrics-Based Aroma Discrimination of 14 Egyptian Mango Fruits of Different Cultivars and Origins, and Their Response to Probiotics Analyzed via SPME Coupled to GC−MS

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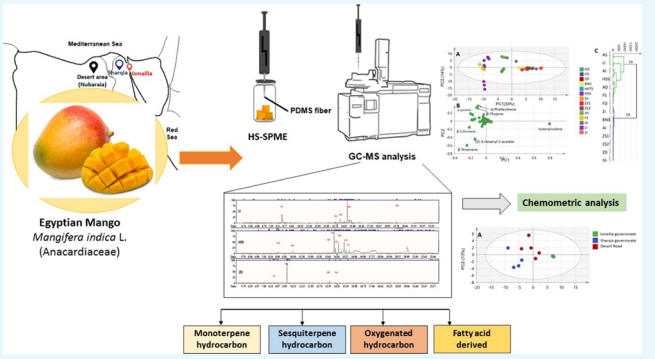
**ABSTRACT:** The present study investigated the volatile organic compounds (VOCs) in 14 Egyptian mango specimens collected from three different regions and of different cultivars (cvs). VOCs were extracted via solid-phase microextraction, followed by gas chromatography−mass spectrometry analysis. The results obtained for sesquiterpene hydrocarbons' qualitative abundance were represented by 28 peaks, whereas monoterpene hydrocarbons amounted for the highest levels in most of the mango cvs. Multivariate data analyses were employed for sample classification and identification of markers. Unsupervised principal component analysis revealed that "zebdia" cv from the three origins combined together being enriched in terpinolene. Moreover, supervised orthogonal partial least square-discriminant analysis identified $\beta$-terpinene and (*Z*)-geranylacetone in the premium "awees" cv. The impact of probiotic bacteria on mango juice aroma was further assessed revealing no potential changes in the composition. This study provides the first comprehensive insights into Egyptian mango aroma and reveals that the cv type overcomes the geographical origin in their aroma profile.






## 1. INTRODUCTION

*Mangifera indica* Linn. (family Anacardiaceae) is a tropical climacteric fruit native to Asia, specifically to the Indo-Burmese peninsula.[1,2] Owing to its economic value, nutritional micro- and macroelements, health benefits, and delightful unique taste, ca. 1000 mango varieties are cultivated worldwide including subtropical regions such as Florida, Egypt, southern Latin America, Australia, and Europe.[1,3−5] Based on FAO statistics, the global production of mango is valued at 42 million tons per year in 2015.[1] India is the largest producer with 1,525,000 tons per year, while Mexico is the largest exporter with 287,771 tons per year.[6] Interestingly, Egypt is among the non-traditional geographical region producers, with a steep increase in its export market in the last few years.[2]

Phytochemical analysis of different mango fruits confirmed their richness in essential nutrients, that is, amino acids, minerals, vitamins, sugars, and organic acids. In addition, various plant parts are rich in phytonutrients including phenolic acids, that is, ferulic, protocatechuic, chlorogenic, and caffeic acids; polyphenols and flavonoids, that is, mangiferin, gallic acid, gallotannins, quercetin, isoquercetin, ellagic acid, and $\beta$-glucogallin; carotenoids, that is, carotenes and xanthophylls; and volatile constituents, in addition to structural polysaccharides, that is, pectin and cellulose.[5,7−9] These constituents contribute to a wide array of pharmacological activities, including antioxidant, anti-inflammatory, antidiabetic, hypolipidemic, and anticancer among others.[1,10] Nevertheless, such a complex chemical composition was found to be significantly influenced by biochemical, physiological, and structural changes that occur during fruit development and affect not only fruit health benefits but rather their sensory characters and consequently consumer acceptance.[5,11,12]

Particularly, fruit aroma depends mainly on the qualitative and quantitative abundance of volatile organic compounds (VOCs) comprised in mango fruits such as esters, alcohols, aldehydes, ketones, lactones, terpenoids, volatile fatty acids, and apocarotenoids.[13] Differences in VOCs were reported in the context of genotypic variations and phenotypic plasticity among cultivars (cvs).[14] Other factors affecting mango fruits' VOCs include fruits' geographic origins, postharvest storage conditions, and preprocessing methods.[15−17] In addition, VOCs of mangos have been used as an identification marker at different maturity stages of different cvs. Consequently, VOCs can be used as optimization factors and determinants of fruit ripening and harvesting stages.[18,19] Previous reports

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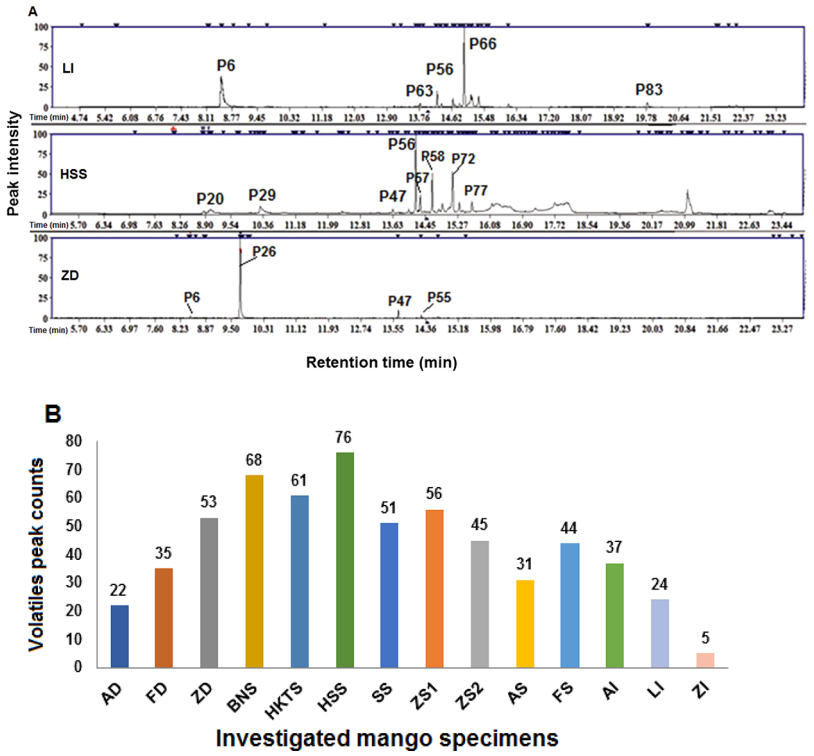


**Figure 1.** (A) Representative chromatograms of three Egyptian mango cultivars from three different geographical origins, that is, Ismailia (LI), Sharqia (HSS), and the desert area in Nubaria (ZD). Peak numbering follows that listed in Table 2. (B) Volatiles peak count per each mango sample. Most of the mango cvs. collected from Sharqia contain a lot of VOCs, including the "hendy sinara" Sharqia (HSS) with 76 compounds followed by "balady nabila" Sharqia (BNS) and "hendy kalb el toor" Sharqia (HKTS) with 68 and 61 compounds, respectively. In contrast, Zebdia from the Ismalia governorate (ZI) recorded the least cultivar with only five volatile compounds. Sample codes follow that listed in Table 1.

demonstrated that more than 285 VOCs were identified in different mango varieties, with monoterpenes as the major class contributing to mango flavors, such as terpinolene, $\delta$-3-carene, $\alpha$-pinene, $\beta$-myrcene, $\beta$-ocimene, and limonene.[5,15,20]

Head space-solid-phase microextraction (HS-SPME) was developed as an alternative preconcentrating step to the dynamic headspace prior to analysis by gas chromatography (GC). The technique was furtherly enhanced through coupling with mass spectrometry (MS) as a detection technique. It has been applied since the early 2000s for a comprehensive isolation and analysis of aromas of different food and foodstuffs, including virgin olive oils,[21] and water as well.[22] In comparison with liquid−liquid extraction and simultaneous distillation-extraction, HS-SPME/GC−MS has been reported as the most valuable and non-destructive analytical technique for profiling of VOCs derived from plant bacteria interactions[23] and herbal and food products.[24,25] However, some drawbacks have been reported, being related mainly to sensitivity, stripping of coating, life times, and a limited number of commercially available stationary phases.[26] Hence, different kinds of adsorbent fibers should be tried as a prerequirement for selection of the best fiber that can facilitate the detection of the highest number of VOCs.

HS-SPME/GC−MS was applied in quality assessment and authentication of fruits including Egyptian palm (*Phoenix dactylifera* L.)[27] and pears (*Pyrus* sp.) based on their aroma.[28] Because the HS-SPMS/GC−MS technique always results in a huge amount of data that should be handled mathematically extracting valuable conclusions, multivariate data analysis (MVA), including the chemometrics tool is applied as a powerful discriminating tool for specimen classification and marker identification.[29−31]

With regard to mango fruits, characterization of Malaysian and Chinese mangoes based on their VOC profile has been recently reported using HS-SPME/GC−MS[32,33] or by solvent extraction, followed by solvent-assisted flavor evaporation applying a comparative aroma extract dilution analysis.[34] Nevertheless, scarce information is available regarding Egyptian mango aroma, with only a single study on Egyptian "baladi" and "alphonso"/"founs" mangoes.[35]

The present study aimed to investigate the sensory properties of different Egyptian mango cvs based on their VOC profile as analyzed using SPME/GC−MS coupled to MVA. Samples included 14 different major Egyptian mango cvs from three different geographical origins. The results highlighted both quantitative and qualitative differences among cvs harvested from three different geographical cultivation regions; namely Sharqia and Ismailia (Nile Delta 2) governorates from the Nile Delta region and the Alexandria desert area (Nubaria region). Mango samples were selected based on their availability within the Egyptian market and popularity, and to have some representative cvs from different origins to assess for geographical origin impacts on aroma. To aid in samples' classification, an untargeted MVA approach was employed to analyze the resulting VOC dataset from 14 samples and to identify the potential markers of each cv if any.

