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PROFILING OF VOLATILE COMPOUNDS IN FOUR EGYPTIAN DATE CULTIVARS

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KEY WORDS:

Volatile compounds,
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ABSTRACT

Volatile compound plays an important role in consumers' satisfaction and influences further consumption of the date fruit. Thus, the study analyzed the volatile compounds of three date fruit cultivars: Barhy, Samani, Zaghloul at khalal stage of ripeness (complete color) and Siwi at tamr stage of ripeness using solid-phase microextraction (SPME) in conjunction with gas chromatography-mass spectrometry (GC-MS). A total of 69 compounds were identified, including aldehydes, alcohols, esters, terpenoids, ketones, hydrocarbons, alkanes, and ethers. Barhy dates featured high levels of aldehydes, saturated hydrocarbons, volatiles, ethers, ketones, and esters. Zaghloul dates predominantly contained aldehydes, ethers, and ketones, while Samani dates were rich in ketones, aldehydes, esters, and ethers. Key compounds such as β -(Z)-2-butenal and β -methyl ionone were identified as significant contributors to the distinctive peculiar aromas of these date varieties. Notably, the Siwi variety exhibited a distinctive profile with prominent ethers, aldehydes, ketones, and esters. The Siwi variety contained the highest number of flavour compounds (48), followed by Zaghloul (25), Barhy (20), and Samani (19). This comprehensive analysis reveals a complex and varied aromatic compounds profile among the date cultivars, with each variety having its unique sensory characteristics. The results provide valuable insights into the volatile profiles of Egyptian date varieties, potentially guiding the production of date-derived products and thus enhancing their application in food processing industries.

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ПРОФИЛИРОВАНИЕ ЛЕТУЧИХ ВЕЩЕСТВ ЧЕТЫРЕХ ЕГИПЕТСКИХ СОРТОВ ФИНИКОВ

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КЛЮЧЕВЫЕ СЛОВА: АННОТАЦИЯ

летучие соединения,
сорта фиников,
ТФМЭ, ГХ-МС анализ

Летучие соединения играют важную роль в удовлетворенности потребителей и влияют на дальнейшее потребление фиников. Таким образом, в данном исследовании были проанализированы летучие соединения трех сортов фиников: Barhy, Samani, Zaghloul на стадии халал (полный цвет) и Siwi на стадии тамр (полностью спелое подсушенное состояние) с использованием твердофазной микроэкстракции (ТФМЭ) в сочетании с газовой хроматографией-масс-спектрометрией (ГХ-МС). Всего было идентифицировано 69 соединений, включая альдегиды, спирты, сложные эфиры, терпеноиды, кетоны, углеводороды, алканы и эфиры. Финики сорта Barhy характеризовались высоким содержанием альдегидов, насыщенных углеводородов, летучих веществ, эфиров, кетонов и эфиров. Финики сорта Zaghloul в основном содержали альдегиды, простые эфиры и кетоны, в то время как финики сорта Samani были богаты кетонами, альдегидами, сложными эфирами и эфирами. Ключевые соединения, такие как β -(Z)-2-бутенал и β -метилионон, были идентифицированы как важные факторы, способствующие формированию характерных ароматов этих сортов фиников. В частности, сорт фиников Siwi продемонстрировал характерный профиль с выраженными эфирами, альдегидами, кетонами и сложными эфирами. Сорт фиников Siwi содержал наибольшее количество вкусовых соединений (48), за ним следовали сорта Zaghloul (25), Barhy (20) и Samani (19). Этот всесторонний анализ выявил сложный и разнообразный ароматический профиль среди сортов фиников, причем каждый сорт вносит свой вклад в уникальные органолептические характеристики. Результаты дают ценную информацию о профилях летучих соединений в сортах египетских фиников, определяя потенциальные пути производства продуктов, полученных из фиников, и оптимизируя их применение в перерабатывающей промышленности.