## 2. RESULTS AND DISCUSSION

**2.1. Volatile Identification and Classification.** Experimental design was based on choosing similar/different cvs from different geographical origins for their VOC analysis


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using HS-SPME/GC−MS. Also, two different fibers were assessed prior to the main analytical experiments that include PDMS and DVB/CAR/PDMS. Compared to PDMS, DVB/CAR/PDMS showed a less affinity to mango volatiles with qualitative differences in the number of observed peaks (Figure S1). Consequently, the PDMS type was used for all fruit cvs' analysis, with a total of 92 volatile peaks identified in the GC−MS chromatograms, Figure 1A. Details of the volatile collection method optimization and analysis using SPME coupled to GC/MS have been previously reported.[36]

Apparent differences were observed in the VOC profile among the different mango cvs and to a less extent from the three different locations by visual inspection of the GC−MS chromatograms of representative samples, that is, HSS, LI, and ZD, Figure 1A. The detected volatiles, Table 1, were listed according to their origin, including sesquiterpene hydrocarbon (27), monoterpene hydrocarbon (21), oxygenated monoterpenes (10), oxygenated sesquiterpenes (2), fatty acid-derived volatiles (14), benzenoids/aromatics (4), in addition to diterpene hydrocarbons (2), and aliphatic hydrocarbons (3). Moreover, other miscellaneous (5) and unknown volatiles (4) were distinguished.

**Table 1. List of Collected Mango Samples Including the Location, Code, and Description**

| location | sample code | description |
|---|---|---|
| Sharqia governorate | HKTS | hendy kalb el toor Sharqia |
| | BNS | baladi Nabila Sharqia |
| | HSS | hendy Sinara Sharqia |
| | FS | founs Sharqia |
| | AS | awees Sharqia |
| | ZS1 | zebdia Sharqia 1 |
| | ZS2 | zebdia Sharqia 2 |
| | SS | sokary Sharqia |
| desert area (Nubaria) | FD | founs desert |
| | ZD | zebdia desert |
| | AD | awees desert |
| Ismailia governorate | ZI | zebdia Ismailia |
| | AI | awees Ismailia |
| | LI | langara Ismailia |

Qualitative differences among cvs could be revealed by simple visual inspection of GC-MS chromatograms (Figure 1A), with "hendy sinara" Sharqia (HSS) with 76 compounds being the richest in volatiles versus "zebdia" from Ismalia governorate (ZI) showing the least number of peaks (five compounds), Figure 1B.

Moreover, peaks representing terpene hydrocarbons, that is, monoterpenes and sesquiterpenes, accounted for more than 90% of the peaks in most of the Egyptian mango cvs, Figure 2, which is in accordance with the results of Pino and Mesa who investigated the VOCs in 20 mango cvs harvested from the National Botanic Garden in Havana.[37] Such a pattern was not identified in the case of "awees" cv, including AS and AD displaying more percentiles of miscellaneous, specifically carboxylic acid ester (62%) and oxygenated monoterpene hydrocarbons (30%), respectively, in their volatile blend, rationalizing why "awees" cv is a premium one in the Egyptian market. It is also worth noting that ZI and AI were the richest in monoterpene hydrocarbons with 99.8 and 97%, respectively, while AD in oxygenated monoterpenes with 31%.

In more details, variations of each class of VOCs shall be discussed among the investigated Egyptian mango fruit cvs in the following subsections.

*2.1.1. Monoterpene Hydrocarbons.* Overall, 21 monoterpene hydrocarbons were found to be abundant in most of the Sharqia and Ismailia cvs, that is, HKTS, SS, ZS2, AI, LI, and ZI, at a percentile level of more than 80%, except for AS,

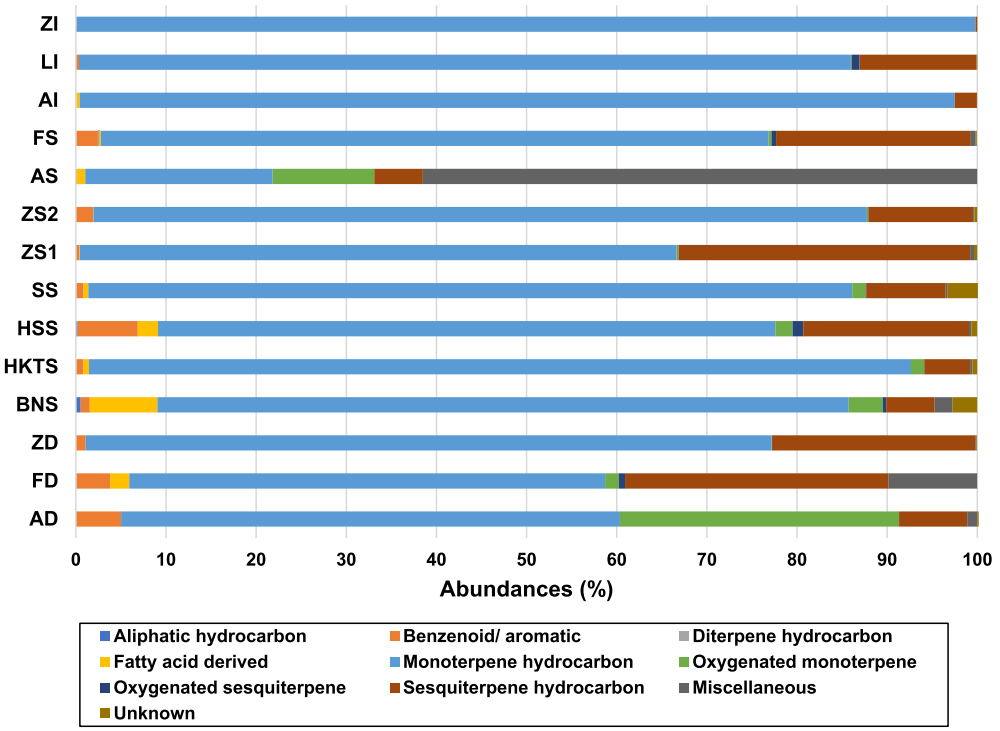


**Figure 2.** Volatile constituents per each sample of mango. Mostly, monoterpene hydrocarbon represents the major constituents, followed by sesquiterpene hydrocarbon. Sample codes follow that listed in Table 1.


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BNS, HSS, FS, and ZS1, in addition to the three samples of the desert area in Nubaria, that is, AD, FD, and ZD. Terpinolene (P26), which is the primary constituent of the mango fruit to impart a citrus and green note, recognized as the characteristic aroma of the New World Mangoes[13] is found to be predominant in many of the cvs. For instance, terpinolene was found at high levels in most cvs ranging from 20 to 98%, except in the case of FD, FS, HSS, and LI being found at trace levels, Table 2. These cvs were reported to have Indian origins showing the same pattern of Indian originated fruits.[38] Another volatile of interest is $\beta$-terpinene (P25), found mainly in AI and HSS and AI at 39.2 and 19.4%, respectively, and at a trace level in "founs" cv regardless of its geographic origin, that is, FD and FI, Table 2, and in agreement with the report by Kulkarni et al.[39]

Moreover, $\alpha$-pinene (P6), a pine-like odorous compound with a low odor threshold, was identified in all cvs, but found to be more abundant in FS, LI, and FD at 17−9% providing another evidence that the geographical origin did not much affect Egyptian mango cvs' aroma. Similarly, $\beta$-thujene (P11) was abundant in "founs" cvs, that is, FS and FD at ca. 12% suggesting no origin effect on aroma, whereas LI cv showed a much higher level of 46%. Additionally, $\beta$-myrcene (P7) imparts a terpenyl/rosin-like fragrance characteristic of "founs"/"alphonso" mango from India[20] and is found at 8−14% in Egyptian "founs", that is, FD and FS. Both $\beta$-myrcene and $\beta$-ocimene are the chief aroma compounds of green mangoes with their warm, herbaceous, and floral odor found herein in several cvs of Indian origins, that is, HSS, FS, and FD in addition to a local cv ("baladi"). Although $\beta$-phellandrene (P18) was detected at 26.5% in LI compared to other cvs, its contribution to mango fruit aroma is rather minimal based on its odor activity value (OAV) according to Pino et al.[9] OAV is a valuable measurement of the potency of a specific volatile compound on the overall odor of a food matrix, where values inferior to 1 mean that its concentration will be lower than the detection threshold.

*2.1.2. Oxygenated Monoterpene Hydrocarbons.* Oxygenated monoterpenes are known to be potentially involved in fruit fragrance.[40] It was found at higher abundances in the "awees" samples with 31 and 11% in AD and AS, respectively. Ten compounds were identified, but they were in trace abundances in all investigated cvs, except for the AD and AS owing to their richness in geranyl acetone (P 59) and hotrienol (P 24) with 29.5 and 11%, respectively. Interestingly, geranyl acetone is a well-known terpenoid flavor,[13] and for the first time it has been reported in mango cvs from Egypt. The richness of geranyl acetone in AD is strong evidence of the premium and different quality of "awees" cv. Whether these mango aroma compounds can be identified from other resources or origins needs further analysis.

Also, it was worthy of note that the compounds such as cineole and of this class were nearly absent in all Ismailia cvs, that is, AI, LI, and ZI, shown in Table 2, proving that they are affected by the geographical origin.

*2.1.3. Sesquiterpene Hydrocarbons.* Sesquiterpene hydrocarbons were the second most abundant volatile class in most of the Nubaria and Sharqia samples, that is, FD, ZD, HSS, SS, ZS1, ZS2, BNS, and FS at 5.4−32%. A pattern that was observed in all "awees" cvs from the three origins, that is, AD, AS, and AI, was their much lower sesquiterpene levels ranging from 2.5 to 7.5%, which suggest that they are not the major components in that premium mango fruit cv.

$\alpha$-Humulene (P 58) was the major component found at the highest levels in the "founs" cvs, that is, FD and FS, at ca. 9−14% to attribute a wood-spicy aroma and known to have a variable odor potency in a mango flavor based on its OAV value. Other less-abundant sesquiterpenes detected at lower levels mostly in "baladi" cv (ZD and ZS) included $\alpha$-ylangene, $\alpha$-cubebene, and copaene at 4−6%.

*2.1.4. Benzenoid/Aromatic Compounds.* Benzenoid/aromatic compounds were present in all cvs with quantitative differences, except for ZI cv. Identified VOCs included $\alpha$-methyl phenethyl alcohol, benzyl ethyl ether, 5-methoxyindane, and (*E*)-1-methyl-2-phenylethenyl acetate, of which benzyl ethyl ether and (*E*)-1-methyl-2-phenylethenyl acetate are reported for the first time in mango fruit. It was worth noting that, benzyl ethyl ether and 1-methyl-2-phenylethenyl acetate were detected in nearly all cvs. In addition, both compounds represented the major constituents particularly in Nubaria cvs, that is, AD, FD, and ZD. For instance, they are responsible for the 5% of benzenoid/aromatic fraction in AD, Table 2.

*2.1.5. Fatty Acid-Derived Compounds.* Fatty acid-derived volatiles were detected in nearly all investigated mango cvs at low levels though mango is very rich in fats suggesting that $\beta$-oxidation reactions, that is, LOX, are rather limited in mango fruits. Highest levels of that class were detected in BNS cv. at 7.5% mostly represented by (2*E*,6*E*)-2,6-nonadienal (P 33), Table 2. It is among the aroma compounds characteristic of mango fruits imparting a cucumber-like odor.[17] The results were consistent with those of other previous reports on mango fruits suggesting that fatty acid-derived VOCs are characteristic aroma compounds for various mango cvs, including the "Glenn" harvested from the Tropical Research and Education Center, the University of Florida. Yet, stereochemistry of nonadienal was also detected to change as the (2*E*,6*Z*)-2,6-nonadienal isomer in the mango fruit of the cv Haden harvested from the USDA ARS Station in Miami, Florida.[41]

*2.1.6. Miscellaneous Compounds.* The miscellaneous groups involve VOCs belonging to heterogenous chemical classes. The key aroma compound to distinguish among them is premium cv AS, that is, the carboxylic acid ester (*Z*)-3-hexenyl-1-acetate (P 10) to amount for ca. 62% of the total volatile blend, followed by Nubaria cv FD with 9.8%, Table 2. (*Z*)-3-Hexenyl-1-acetate has a unique green, sweet, and fruity odor and is considered among the principal odorant volatiles in mango fruits.[42] Despite the similar climate for Sharqia governorate and Ismailia governorate, the Ismailia mango cvs showed the absence of such fragrant compounds. (*Z*)-3-hexenyl acetate was previously reported as a major aroma constituent in mango cvs "Glenn" and "Saigon" of the tropical origin,[17] and extends to be identified in other regions of the world such as Egypt.

*2.1.7. Others.* In addition to the major previously discussed classes, other VOCs were detected but with lower contributions. Examples include oxygenated sesquiterpene represented by two compounds only, including nerolidol (P 64). Oxygenated sesquiterpene was detected with the maximum presence in HSS cv at 1.2% mainly as nerolidol. This class was observed to be completely absent in "awees" cv in all harvesting regions.

Moreover, aliphatic hydrocarbons were present at minor levels in cvs such as FD, BNS, HKTS, HSS, SS, ZS1, FS, and AI ranging from 0.0 to 0.53%, and were found to be absent in most others. *n*-Heptadecane, 9-tetradecenal, in addition,


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**Table 2. Chemical Classes of the Identified Volatile Metabolites Showing Their Abundances (%) ±SD Associated with Various Egyptian Mango Cultivars**[a]

| | | | investigated mango specimens | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | desert area (Nubaria) | | | Sharqia governorate | | | | | | | | Ismailia governorate | | |
| volatiles class | peak no. | metabolite name | AD | FD | ZD | BNS | HKTS | HSS | SS | ZS1 | ZS2 | AS | FS | AI | LI | ZI |
| aliphatic hydrocarbon | P49 | unknown hydrocarbon | | | 0.0 ± 0.01 | 0.17 ± 0.12 | 0.01 ± 0.01 | 0.05 ± 0.03 | 0.05 ± 0.03 | 0.02 ± 0.02 | | | | | | |
| | P82 | 9-tetradecenal | | | | 0.21 ± 0.24 | 0.02 ± 0.02 | 0.06 ± 0.03 | | | | | | | | |
| | P85 | heptadecane | | 0.03 ± 0.04 | | 0.15 ± 0.13 | 0.0 ± 0.01 | 0.09 ± 0.01 | | | | | 0.03 ± 0.02 | 0.02 ± 0.02 | | |
| | | **Total** | **0** | **0.03** | **0** | **0.53** | **0.03** | **0.2** | **0.05** | **0.02** | **0** | **0** | **0.03** | **0.02** | **0** | **0** |
| benzenoid/ aromatic | P3 | *α*-methylphenethyl alcohol | | | | 0.19 ± 0.13 | 0.03 ± 0.01 | | 0.06 ± 0.06 | | | | | | | |
| | P19 | benzyl ethyl ether | 2.4 ± 2.15 | 3.55 ± 0.69 | 1.06 ± 1.55 | 0.7 ± 0.07 | 0.74 ± 0.15 | 5.33 ± 2.45 | 0.7 ± 0.05 | 0.37 ± 0.28 | 1.83 ± 2.54 | 0.12 ± 0.12 | 2.32 ± 1.65 | 0.01 ± 0.02 | | |
| | P40 | 5-methoxyindane | | | | 0.09 ± 0.08 | | 1.23 ± 1.23 | | | | | | | | |
| | P60 | 1-methyl-2-phenylethenyl acetate | 2.62 ± 2.62 | 0.22 ± 0.05 | 0.01 ± 0.02 | 0.03 ± 0.02 | 0.04 ± 0.02 | 0.1 ± 0.06 | 0.03 ± 0.01 | 0.01 ± 0.01 | 0.08 ± 0.07 | | 0.09 ± 0.07 | 0.01 ± 0.01 | 0.35 ± 0.34 | |
| | | **Total** | **5.02** | **3.77** | **1.07** | **1.01** | **0.81** | **6.66** | **0.79** | **0.38** | **1.91** | **0.12** | **2.41** | **0.02** | **0.35** | **0** |
| diterpene hydrocarbon | P87 | *α*-springene | | | | | | | | | | | 0.02 ± 0.01 | | | |
| | P88 | isophyllocladene | | | | | | | | | | | 0.12 ± 0.09 | | | |
| | | **Total** | **0** | **0** | **0** | **0** | **0** | **0** | **0** | **0** | **0** | **0** | 0.14 | **0** | **0** | **0** |
| fatty acid derived | P23 | isoamyl isobutanoate | | 0.02 ± 0.03 | | 0.05 ± 0.07 | | 0.11 ± 0.11 | 0.01 ± 0.01 | | | 0.51 ± 0.25 | 0.01 ± 0.01 | | | |
| | P32 | 2,6-dimethyl-1,3(*E*),5(*E*),7-octatetraene | | | 0 ± 0.01 | 0.28 ± 0.08 | 0.14 ± 0.01 | 0.29 ± 0.29 | 0.1 ± 0.01 | 0.01 ± 0.01 | | | | | | |
| | P33 | (2*E*,6*E*)-2,6-nonadienal | | | | 6.69 ± 7.35 | 0.23 ± 0.23 | 0.07 ± 0.01 | 0.36 ± 0.34 | | | | | | | |
| | P35 | 3-hexenyl butyrate | | 0.27 ± 0.16 | 0 ± 0.01 | 0.06 ± 0.04 | 0.02 ± 0.01 | 0.03 ± 0.03 | | 0.03 ± 0.02 | | 0.03 ± 0.03 | 0.02 ± 0.02 | | | |
| | P42 | hex-3-enyl (*E*)-2-methylbut-2-enoate | | | | 0.26 ± 0.19 | 0.01 ± 0.01 | 0.03 ± 0.01 | | | | | 0.08 ± 0.06 | | | |
| | P51 | capric acid, ethyl ester | | | | | | | | | | | | 0.02 ± 0.03 | | |
| | P78 | ethyl laureate | | 0.49 ± 0.05 | | 0.01 ± 0.02 | 0.01 ± 0.01 | 0.03 ± 0.03 | | | | 0.16 ± 0.03 | | 0.13 ± 0.23 | | |
| | P79 | methyl dodecanoate | | 0.08 ± 0.05 | | | | | | | | | | 0.05 ± 0.08 | | |
| | P83 | myristylaldehyde | | | | 0.03 ± 0.02 | 0.15 ± 0.11 | 0.02 ± 0.01 | 0.05 ± 0.05 | | | | 0 ± 0.01 | | | |
| | P84 | myristic acid, ethyl ester | | 0.55 ± 0.03 | | | 0.02 ± 0.01 | | | | 0.03 ± 0.05 | 0.11 ± 0.11 | | 0.16 ± 0.29 | | |
| | P86 | methyl palmitate | | | | | | | | | | | | | | |
| | P90 | unknown acyl aldehyde | | | | 0.12 ± 0.14 | 0.01 ± 0.01 | 1.66 ± 1.66 | | | | | 0.08 ± 0.06 | 0 ± 0.01 | | |