1. Introduction

The date palm (*Phoenix dactylifera* L.) is one of the most important fruit crops cultivated in arid and semi-arid regions, particularly in the Middle East and North Africa. Egypt ranks among the world's top producers, contributing approximately 1.7 million tons annually, making date palm cultivation a cornerstone of the country's agricultural sector and rural economy [1]. The fruit's resilience to high temperatures, low humidity, and limited water availability renders it highly suitable for desert

agriculture, which is critical under current and projected climate change scenarios. Dates are nutritionally substantial, offering a rich matrix of macronutrients and micronutrients. They contain 40–80% sugars — mainly glucose, fructose, and sucrose — depending on cultivar, ripening stage, and environmental factors [2]. In addition to their high sugar content, dates provide significant amounts of dietary fiber, proteins, fats, vitamins, minerals (particularly potassium, magnesium, and calcium), and bioactive compounds including phenolic acids and carotenoids,

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which contribute to their antioxidant and health-maintaining properties [3]. These attributes make dates a functional food with potential roles in glycemic regulation, cardiovascular health, and anti-inflammatory activity. In Egypt, soft varieties such as Barhi, Samani, and Zaghloul prevail in production (52 %), followed by semi-dry cultivars like Siwi (20 %), with smaller contributions from dry (3 %) and seed (25 %) dates [2]. The fruit is harvested at different ripeness stages: khalal (full size, full color, but still firm), rutab (soft and ripe), and tamr (fully ripe and dried). These stages significantly influence the chemical composition, sensory characteristics, and postharvest dynamics of the fruit condition [3]. Aroma is one of the most critical sensory attributes affecting consumers' preference, marketability, and product value. The peculiar aroma of dates arises from a complex mixture of volatile organic compounds (VOCs), including alcohols, aldehydes, esters, ketones, acids, hydrocarbons, and terpenoids [4,5]. These volatiles are biosynthesized during ripening and are further influenced by postharvest handling and processing. Reducing sugars such as glucose and fructose serve as precursors in Maillard reaction and enzymatic reactions, contributing to the generation of aroma-active compounds during thermal treatments and natural maturation. Despite their importance, the volatile profiles of Egyptian date varieties remain underexplored. Most available studies have focused on the cultivars from Tunisia, Saudi Arabia, or Algeria [3,4], with limited application of advanced analytical techniques to Egyptian varieties. Conventional extraction techniques, such as steam distillation or solvent extraction, often compromise the volatile integrity due to thermal degradation or solvent interference [6]. In contrast, solid-phase microextraction (SPME) has emerged as a preferred, solvent-free method that offers high sensitivity and selectivity in the extraction of the VOCs. When combined with gas chromatography–mass spectrometry (GC–MS), SPME allows comprehensive qualitative and quantitative profiling of complex volatile mixtures in fruits [7]. Recent studies that have used the combined SPME–GC–MS method have reported significant differences in the volatile composition of date fruits based on their cultivar and ripening stage. For instance, Bouguedoura et al. [8] identified 69 volatile compounds across three Tunisian date cultivars at different ripeness levels, observing dynamic shifts in volatile abundance: alcohols and aldehydes predominated at the lesser (early) stage, while esters and ketones increased at the rutab and tamr stages. Similarly, Saafi et al. [3] reported that alcohols, aldehydes, and hydrocarbons constituted the major chemical classes in Tunisian varieties, while others, such as terpenoids, contributed to floral and fruity notes. Furthermore, few studies have explored VOC changes in date-derived processed food products. For example, Saafi et al. [3] analyzed the date bars enriched with cereals and legumes and identified 32 aroma compounds that included alcohols, aldehydes, terpenoids, and esters, suggesting that processing can significantly alter the volatile compounds composition. This underlines the importance of understanding how thermal and mechanical processing influences the aroma profile, particularly in the context of value-added products like syrups, bars, powders, and vinegars. Nowadays there remains a clear knowledge gap concerning the aroma chemistry and volatile compounds profiles of Egyptian date cultivars, especially at critical ripening stages. Understanding these profiles is essential not only for ensuring high product quality and consumers' satisfaction but also for cultivar authentication, breeding programs, and postharvest management. Therefore, the present study aims to comprehensively identify and compare the volatile compounds profiles of selected Egyptian date fruits — specifically soft varieties (Barhi, Samani, and Zaghloul) at the khalal stage and the semi-dry variety (Siwi) at the tamr stage—using SPME combined with GC–MS. This research will contribute to filling in the existing gap in the specialised literature regarding Egyptian dates' flavor chemistry and provide valuable insights for the development of premium date products and aroma-focused programs of selection and breeding.