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**Table 2. continued**

| | | | investigated mango specimens | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | desert area (Nubaria) | | | Sharqia governorate | | | | | | | | Ismailia governorate | | |
| volatiles class | peak no. | metabolite name | AD | FD | ZD | BNS | HKTS | HSS | SS | ZS1 | ZS2 | AS | FS | AI | LI | ZI |
| | P91 | ethyl palmitate | | 0.71 ± 0.05 | | | 0.02 ± 0.02 | 0.01 ± 0.01 | 0.02 ± 0.02 | | 0.01 ± 0.02 | 0.12 ± 0.12 | 0 ± 0.01 | 0.03 ± 0.05 | | |
| | P92 | unknown fatty acid ester | | | | | | | | | | | | 0 ± 0.01 | | |
| | | **Total** | **0** | **2.12** | **0** | **7.5** | **0.61** | **2.25** | **0.54** | **0.04** | **0.04** | **0.93** | **0.19** | **0.39** | **0** | **0** |
| monoterpene hydrocarbon | P2 | 3-thujene | | | 0.02 ± 0.03 | 1.05 ± 0.83 | 0.18 ± 0.05 | 0.06 ± 0.02 | 0.32 ± 0.23 | 0.14 ± 0.17 | 0.05 ± 0.07 | | 0.01 ± 0.01 | | | |
| | P6 | $\alpha$-pinene | 0.32 ± 0.24 | 8.5 ± 1.3 | 1.76 ± 2.82 | 1.05 ± 0.41 | 0.95 ± 0.25 | 0.6 ± 0.45 | 1.45 ± 0.32 | 0.4 ± 0.38 | 0.2 ± 0.18 | 0.14 ± 0.14 | 17.16 ± 5.46 | 0.93 ± 1.41 | 13.14 ± 12.85 | 0.72 ± 0.3 |
| | P7 | $\beta$-myrcene | 0.19 ± 0.1 | 7.78 ± 1.33 | 0.08 ± 0.13 | 0.93 ± 0.21 | 0.82 ± 0.17 | 0.11 ± 0.05 | 1.3 ± 0.31 | 0.33 ± 0.29 | 0.17 ± 0.15 | 0.09 ± 0.09 | 13.93 ± 2.81 | 0.01 ± 0.01 | 0.16 ± 0.16 | |
| | P9 | $\alpha$-phellandrene[b] | 4.7 ± 3.4 | 3.2 ± 2.3 | 0.89 ± 0.82 | 1.44 ± 0.29 | 1.69 ± 0.3 | 0.03 ± 0.03 | 2.14 ± 0.54 | 1.63 ± 1.39 | 2.05 ± 1.13 | 0.7 ± 0.11 | 3.11 ± 2.21 | 0.66 ± 0.9 | 26.5 ± 7.83 | 0.21 ± 0.14 |
| | P11 | $\beta$-thujene | 11.58 ± 0.49 | 11.18 ± 7.97 | 2.27 ± 0.87 | 3.23 ± 0.95 | 3.36 ± 0.75 | 0.02 ± 0.02 | 4.65 ± 1.3 | 3.51 ± 3.49 | 3.64 ± 2.73 | 1.44 ± 0.05 | 12.74 ± 9.03 | 0.91 ± 1.24 | 45.81 ± 12.68 | 0.44 ± 0.25 |
| | P12 | $\beta$-pinene | | | 0.37 ± 0.41 | 0.73 ± 0.22 | 0.62 ± 0.22 | 1.58 ± 1.59 | 1.7 ± 0.17 | 0.47 ± 0.42 | 0.69 ± 0.68 | 0.06 ± 0.06 | | 0.21 ± 0.26 | 0.02 ± 0.02 | |
| | P13 | isoterpinolene | | | 0.06 ± 0.1 | 0.24 ± 0.19 | 0.15 ± 0.02 | 3.11 ± 2.96 | 0.39 ± 0.06 | 0.15 ± 0.08 | 0.12 ± 0.11 | | | 0 ± 0.01 | | |
| | P14 | $\alpha$-terpinene | | | 0.63 ± 0.63 | 0.7 ± 0.18 | 0.67 ± 0.17 | 0.03 ± 0.02 | 1.7 ± 0.17 | 0.83 ± 0.6 | 0.71 ± 0.21 | 0.29 ± 0.15 | | 0.21 ± 0.26 | | |
| | P15 | 2-carene | | | 0.38 ± 0.39 | 0.45 ± 0.16 | 0.38 ± 0.11 | | 1.13 ± 0.15 | 0.47 ± 0.34 | 0.62 ± 0.42 | 0.13 ± 0.13 | | 0.02 ± 0.03 | | |
| | P16 | *p*-Cymene | | | 0.06 ± 0.11 | 1.55 ± 0.59 | 1.58 ± 0.22 | 0.23 ± 0.24 | 0.54 ± 0.07 | 0.23 ± 0.2 | 0.08 ± 0.07 | 0.21 ± 0.06 | | | | |
| | P17 | limonene | 7.3 ± 2.59 | 0.3 ± 0.17 | 0.41 ± 0.36 | 1.45 ± 0.41 | 1.09 ± 0.27 | 0.56 ± 0.28 | 1.59 ± 0.01 | 0.75 ± 0.58 | 0.69 ± 0.17 | 0.64 ± 0.13 | 0.22 ± 0.15 | 0.02 ± 0.03 | | |
| | P18 | $\beta$-phellandrene | 1.68 ± 1.68 | 5.96 ± 0.91 | 0.51 ± 0.48 | 1.42 ± 0.34 | 1.9 ± 0.41 | 12.45 ± 6.27 | 1.18 ± 0.14 | 0.97 ± 0.63 | 1.13 ± 0.53 | 0.42 ± 0.07 | 9.76 ± 3.42 | 0.03 ± 0.05 | | |
| | P20 | unknown monoterpene | | 0.56 ± 0.16 | 0.02 ± 0.03 | 0.65 ± 0.42 | 0.82 ± 0.47 | 1.59 ± 0.82 | 0.01 ± 0.01 | 0.03 ± 0.03 | 0.02 ± 0.03 | | 0.63 ± 0.44 | | | |
| | P21 | $\beta$-*Z*-ocimene | 7.29 ± 6.46 | 7.77 ± 1.28 | 1.59 ± 1.91 | 0.25 ± 0.06 | 0.34 ± 0.02 | 19.38 ± 1.46 | 1.2 ± 0.17 | 1.77 ± 1.47 | 1.38 ± 2.21 | | 9.87 ± 3.34 | | | |
| | P25 | $\beta$-terpinene | 0.38 ± 0.38 | 7.47 ± 5.33 | 0.03 ± 0.05 | 0.3 ± 0.11 | 0.17 ± 0.03 | 19.42 ± 0.62 | 0.38 ± 0.07 | 0.16 ± 0.11 | 0.14 ± 0.13 | | 6.46 ± 4.58 | 39.2 ± 36.03 | | |
| | P26 | terpinolene | 20.3 ± 7.39 | 0.07 ± 0.06 | 67.01 ± 8.14 | 58.21 ± 13.35 | 72.64 ± 0.98 | 0.01 ± 0.01 | 63.33 ± 2.91 | 53.99 ± 6.8 | 73.72 ± 6.48 | 16.31 ± 2.28 | 0.03 ± 0.03 | 54.82 ± 31.82 | | 98.44 ± 0.69 |
| | P27 | *p*-cymene | 0.98 ± 0.59 | | 0.03 ± 0.06 | 1.88 ± 0.46 | 3.02 ± 0.66 | | 1.16 ± 0.28 | 0.34 ± 0.24 | 0.3 ± 0.26 | 0.14 ± 0.14 | | 0.06 ± 0.1 | | |
| | P28 | *o*-cymene | | | 0 ± 0.01 | 0.3 ± 0.06 | 0.19 ± 0.04 | 0.58 ± 0.53 | 0.16 ± 0.03 | 0.01 ± 0.01 | | | | | | |
| | P29 | allo-ocimene | | | | 0.09 ± 0.1 | 0.2 ± 0.29 | 2.35 ± 2.13 | | | | | 0.01 ± 0.01 | | | |
| | P30 | 2,6-dimethyl-1,3,5,7-octatetraene | 0.51 ± 0.51 | | 0.01 ± 0.02 | 0.74 ± 0.06 | 0.41 ± 0.06 | 6.35 ± 5.35 | 0.42 ± 0.08 | 0.03 ± 0.02 | 0.1 ± 0.11 | 0.21 ± 0.13 | 0.16 ± 0.12 | | | |
| | P31 | dihydrodicyclopentadiene | 0.1 ± 0.1 | | | 0.04 ± 0.01 | 0.02 ± 0.02 | 0.03 ± 0.03 | | 0 ± 0.01 | | | | | | |



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**Table 2. continued**

| | | | investigated mango specimens | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | desert area (Nubaria) | | | Sharqia governorate | | | | | | | | Ismailia governorate | | |
| volatiles class | peak no. | metabolite name | AD | FD | ZD | BNS | HKTS | HSS | SS | ZS1 | ZS2 | AS | FS | AI | LI | ZI |
| | | **Total** | **55.33** | **52.79** | **76.13** | **76.7** | **91.2** | **68.49** | **84.75** | **66.21** | **85.81** | **20.78** | **74.09** | **97.08** | **85.63** | **99.81** |
| oxygenated monoterpene | P22 | cineol[b] | 1.45 ± 1.17 | 0.54 ± 0.32 | 0.02 ± 0.03 | 1.14 ± 0.74 | 0.39 ± 0.16 | 0.75 ± 0.3 | 0.06 ± 0.01 | 0.18 ± 0.23 | 0.04 ± 0.04 | 0.14 ± 0.14 | 0.28 ± 0.2 | | | |
| | P24 | hotrienol | | 0.75 ± 0.97 | | 0.12 ± 0.09 | 0.01 ± 0.01 | 0.01 ± 0.01 | 0 ± 0 | | | 11.14 ± 3.14 | | | | |
| | P34 | *p*-menthan-1-ol | | | | 0.08 ± 0.05 | 0.03 ± 0.03 | 0.02 ± 0.01 | 0.01 ± 0.01 | | | | | | | |
| | P36 | *p*-cymene-8-ol | | | | 0.51 ± 0.21 | 0.89 ± 0.86 | 0.01 ± 0.01 | 0.16 ± 0.05 | | | | | | | |
| | P37 | safranal | | | | 0.01 ± 0.02 | 0.04 ± 0.07 | | 0.22 ± 0.07 | | | | | | | |
| | P39 | methyl acetate | | | | 0.05 ± 0.03 | 0 ± 0.01 | 0.02 ± 0.02 | | | | | | | | |
| | P41 | *p*-Cymen-3-ol | | | | 0.02 ± 0.02 | | 0.01 ± 0.01 | 1.02 ± 1.02 | | | | | | | |
| | P43 | unknown monoterpene | | | 0 ± 0.01 | 0 ± 0.01 | 0.02 ± 0.03 | 0.05 ± 0.04 | | | | | | | | |
| | P59 | geranylacetone | 29.52 ± 29.52 | 0.24 ± 0.34 | 0.01 ± 0.01 | 1.82 ± 1.66 | 0.06 ± 0.04 | 0.91 ± 0.82 | 0.02 ± 0.02 | 0.01 ± 0.01 | 0.09 ± 0.15 | | 0.03 ± 0.03 | | 0.08 ± 0.08 | |
| | P77 | geranyl isobutyrate | | | | 0.02 ± 0.03 | | 0.13 ± 0.05 | | | | | | | | |
| | | **Total** | **30.97** | **1.53** | **0.03** | **3.77** | **1.44** | **1.91** | **1.49** | **0.19** | **0.13** | **11.28** | **0.31** | **0** | **0.08** | **0** |
| oxygenated sesquiterpene | P64 | nerolidol[b] | | 0.54 ± 0.76 | 0.01 ± 0.02 | 0.38 ± 0.3 | 0.05 ± 0.02 | 1.17 ± 1.03 | 0.05 ± 0.02 | 0.02 ± 0.02 | 0.02 ± 0.03 | | 0.03 ± 0.03 | | 0.87 ± 0.85 | |
| | P89 | unknown terpene | | 0.14 ± 0.11 | | | | 0.02 ± 0.02 | | | | | 0.48 ± 0.35 | | | |
| | | **Total** | **0** | **0.68** | **0.01** | **0.38** | **0.05** | **1.19** | **0.05** | **0.02** | **0.02** | **0** | **0.51** | **0** | **0.87** | **0** |
| sesquiterpene hydrocarbon | P45 | *α*-ylangene | | 0.34 ± 0.06 | 4.52 ± 1.34 | 0.42 ± 0.23 | 0.03 ± 0.02 | 0.07 ± 0.07 | 1.83 ± 0.05 | 6.25 ± 0.65 | 1.78 ± 0.68 | 0.21 ± 0.05 | 0.45 ± 0.32 | 0.13 ± 0.23 | | |
| | P46 | *α*-cubebene | | 0.16 ± 0.12 | 3.46 ± 0.71 | 0.25 ± 0.15 | 0.02 ± 0.01 | 0.05 ± 0.05 | 1.43 ± 0.08 | 5.52 ± 0.59 | 1.45 ± 0.52 | 0.17 ± 0.07 | 0.38 ± 0.27 | 0.11 ± 0.19 | | |
| | P47 | copaene | | 0.34 ± 0.06 | 4.52 ± 1.34 | 0.42 ± 0.23 | 0.03 ± 0.02 | 0.07 ± 0.07 | 1.83 ± 0.05 | 6.25 ± 0.65 | 1.78 ± 0.68 | 0.21 ± 0.05 | 0.45 ± 0.32 | 0.13 ± 0.23 | | |
| | P48 | *β*-ylangene | | | 0.64 ± 0.22 | | | 0.06 ± 0.06 | 0.26 ± 0.03 | 1.11 ± 0.21 | 0.15 ± 0.15 | | 0.06 ± 0.04 | 0.02 ± 0.04 | | |
| | P50 | *α*-copaene | | 0.16 ± 0.12 | 3.46 ± 0.71 | 0.46 ± 0.37 | 0.02 ± 0.01 | 0.06 ± 0.06 | 1.43 ± 0.08 | 5.52 ± 0.59 | 1.45 ± 0.52 | 0.17 ± 0.07 | 0.38 ± 0.27 | 0.11 ± 0.19 | | |
| | P53 | *β*-elemene | | | 1.29 ± 2.23 | | | 0.4 ± 0.4 | | 5.57 ± 0.54 | | | | 0.04 ± 0.07 | 0.3 ± 0.29 | |
| | P54 | *β*-famesene | 0.94 ± 0.94 | 8.17 ± 1.75 | 0.99 ± 0.33 | 0.94 ± 0.6 | 1.3 ± 0.66 | 2.3 ± 1.26 | 0.87 ± 0.28 | 0.39 ± 0.06 | 1.18 ± 0.81 | 0.95 ± 0.36 | 5.34 ± 1.66 | 0.63 ± 0.09 | 1.31 ± 1.28 | |
| | P55 | *α*-farnesene | | 1.3 ± 0.95 | 0.42 ± 0.23 | 0.2 ± 0.2 | 0.33 ± 0.18 | 0.24 ± 0.07 | 0.19 ± 0.06 | 0.12 ± 0.03 | 0.25 ± 0.31 | 0.21 ± 0.11 | 1.37 ± 0.28 | 0.07 ± 0.11 | 0.09 ± 0.09 | |
| | P56 | *β*-humulene | 5.63 ± 5.63 | 3.42 ± 2.47 | 0.8 ± 0.25 | 0.66 ± 0.4 | 0.88 ± 0.44 | 0.47 ± 0.3 | 0.6 ± 0.19 | 0.27 ± 0.06 | 0.81 ± 0.55 | 0.51 ± 0.12 | 3.82 ± 1.03 | 0.17 ± 0.28 | | |



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**Table 2. continued**

| volatiles class | peak no. | metabolite name | investigated mango specimens: desert area (Nubaria) AD | FD | ZD | Sharqia governorate BNS | HKTS | HSS | SS | ZS1 | ZS2 | AS | FS | Ismailia governorate AI | LI | ZI |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | P57 | α-guaiene | | | 0.05 ± 0.09 | | | 0.28 ± 0.16 | | | | | | 0 ± 0.01 | 0.18 ± 0.18 | |
| | P58 | α-humulene[b] | 0.99 ± 0.99 | 14.86 ± 2.7 | 1.91 ± 0.84 | 1.44 ± 0.86 | 2.06 ± 0.84 | 2.03 ± 0.17 | 1.83 ± 1.11 | 0.7 ± 0.24 | 2.15 ± 1.8 | 1.81 ± 0.68 | 8.93 ± 3.69 | 0.9 ± 0.89 | 3.32 ± 1.31 | |
| | P61 | muurola-4(14),5-diene | | 0.21 ± 0.15 | 0.02 ± 0.04 | 0.05 ± 0.03 | 0 ± 0 | 1.2 ± 1.05 | | 0.02 ± 0.02 | 0.03 ± 0.05 | | 0.13 ± 0.1 | 0.02 ± 0.03 | 0.56 ± 0.55 | |
| | P62 | aromandendrene | | 0.29 ± 0.22 | 0.03 ± 0.04 | | | 0.34 ± 0.28 | | 0.02 ± 0.02 | 0.05 ± 0.09 | | 0.19 ± 0.15 | | 0.18 ± 0.18 | |
| | P63 | cadine-3,9-diene | | | 0.01 ± 0.02 | 0.05 ± 0.03 | | 0.61 ± 0.47 | | 0.04 ± 0.04 | 0.02 ± 0.03 | | | 0.02 ± 0.03 | 0.56 ± 0.55 | |
| | P65 | α-bergamotene | | | | 0.13 ± 0.11 | 0.01 ± 0 | 0.04 ± 0.03 | | | | | | | 0.1 ± 0.1 | |
| | P66 | D-germacrene | | | 0.04 ± 0.06 | 0.01 ± 0.01 | | 1.04 ± 0.98 | | 0.01 ± 0.01 | | | | 0.02 ± 0.03 | 3.56 ± 1.16 | 0.15 ± 0.03 |
| | P67 | β-selinene | | | 0.02 ± 0.04 | 0.08 ± 0.01 | 0.29 ± 0.45 | 1.45 ± 0.46 | 0.08 ± 0.04 | 0.03 ± 0.02 | 0.04 ± 0.05 | | 0.04 ± 0.03 | 0.01 ± 0.01 | 0.22 ± 0.22 | 0.04 ± 0.0 |
| | P68 | γ-patchoulene | | | 0.01 ± 0.01 | | | 0.56 ± 0.17 | | 0.01 ± 0.01 | | | | | 0.12 ± 0.12 | |
| | P69 | 8,8-dimethyl-9-methylene-1,5-cycloundecadiene | | | 0.01 ± 0.03 | 0.13 ± 0.04 | 0.03 ± 0.01 | 1.98 ± 1.36 | 0.06 ± 0 | 0.02 ± 0.02 | 0.05 ± 0.05 | | 0.02 ± 0.02 | | 0.94 ± 0.92 | |
| | P70 | α-muurolene | | | 0.02 ± 0.03 | 0.02 ± 0.02 | | 3.24 ± 3.08 | | 0.02 ± 0.02 | 0.2 ± 0.33 | | | 0.03 ± 0.05 | 0.03 ± 0.03 | |
| | P71 | unknown sesquiterpene | | | 0.01 ± 0.01 | 0.01 ± 0.01 | 0.02 ± 0.02 | 0.36 ± 0.07 | | 0.01 ± 0.01 | | | | | | |
| | P72 | α-bulnesen | | | 0.01 ± 0.01 | | | 0.56 ± 0.17 | | 0.01 ± 0.01 | | | | | | |
| | P73 | δ-guaiene | | | 0.01 ± 0.02 | | | 0.66 ± 0.28 | | 0.02 ± 0.02 | | | | | | |
| | P74 | cadina-1(10),4-diene | | | 0.27 ± 0.11 | 0.11 ± 0.06 | 0.03 ± 0.02 | 0.25 ± 0.15 | 0.24 ± 0.19 | 0.35 ± 0.08 | 0.22 ± 0.27 | | 0.04 ± 0.03 | 0.03 ± 0.05 | 1.51 ± 1.47 | |
| | P75 | δ-cadinene | | | 0.02 ± 0.03 | | | 0.03 ± 0.03 | | 0.03 ± 0.03 | 0.02 ± 0.03 | | | | | |
| | P76 | 4-isopropyl-1,6-dimethyl-1,2,3,4,4a,7-hexahydronaphthalene | | | 0.01 ± 0.02 | | | 0.03 ± 0.02 | | 0.02 ± 0.02 | | | | | | |
| | P81 | unknown sesquiterpene | | | 0.03 ± 0.05 | | 0.01 ± 0.01 | 0.01 ± 0.01 | | 0.04 ± 0.04 | | 1.13 ± 0.36 | | 0.01 ± 0.02 | | |
| | | **Total** | **7.56** | **29.25** | **22.58** | **5.38** | **5.06** | **18.39** | **8.82** | **32.35** | **11.63** | **5.38** | **21.6** | **2.45** | **12.98** | **0.19** |
| miscellaneous | P1 | isobutyl acetate | | | | | | | 0.24 ± 0.16 | | | | | | | |
| | P5 | benzyl ethyl oxide | 0.39 ± 0.39 | | 0.02 ± 0.03 | 1.07 ± 0.75 | 0.2 ± 0.04 | 0.02 ± 0.02 | | 0.13 ± 0.16 | 0.06 ± 0.08 | | 0.03 ± 0.03 | | | |
| | P8 | isobutyl isobutyrate | | | | 0.01 ± 0.02 | | | | | | | | | 0.02 ± 0.02 | |
| | P10 | Z-3-hexenyl-1-acetate[b] | 0.74 ± 0.74 | 9.73 ± 2.88 | 0.01 ± 0.01 | 0.24 ± 0.11 | 0.04 ± 0.04 | | 0.04 ± 0 | 0.03 ± 0.03 | | 61.21 ± 1.66 | 0.5 ± 0.36 | | | |