2. Objects and methods

2.1. Date fruits

Egyptian date varieties Barhi, Samani, and Zaghloul at the khalal stage of ripeness (complete colour) were obtained from a private farm in Giza governorate, Egypt. Siwi dates at the tamr stage of ripeness were obtained from a private farm, Dakhla Oasis, New Valley Governorate, Egypt. Date fruits were obtained during the 2022 and 2023 seasons.

2.2. Date samples preparation

The date samples were cut and finely ground. A 50 g portion of the sample was then placed into a 100 ml glass vial, and sodium chloride (150 g/kg of the sample) was added to promote the release of volatiles in the headspace and inhibit enzymatic degradation [9]. The vial was subsequently incubated (Labnet Mini Incubator I5110A located in: Union Point, GA, United States) at 50 °C for 30 minutes to facilitate the extraction of volatile compounds [10].

2.3. Extraction of volatile components by solid-phase micro extraction (SPME)

Headspace sampling was performed using Solid Phase Microextraction (SPME) devices (Supelco, Bellefonte, PA, USA) equipped with a 100 µm polydimethylsiloxane (PDMS) coating. A single process, with new fibre — preconditioned according to the manufacturer's instructions — was implemented throughout all analyses to ensure consistency. The sampling process was carried out in a temperature-controlled environment maintained at 22 ± 1 °C to provide uniform experimental conditions. Following sample equilibration, the SPME fibre was exposed to the headspace for 50 minutes. Upon completion, the fibre was retracted into its needle and immediately introduced into the injection port of the GC–MS system. All SPME sampling and desorption conditions were maintained consistently across all samples. Blank runs were performed prior to the first extraction and were randomly repeated within each experimental series. Relative peak areas of identical compounds were quantitatively compared with different samples [10,11].

Analysis of volatile compounds

After extraction, the desorption of volatile compounds from the fiber was performed in the injection port of a gas chromatography–mass spectrometry (GC–MS) system (Agilent 8890 GC System), combined with a mass spectrometer (Agilent 5977B GC/MSD). The system was equipped with an HP-5MS fused silica capillary column (30 m length, 0.25 mm internal diameter, and 0.25 µm film thickness). The injection temperature was set to 240 °C. Mass spectra were recorded in electron impact (EI) mode at 70 eV, scanning a mass-to-charge (m/z) range from 39 to 500 amu. The isolated peaks were identified by comparing them to reference data obtained from the National Institute of Standards and Technology (NIST) (USA) mass spectral library [12].

3. Results and discussion

3.1. Volatile compounds profile of Barhi date

The chromatogram for Barhi dates, presented in the Figure 1 and detailed in the Table 1, highlights 20 key volatile compounds that contribute to the distinctive aroma profile of this date variety. The data provides insights into retention times (RT), area percentages, and relative retention times (RRT), each reflecting the prominence and characteristics of the identified compounds. These volatile compounds collectively shape the complex sensory attributes of Barhi variety dates. (Z)-2-Butenal emerged as the dominant compound with an area summation of 46.46 %, detected at an RT of 2.772 minutes (RRT=1.000). This aldehyde, known for its pungent and suffocating odor, is commonly used in the production of pharmaceuticals, perfumes, and plastics. Other aldehydes, such as nonanal (RT=21.666 minutes, RRT=7.814, area=7.22 %) and decanal (RT=26.639 minutes, RRT=9.612, area=6.13 %), impart fatty, citrus-like,