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**Table 2. continued**

| volatiles class | peak no. | metabolite name | investigated mango specimens | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | desert area (Nubaria) | | | Sharqia governorate | | | | | | | Ismailia governorate | | |
| | | | AD | FD | ZD | BNS | HKTS | HSS | SS | ZS1 | ZS2 | AS | FS | AI | LI | ZI |
| | P52 | 2,2,5-trimethylhexane-3,4-dione | | 0.08 ± 0.07 | 0.05 ± 0.08 | 0.68 ± 0.46 | 0.04 ± 0.03 | 0.23 ± 0.18 | 0.13 ± 0.02 | 0.26 ± 0.1 | 0.07 ± 0.12 | 0.29 ± 0.29 | 0.02 ± 0.02 | 0 ± 0.01 | | |
| | | **Total** | **1.13** | **9.81** | **0.08** | 2 | **0.28** | **0.25** | **0.17** | **0.42** | **0.13** | **61.5** | **0.55** | **0** | **0.02** | **0** |
| unknown | P4 | unknown | 0.3 ± 0.14 | | 0.04 ± 0.07 | 2.56 ± 1.72 | 0.49 ± 0.13 | 0.4 ± 0.33 | 2.39 ± 1.14 | 0.34 ± 0.46 | 0.29 ± 0.14 | | 0.09 ± 0.08 | | | |
| | P38 | unknown | | | | 0.15 ± 0.12 | 0.01 ± 0.02 | 0.14 ± 0.11 | 1.02 ± 0.02 | | 0.02 ± 0.04 | | 0 ± 0.01 | | | |
| | P44 | unknown | | | | | | | | | | | | | 0.08 ± 0.08 | |
| | P80 | unknown | 1.07 ± 1.07 | | 0.03 ± 0.06 | | | 0.1 ± 0.1 | | 0.02 ± 0.02 | | | 0.02 ± 0.02 | 0.01 ± 0.02 | | |
| | | **Total** | **1.37** | **0** | **0.07** | **2.71** | **0.5** | **0.64** | **3.41** | **0.36** | **0.31** | **0** | **0.11** | **0.01** | **0.08** | **0** |

[a]They were investigated by HS-SPME combined with GC/MS analysis ($n$ = 3). The corresponding sample codes are listed in Table 1. [b]Identified compound in relation to a standard reference in addition to the Kovat index.

another unknown compound were detected in accordance with those reported in Chinese mangoes.[43]

Furthermore, the contribution of diterpene hydrocarbons as volatile determinants in mango is rather limited owing to their low abundance and volatility.[9,44] Among the examined cvs, only FS showed trace levels at 0.14% represented by $\alpha$-springene (0.02%) and isophyllocladene (0.12%).

**2.2. MVAs of Mango Fruit cvs' Aroma Composition.** To aid in cvs' classification and identify potential markers in an untargeted manner, MVAs were further employed including principal component analysis (PCA), hierarchical cluster analysis (HCA), and orthogonal partial least square (OPLS) analysis, see Figures 3 and 4, and S2 for the extracted GC/MS dataset.

*2.2.1. Unsupervised PCA and HCA Data Analyses of 14 Mango cvs.* PCA provides a more holistic approach to analyze the GC−MS dataset and explores relative variability within the different cvs of different origins. From the 14 cvs, peak mass signals from the GC−MS data set were utilized for PCA modeling, with each cv represented by three biological replicates. Triplicate measurements of each sample were found to be reproducible, as the scores of replicate measurements were more or less superimposable. The model prescribed by principal component 1 (PC1) and PC2 explained 47% of the total variance, Figure 3A. On the left side of the score plot, FD, FS, LI, and HSS specimens were positioned (negative PC1 values), whereas other cvs segregated on the right side with positive PC1 score values. The close clustering of FD and FS suggested that the cv effect overcomes regional differences with regard to the aroma composition at least in the case of "founs" cv. Samples of all other cvs were scattered along the right side of PC1, with no separation along PC2.

Moreover, examination of the loadings plot (Figure 3B) suggested that MS signals of monoterpene hydrocarbons mostly accounted for cv segregation. For instance, $\alpha$-pinene, $\beta$-$Z$-ocimene, and $\beta$-terpinene were abundant in cvs with negative score values along PC1, especially for FD, FS, LI, and HSS. The distant segregation of LI appearing as an outlier in the lower negative quadrant of PCA was due to its abundance in ($Z$)-3-hexenyl-1-acetate. The close clustering of "zebdia" cvs from different origins, that is, ZS1, ZS2, ZD, and ZI, was attributed to their abundance in monoterpene hydrocarbon terpinolene, as a key marker of that cv especially in the case of ZI in which it amounted for 98.4% of the total monoterpene hydrocarbons.

Furthermore, HCA allowed for interpretation of the results in an intuitive graphical display, Figure 3C, allowing for determination of VOC heterogeneity among investigated cvs. HCA showed two clear clusters referred to as groups 1a and 1b, respectively. Inspection of group 1a revealed that AS, LI, AI, HSS, AD, FS, and FD were clustered together, whereas all four "zebdia" fruits, that is, ZI, ZS1, ZS2, and ZD of different origins clustered together in cluster 1b along with fruits belonging to BNS, SS, and AI. The close clustering of those cvs indicated their more or less similar volatile content.

*2.2.2. Unsupervised PCA of "Zebdia" Fruits from Different Origins.* The GC−MS aroma dataset was examined for the regional discrimination of "zebdia" fruits modeled separately being represented by three samples of different geographical origins, Table 1. The PCA score plot showed only clear separation of fruits collected from the Ismailia region (LI, ZI, and AI) on the right with positive PC1 score values (Figure


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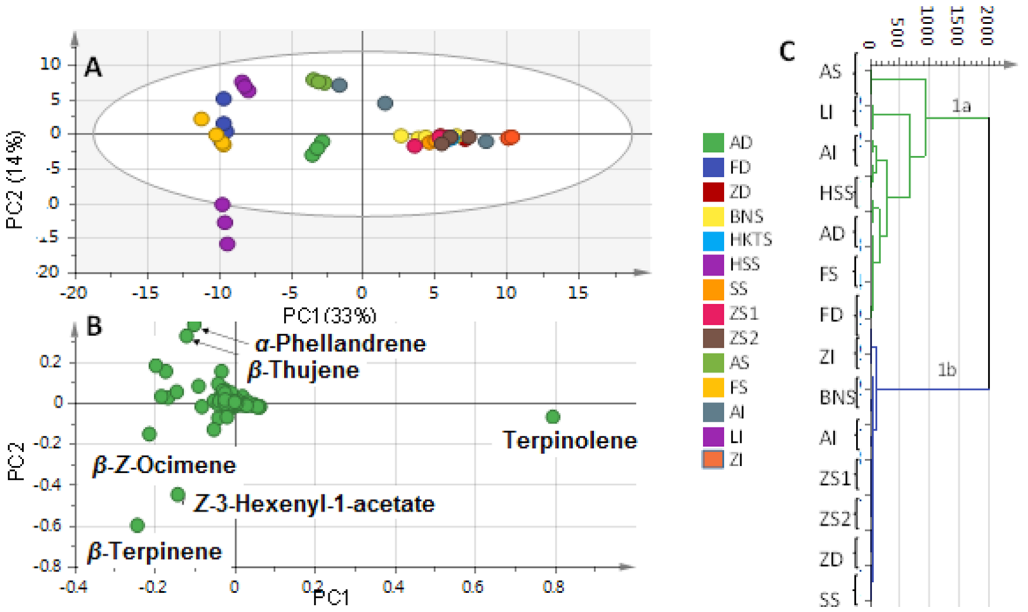


**Figure 3.** GC−MS ($m/z$ 100−1000) principal component analyses of the different mango cultivars ($n$ = 14). The volatilome clusters are located at the distinct positions described by two vectors of PC1 (33%) and PC2 (14%). (A) Score plot of PC1 versus PC2 scores. (B) Loading plot for PC1 and PC2 with contributing mass peaks and their assignments. (C) HCA dendrogram analysis of mango cultivars based on group average cluster analysis using GC−MS, $n$ = 3. Sample codes follow that listed in Table 1.

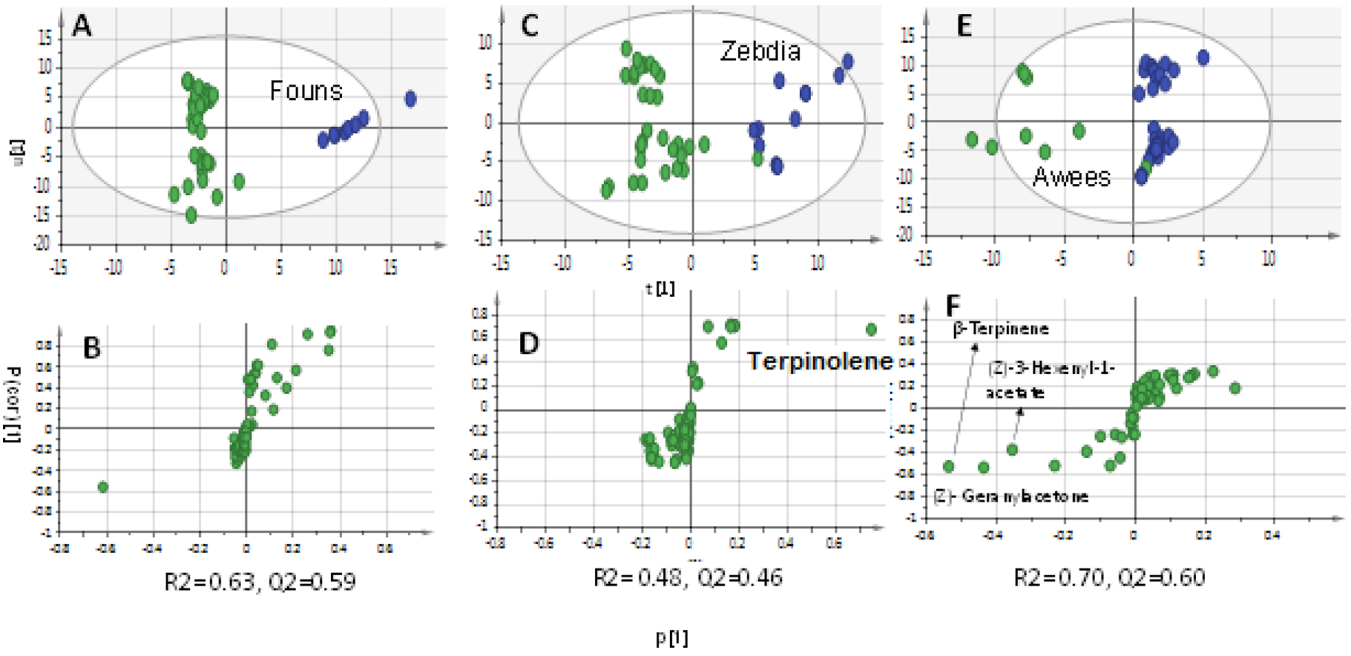


**Figure 4.** OPLS-DA score plot (A) and loading S-plots (B) derived from modeling Founs mango fruits, that is, FS and FD against other cultivars in a separate group. OPLS-DA score plot (C) and loading S-plots (D) derived from modeling Zebdia fruits, that is, ZS1, ZS2, ZD, and ZI, against other fruit cultivars' OPLS-DA score plot (E) and loading S-plots (F) derived from modeling Awees fruits, that is, AS, AD, and AI, against fruit cultivars in a separate group. The S-plot shows the covariance $p[1]$ against the correlation $p(\mathrm{cor})[1]$ of the variables of the discriminating component of the OPLS-DA model. Sample codes follow that listed in Table 1.

S2A). In contrast, other origins were clustered on the opposite side with some overlap from Sharqia and Nubaria regions (negative score values) along PC1 (Figure S2A). In addition, examination of the loading plot revealed the higher abundance of terpinolene in Ismailia fruits compared to other fruit origins (Figure S2B). Ismailia is considered the best location for mango-growing trees in Egypt and whether that is reflected as well on unique flavor of its mango has yet to be confirmed by examining more cvs.

*2.2.3. Supervised OPLS-DA of Mango cvs' Aroma Profile.* Supervised OPLS-discriminant analysis (DA) was further applied to identify markers for fruit cvs that were collected from several origins in addition to their recognition as premium cvs in the Egyptian market liable to adulteration. Supervised methods represent another attempt of classification


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enhancing the discrimination between specimens via minimizing variance.[45] OPLS-DA functions to identify volatile markers correlated with a certain cv, while downweighting the other sources of variance.[27] An OPLS model was constructed with "founs" specimens labeled as one class, against other mango cvs present in another class group, Figure 4A. The model showed one orthogonal component with $R^2$ = 0.63 and $Q^2$ = 0.59 suggestive of no overfit models, though the OPLS-DA-derived S-plot revealing no clear marker for that cv (Figure 4B). Other OPLS-DA modeling score plots (Figure 4C,E) revealed that "zebdia" and "awees" were chemically distinct from other cvs, when each was modeled separately. The S-plot derived from the "zebdia" model against all other cvs showed that terpinolene (P26) mediated for its discrimination, Figure 4D. In contrast, S-plot modeling of "awees", a premium Egyptian mango cv, against all other specimens showed the best model validation parameters with $R^2$ = 0.7 and $Q^2$ = 0.6 attributed to its richness in monoterpenoids, that is, $\beta$-terpinene and (*Z*)-geranylacetone terpenoids, in addition to (*Z*)-3-hexenyl-1-acetate (Figure 4F).

These results are consistent with quantification explained in Section 2.1.1 demonstrating that $\beta$-terpinene is found mainly in HSS and AI and absent in all "founs" cvs regardless of its geographic origin and in agreement with findings of Kulkarni et al.[39] In addition, (*Z*)-geranylacetone and hexenyl acetate are chief aroma compounds in different fruits, including mangoes.[13,46] Consequently, a mixture of these volatiles should be considered to imitate the mango flavor in food and beverages such as vodka cocktail.[42] All three OPLS-DA model validations were carried out using the diagnostic metrics $R^2$ (total variance) and $Q^2$ (goodness parameters), which were greater than 0.4, with most models showing a regression line crossing zero, and with negative $Q^2$ and $R^2$ close to 1, signifying the model validation. Moreover, the *p*-values for each model were calculated using CV-ANOVA (ANOVA of the cross-validated residuals), which were all below 0.005 (Figures S3−S5).

**2.3. Changes in Mango Aroma Composition with Probiotics.** *Lactiplantibacillus plantarum* is one of the probiotic bacteria that resides in human gut symbiotically.[47] Considering that mango is commonly incorporated in several fermented dairy products, that is, acidophilus milk, to impart its flavor, it is important to identify whether a change in its aroma composition can occur upon incubation with probiotics. "sokary" (SS) and "zebdia" (ZI) were considered representatives for this study.

*2.3.1. Effect on pH and Bacterial Viable Count.* *L. plantarum* growth and metabolite consumption were monitored through determination of bacterial counts and pH, respectively, along the fermentation process at the beginning (0 h) and specific time intervals after 24 and 48 h. The results are summarized in Table S1 showing that pH and the viable count were inversely changed along the period of the fermentation process. Hence, it was confirmed that the probiotic bacterium *L. plantarum* was able to use the sugar available in mango juice as a carbon source and to multiply. The bacterium appeared to reach the maximum growth after 24 h with an average viable count of $4 \times 10^9$ cfu/mL, which was not changed till 48 h of cultivation inferring that bacterial growth reached its stationary phase. In parallel, consumption of sugars resulted in the production of acids, that is, lactic acid, which contributed to a decrease in pH of the growth medium viz. mango juice. Profiling of the fermented non-volatile chemicals in mango probiotics should now follow using techniques such as liquid chromatography coupled to MS (LC−MS).

*2.3.2. Probiotic Effect on the Mango Volatile Profile.* Volatile constituents of both investigated cvs, that is, SS and ZI, were identified and classified as previously described (Section 2.1) and demonstrated in Figure S6A,B and Table S2 at the same time intervals of monitoring the pH and viable count, that is, 0, 24, and 48 h. The volatile constituents at the beginning of fermentation were less than the non-autoclavable juice, which may be attributed to the heat-induced degradation as monoterpene hydrocarbons. However, it was observed that there were slight differences between VOC classes over the fermentation period for each investigated mango cvs.