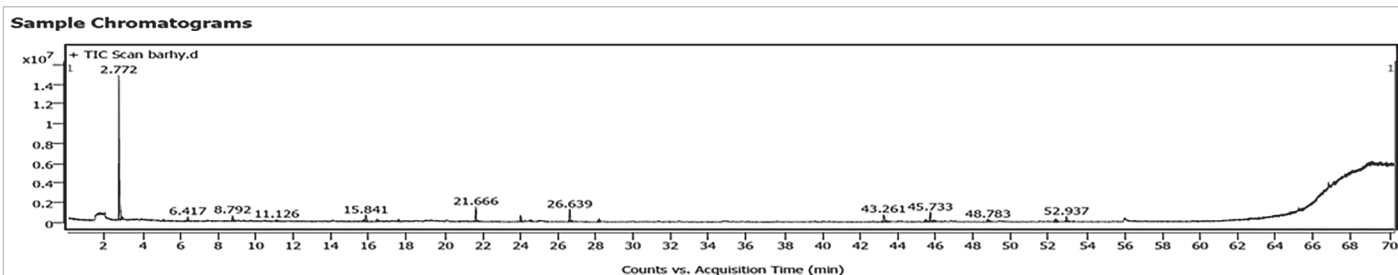


Figure 1. Chromatogram of Barhi date sample obtained by Counts vs. Acquisition time (min)

Рисунок 1. Хроматограмма образца финика сорта Barhi, полученная методом подсчета частиц в зависимости от времени их обнаружения (мин)

Table 1. Volatile compounds in Barhy Date

Таблица 1. Летучие соединения в составе финика Barhy

Peak No.	Component name	RT (min)	Area sum-mation (%)	RRT
1	(Z)-2-b utenal	2.772	46.46	1.000
2	5,7-dodecadiyn-1,12-diol	5.135	0.61	1.852
3	Hexanal	6.417	1.62	2.315
4	(E)-2-hexenal	8.792	3.09	3.172
5	Heptanal	11.126	0.78	4.015
6	Oktamethylcyklotetrasiloxan	15.841	5.15	5.715
7	Octanal	16.448	1.48	5.933
8	β -Terpinyl acetate	17.581	0.91	6.342
9	Nonanal	21.666	7.22	7.814
10	p-Menthan-3-one	24.035	3.02	8.673
11	Isomenthone	24.567	0.75	8.859
12	Decanal	26.639	6.13	9.612
13	Pulegone	28.189	1.45	10.173
14	Diethyl phthalate	43.261	6.04	15.603
15	Patchouli alcohol	45.487	1.83	16.408
16	β -methylionone	45.733	5.26	16.492
17	-(1,3-dimethylbuta-1,3-dienyl)-1,6,6-trimethyl-3,8-dioxatricyclo[5.1.0.0(2,4)] octane	45.933	0.96	16.563
18	α -hexylcinnamaldehyde	48.783	1.95	17.603
19	Galaxolide	52.382	2.38	18.899
20	Phthalic acid, isobutyl octyl ester	52.937	2.90	19.092

Retention Time (RT); Relative Retention Time (RRT)