Monoterpene followed by sesquiterpene hydrocarbons were the principal components of the mango all the time similar to those previously observed in volatile analysis of mango pieces. Volatile constituents showed a variable behavior with time. For instance, fatty acid-derived components in examined cvs showed a slight increase in SS and ZI, especially after 24 h of incubation, likely attributed to the inherent ability of the probiotic to synthesize short-chain fatty acids (SCFA) or trigger $\beta$-oxidation of fatty acids.[48] However, monoterpene hydrocarbons showed different pattern in both cvs, where they increased in SS, in contrast to a decline in ZI. The absence of a potential change in most of the mango volatiles indicated that none of these constituents were clearly affected by bacterial-mediated metabolism. Therefore, chemometric tools for sample classification and marker identification were not applicable for such a case.

Analysis of volatile metabolites, for instance monoterpene hydrocarbons, revealed that terpinolene was the most abundant form with 64.4% at $T$ = 0 to 65% at $T$ = 48 h of the total monoterpene hydrocarbons in ZI fermentation. However, $\beta$-terpinene was the major form with 36% at the beginning at $T$ = 0 in the case of SS-amended cultures. Yet, limonene and $\beta$-phellandrene with 27% of the aroma blend at $T$ = 48 h were the major forms, which were totally absent at $T$ = 0. Nevertheless, a large observed variance in the volatile level within different cultures replicated suggests that no conclusive evidence can be made regarding that probiotics affect mango flavors, as observed before in our previous study on roselle flower.[25]

## 3. CONCLUSIONS

A comprehensive investigation of the Egyptian mango fruits represented by different cvs and origins is presented based on VOCs analyzed using HS-SPME/GC−MS combined with MVA herein for the first time. Qualitative and quantitative differences were observed among cvs and also, within the same cv, the geographical origins. Differences in the VOC composition appeared to affect the aroma composition mostly evident in the case of $\beta$-terpinene, which was highly abundant in AI with 39% versus trace levels in "awees" cv of Nubaria (AD) and Sharqia (AS). However, AD and AS were rich in geranyl acetone and (*Z*)-3-hexenyl-1-acetate, respectively. Geranyl acetone was reported for first time in mango cvs. Regarding volatiles' chemical class, monoterpene hydrocarbons appeared as the major components in all investigated cvs, except AS, and to likely contribute to mango characteristic flavors among others. The composition of "awees" cvs, that is, AS, AI, and AD proves their premium quality and explains consumers' preference. Moreover, the modeling results


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confirmed that terpinolene is a key marker of "zebdia" Ismailia (ZI) versus (*Z*)-3-hexenyl-1-acetate as a potential marker for AS. Furthermore, the probiotic effect on mango volatiles' profile revealed variable patterns with time, where constituents such as fatty acid-derived compounds were increased, and others were slightly decreased. The study revealed several aroma determinants in Egyptian mango fruits that can be used to explore the variations in flavors. The study results may open new opportunities for further analysis based on other analytical platforms, that is, LC/MS and NMR for exploring the unique phytochemical composition in relation to quality, authentication, and nutraceutical benefits of the Egyptian mangoes and of other diverse cvs for mango consumers worldwide.

## 4. MATERIALS AND METHODS

**4.1. Plant Materials.** Eight Egyptian cvs of the mango (*M. indica* L., Anacardiaceae) were collected from three different geographical origins. These cvs included hendy kalb el toor, baladi Nabila, hendy Sinara, founs, awees, zebdia, sokary, and langara. The fresh ripe fruits (14 samples) were collected from farms of Sharqia (eight samples, seven cvs) and Ismailia (three samples, three cvs) governorates at the east region of the Nile Delta, in addition to Nubaria (three samples, three cvs) at the west region of the Nile Delta at the desert area, Figure S7. Specimens were coded according to the cv type and geographical origin, as shown in Table 1. In addition, the stage of maturity was confirmed by its external firmness and color, which differ according to each cv. The selected fruits were directly peeled and sliced into small pieces prior to being stored at −20 °C till analysis.

**4.2. Chemicals and Fibers.** Chemicals and standards were purchased from Sigma-Aldrich (St. Louis, MO, USA). SPME StableFlex fibers coated with divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS 50/30 μm) or polydimethylsiloxane (PDMS) were purchased from Supelco (Oakville, ON, Canada) and conditioned at 250 °C for 5 min following supplier's recommendations.

**4.3. Headspace Volatile Analysis of Mango Puree.** Volatiles of mango pastes were collected using HS-SPME and analyzed using GC−MS. Three to four biological replicates from each mango cv of fruits collected from different trees were included to assess for biological variance.

The HS-SPME volatile analysis was conducted following our previous reports[25,49] with slight modifications. Three grams of mango slices were introduced into a (20 mL) screw-capped vials, where the SPME fiber was manually introduced above the sample and heated at 50 °C for 20 min. The fiber was subsequently retracted inside the needle and immediately introduced inside the GC injection port.

Further GC−MS analysis was performed on a Shimadzu GC-17A gas chromatograph equipped with a DB-5 column (30 mL, 0.25 mm × 0.25 μm film thickness; Supelco) and coupled to a Shimadzu QP5050A mass spectrometer. Both the injector and the interface temperature were stabilized at 220 °C, whereas the VOCs' analysis was set at a specific temperature gradient. The oven temperature was first set at 38 °C for 3 min, then suddenly increased to 180 °C at a rate of 12 °C $min^{-1}$, kept at 180 °C for 5 min, and finally ramped at a rate of 40 °C $min^{-1}$ to 240 °C, before being kept at that temperature for another 5 min. The carrier gas used was helium at a flow rate of 0.9 mL $min^{-1}$. Splitless injection was used and the first 5 min of the analysis was considered as solvent delay and omitted from the final chromatograms. For any subsequent analysis, SPME was reconditioned by placing it in the injection port for 2 min at 220 °C to ensure a complete elution of any residual volatile. The HP quadrupole mass spectrometer was operated in the EI mode at 70 eV and the scan range was set at $m/z$ 40−500.

**4.4. Probiotic Bacterial Inoculation.** Assessment of probiotic culture on mango fruit aroma followed the exact protocol previously described for *Hibiscus sabdariffa* L.[25] Briefly, two mango fruit cvs were chosen for this experiment to include "sokary" (SS) and "zebdia" (ZI). Mango juice was diluted with distilled water at a ratio of 1:2, then autoclaved in a 250 mL cotton-plugged flask at 110 °C for 3 min, followed by rapid cooling at 4 °C. Inoculum was prepared by culturing *L. plantarum* stored at −70 °C in glycerol stock on a fresh MRS agar medium. A colony was used to inoculate MRS broth (100 mL) overnight, 20 mL of the culture was centrifuged, and washed two times with phosphate-buffered saline (PBS, pH 7.4). The bacterial pellet was resuspended/diluted by PBS to yield $10^9$ cfu/mL ($OD_{600}$ = 0.36). Finally, 2 mL of the adjusted culture was used to inoculate 100 mL of mango juice at initial pH = 5.0 ± 0.2.

Samples (15 mL) were aliquoted in cotton-plugged glass tubes at a regular time interval; 0, 24, and 48 h, whereas pH, viable count (cfu/mL), and volatiles were analyzed. A 20 μL aliquot was taken at each time interval and serially diluted in 180 μL peptone saline diluents. 10 μL from each dilution was spotted on the MRS agar plate and incubated for 48 h. The viable count was expressed as cfu/mL. The viable count was calculated according to eq 1.

$$\text{Viable count (cfu\mL)} = \text{count} \times \text{dilution factor} \times 10 \times 100 \quad (1)$$

VOCs' extraction and analysis of the two fermented mango juices were performed via HS-SPME coupled to GC−MS by placing 5 mL in 20 mL glass vials as previously described. Volatiles were collected from three independent bacterial inoculated mango juice cultures for assessing biological variance ($n$ = 3).

**4.5. GC−MS Analysis and Data Extraction.** VOCs were identified by comparing the retention Kovat index (RI) relative to *n*-alkanes ($C_6$−$C_{20}$), mass matching to the NIST database and the Wiley Library database and standard, whenever available. Compounds identified in relation to reference standards were marked in Table 2, including α-phellandrene (P9), *Z*-3-hexenyl-1-acetate (P10), cineol (P22), and others. Peaks were first deconvoluted using AMDIS software (www.amdis.net) prior to spectral matching. Volatile abundance data were prepared for MVA by extraction using MET-IDEA software for data extraction. Data were then subjected to PCA, HCA, and orthogonal OPLS-DA using the SIMCA-P version 13.0 software package (Umetrics, Umea Sweden). All variables were mean centered and scaled to Pareto Variance.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c06341.

GC/MS total ion chromatograms (TIC) showing a comparison between the DVB/CAR/PDMS and PDMS GC−MS PCA of the different zebdia fruits of different origins, score plot of PC1 versus PC2 scores, and loading plot for PC1 and PC2 with contributing mass peaks and


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their assignments ($n$ = 3); OPLS-DA model validation for modeling founs cv mango fruits against other cvs; OPLS-DA model validation for modeling zebdia cv mango fruits against other cvs; OPLS-DA model validation for modelling awees cv mango fruits against other cvs; volatile constituents of fermented mango juices; Egypt map showing the regions of mango samples collection; pH and viable count values for *L. plantarum* in mango juices for "sokary" (SS) and "zebdia" (ZI); relative abundances of fermented mango juice volatiles of "zebdia" Ismailia (ZI) and "sokkary" Sharqia (SS) by *L. plantarum* at different time intervals (0, 24, and 48 h) (PDF)

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### Notes

The authors declare no competing financial interest.

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