and waxy scents that enhance the aroma profile. Ketones also play a significant role, with β -methylionone (RT=45.733 minutes, RRT=16.492) contributing 5.26% to the area. Its violet-like floral fragrance adds a sophisticated floral note to the sensory profile. Additionally, compounds such as octamethylcyclotetrasiloxane (RT=15.841 minutes, RRT=5.715, area=5.15%) subtly enhance the aroma with their volatility. Aldehydes like (E)-2-hexenal (RT=8.792 minutes, RRT=3.172, area=3.09%) provide fresh, green, and fruity notes that remind of freshly cut grass, while hexanal (RT=6.417 minutes, RRT=2.315, area=1.62%) adds a distinct grassy scent. Spicy and warm notes were identified in α -hexylcinnamaldehyde (RT=48.783 minutes, RRT=17.603, area=1.95%), contributing to the aromatic complexity. Esters such as diethyl phthalate (RT=43.261 minutes, RRT=15.603, area=6.04%) and phthalic acid, isobutyl octyl ester (RT=52.937 minutes, RRT=19.092, area=2.90%) were also detected, adding subtle aromatic elements. The floral, fruity, and waxy characteristics of these compounds balance the aroma profile. This volatile profile aligns with findings by Narain [13], who identified a variety of esters (23%), alcohols (17%), aldehydes (15%), lactones (8%), and ketones (3.5%) in date palm fruit pulp. The inclusion of RRT values in the current study further strengthens the reliability of compound identification and ensures consistency in comparing chromatographic data across different studie.

3.2. Volatile compounds profile of Zaghloul date

The chromatogram for Zaghloul dates, as shown in the Figure 2 and detailed in the Table 2, reveals the key volatile compounds that contrib-

ute to the aroma profile of these dates. The analysis identifies 25 compounds, each playing a distinct role in shaping the scent of Zaghloul dates. Below is a breakdown of the primary compounds, including their retention times (RT) and associated aromatic characteristics, diethyl phthalate: detected at 43.255 minutes with an area sum of 14.28%, this ether features slight aromatic odor. It is commonly found in personal care products such as perfumes and hair sprays, where it helps dissolve and stabilize other ingredients. Nonanal: appearing at 21.649 minutes with a presence of 12.77%, nonanal is an aldehyde with a fatty, citrus-like, and somewhat floral aroma, providing a strong waxy, citrus scent to the dates. (Z)-2-butenal: detected at 2.594 minutes with an area percentage of 10.6%, this aldehyde is commonly used as an intermediate in the production of pharmaceuticals, perfumes, and plastics. β -methyl ionone: appearing at 45.733 minutes with a 10.31% presence, this ketone has a floral, violet-like fragrance with woody undertones, adding a sophisticated floral note to the date's aroma. 2-(2-hydroxypropoxy)-1-propanol: found at 18.834 minutes, this ether, accounting for 6.7% of the area, may be used in formulations requiring both solvent and moisturizing properties.

Table 2. Volatile Compounds Profile of Zaghloul Date

Таблица 2. Профиль летучих соединений в финиках сорта Zaghloul

Peak No.	Component name	RT (min)	Area sum-mation (%)	RRT
1	Methyl formate	2.526	1.39	1.000
2	(Z)-2-butenal	2.594	10.6	1.027
3	Benzyl isopentyl ether	4.952	0.57	1.960
4	Hexanal	6.257	1.66	2.477
5	(E)-2-hexenal	8.694	0.85	3.442
6	Heptanal	11.040	1.82	4.370
7	Octanal	16.396	2.10	6.492
8	β -terpinyl acetate	17.546	2.20	6.947
9	p-hydroxybenzaldehyde	18.342	1.47	7.263
10	2-(2-hydroxypropoxy)-1-propanol	18.834	6.70	7.456
11	1-propanol, 2,2'-oxybis-	19.114	5.58	7.568
12	Citronellol hydrate	20.092	1.38	7.956
13	Nonanal	21.649	12.77	8.570
14	Cycloheptatrienone	23.806	1.21	9.426
15	p-menthan-3-one	24.023	3.35	9.511
16	Decanal	26.633	4.32	10.546
17	Pulegone	28.195	0.95	11.164
18	Diethyl phthalate	43.255	14.28	17.119
19	β -acorenol	44.045	0.87	17.433
20	Patchouli alcohol	45.493	3.60	18.020
21	β -methylionone	45.733	10.31	18.108
22	2-(4a,8-dimethyl-6-oxo-1,2,3,4,4a,5,6,8a-octahydro-2-naphthalenyl)propanal	45.939	1.69	18.191
23	α -hexylcinnamaldehyde	48.777	5.36	19.321
24	Isopropyl myristate	51.529	0.95	20.391
25	Galaxolide	52.393	4.03	20.747

Retention Time (RT); Relative Retention Time (RRT)

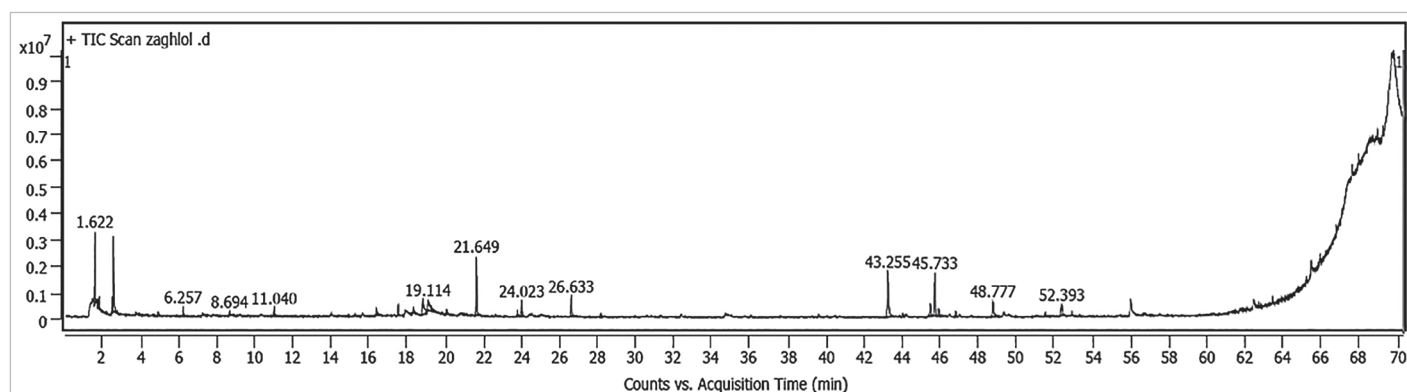


Figure 2. Chromatogram of Zaghloul date sample obtained by Counts vs. Acquisition time (min)

Рисунок 2. Хроматограмма образца финика сорта Zaghloul, полученная методом подсчета частиц в зависимости от времени их обнаружения (мин)

1-propanol, 2,2'-oxybis: detected at 19.114 minutes with a 5.58% area sum, this ether is used in industrial applications such as the production of plasticizers and hydraulic fluids. It also serves as a moisturizer in cosmetics, helping to retain moisture.

α -hexylcinnamaldehyde: detected at 48.777 minutes with a presence of 5.36%, this aldehyde has a sweet, floral, and slightly spicy aroma reminiscent of jasmine. It is used in the flavor industry to impart a mild, spicy, and floral taste. Decanal, detected at 26.633 minutes with a presence of 4.32%, this aldehyde also contributes a waxy, citrus scent, enhancing the overall aromatic complexity. Galaxolide: observed at 52.393 minutes with a 4.03% presence, Galaxolide is used in various cosmetic products for its long-lasting musky fragrance, often used in perfumes, body sprays, and deodorants. p-menthan-3-one: observed at 24.023 minutes with an area sum of 3.35%, this ketone has a minty odor similar to menthol, though less intense.

Heptanal: observed at 11.040 minutes, heptanal is an aldehyde with a fatty, slightly pungent, and fruity aroma, contributing 1.82% to the sensory profile. It is utilized in the flavor and fragrance industries to enhance the overall aroma of the dates. Hexanal: appearing at 6.257 minutes, hexanal is an aldehyde with a pungent, grassy odor. It is often used as a flavoring agent in the food industry due to its fresh aroma, contributing 1.66% to the aroma profile with a distinctive green, grassy note.

(E)-2-hexenal: detected at 8.694 minutes with an area sum of 0.85%, this aldehyde has a strong, fresh, and slightly fruity odor reminiscent of freshly cut grass. It is used in perfumes, colognes, and cosmetic products, adding a fresh, green note to the dates' scent. It is widely used in the flavor and fragrance industry, making it suitable for products like perfumes, soaps, and oral care items. This chromatogram provides a comprehensive overview of the volatile compounds that contribute to the distinctive aroma of Zaghoul dates. Each compound adds a unique element, creating a complex and multifaceted sensory experience associated with these dates.

3.3 Volatile compounds profile of Samani date

The chromatogram for Samani dates, as depicted in the Figure 3 and detailed in the Table 3, reveals the key volatile compounds that contribute to the aroma profile of these dates. It shows the results of the presence of 19 compounds responsible for the distinctive odour of the Samani. The analysis focuses on the retention times (RT) and specific aromatic characteristics of each compound. **(Z)-2-butenal** detected at 69.571 minutes with an area percentage of 38.64%, this aldehyde is commonly used as an intermediate in the production of pharmaceuticals, perfumes, and plastics. It covers the highest percentage in this date variety. It is also used as an excipient in pharmaceutical preparations, contributing to the formulation of various medications. Found at 56.141 minutes, **β -Methylionone** is a ketone belonging to the ionone family. There is 23.67%, it has a floral, violet-like fragrance with woody undertones, adding a sophisticated and aromatic note to the Samani dates. β -terpinyl acetate, observed at 65.239 minutes, is an ether with a slightly aromatic odor. It plays a crucial role in diluting and stabilizing other compounds in cosmetics, such as hair sprays, perfumes, it features a pleasant, fruity, sweet, and floral scent, often similar to lavender or lilac. This compound is used as a flavouring agent in food and beverages, imparting a fruity and floral taste which is 7.08%. 1-propanol, 2,2'-oxybis-observed at 66.813, it is esters 5.58%. Flavoring agent, due to its fruity smell, it is occasionally used as a flavoring agent in some food products. Methyl formate, detected at 68.673 minutes with 4.97%, is responsible for the distinctive smell, this aldehyde also contributes a waxy, citrus scent, enhancing the overall aromatic complexity. Detected at 49.441 minutes, **α -hexylcinnamaldehyde** is an aldehyde with a sweet, floral, and slightly spicy aroma reminiscent of jasmine or

Table 3. Volatile compounds profile of Samani date

Таблица 3. Профиль летучих веществ в финиках сорта Samani

Peak No.	Component name	RT (min)	Area summation (%)	RRT
1	p-Menthan-3-one	26.461	0.78	1.000
2	Decanal	26.633	0.62	1.006
3	α -hexylcinnamaldehyde	49.441	4.74	1.868
4	Isopropyl myristate	51.924	0.68	1.962
5	Galaxolide	52.777	1.63	1.995
6	Phthalic acid, isobutyl octyl ester	52.926	3.33	2.000
7	Diethyl phthalate	53.921	0.48	2.038
8	Patchouli alcohol	54.82	1.57	2.072
9	Nonanal	55.369	2.53	2.092
10	β -methylionone	56.141	23.67	2.122
11	Pulegone	57.126	1.05	2.159
12	Octanal	60.233	0.87	2.276
13	Hexanal	62.396	0.69	2.358
14	(E)-2-hexenal	64.101	0.62	2.422
15	Heptanal	64.702	0.49	2.445
16	β -terpinyl acetate	65.239	7.08	2.465
17	1-propanol, 2,2'-oxybis-	66.813	5.58	2.525
18	Methyl formate	68.673	4.97	2.595
19	(Z)-2-butenal	69.571	38.64	2.629

Retention Time (RT); Relative Retention Time (RRT)

other white flowers. In the taste sector, it is used to add a subtle, peppery, and floral flavor to food and beverages, enhancing the sensory experience of Samani variety dates with 4.74%. Phthalic acid, isobutyl octyl ester is observed at 52.926 minutes, this is an ester with a faint, distinctive odor. It is used in industrial applications to improve the flexibility and durability of coatings, adhesives, and sealants. This compound also contributes to the overall aroma profile of Samani variety dates. **p-menthan-3-one**, detected at 26.461 minutes, is a ketone with a pleasant minty odour. It is widely used in the flavor and fragrance industries for its refreshing scent, similar to menthol but less intense. With a presence of 0.78%, this compound is used in the production of cosmetics, soaps, and fragrances, and also serves as a flavouring additive in food products, beverages, and oral care products such as mouthwash and toothpaste. Additionally, it is found in essential oils and is utilized in aromatherapy for its calming and soothing properties. The chromatogram analysis of Samani variety dates illustrates a diverse range of volatile compounds, each uniquely contributing to the overall aromatic profile. From minty and floral notes to faint esters and ethers, these compounds collectively enhance the sensory appeal of Samani variety date.

3.4 Volatile compounds profile of Siwi date

The chromatogram of the Siwi date sample, presented in the Table 4 and illustrated in the Figure 4, reveals the key compounds responsible for the distinct aroma of Siwi dates and their respective retention times (RT) and area summation%. The graph depicts the prominence and timing of these volatile compounds acquisition, each uniquely contributing to the date's aromatic profile. Methyl formate appeared first on the chromatogram with a retention time of 3.167 minutes and a substantial area percentage of 9.84%. This compound, categorized among pleasant-smelling

Sample Chromatograms

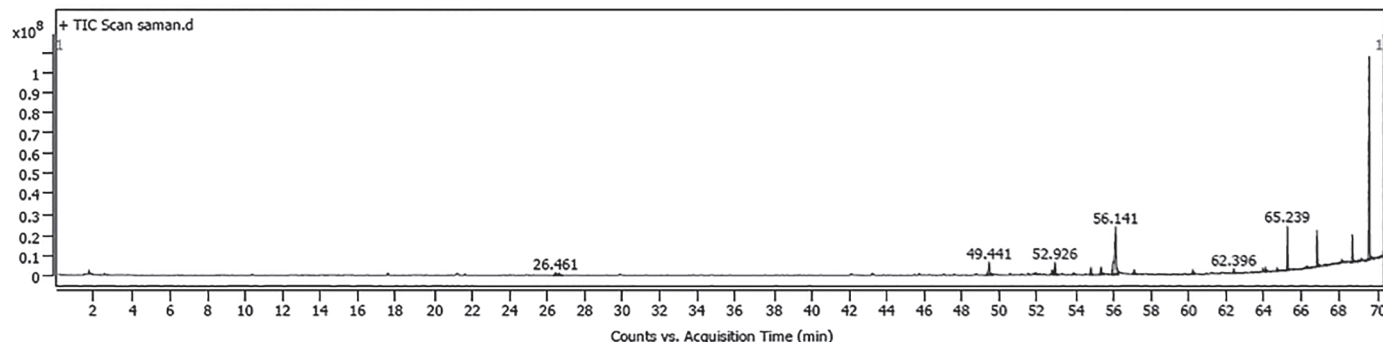


Figure 3. Chromatogram of Samani date sample obtained by Counts vs. Acquisition time (min)

Рисунок 3. Хроматограмма образца финика сорта Samani, полученная методом подсчета частиц в зависимости от времени их обнаружения (мин)

Table 4. Volatile compounds profile of Siwi Date
Таблица 4. Профиль летучих веществ фиников сорта Siwi

Peak No.	Component name	RT (min)	Area sum-mation (%)	RRT	Peak No.	Component name	RT (min)	Area sum-mation (%)	RRT
1	Benzene	2.675	4.24	1.000	26	3-carene	16.013	0.46	5.986
2	Methyl formate	3.167	9.84	1.184	27	Isophorone	16.081	1.75	6.011
3	Benzyl carbazate	4.466	2.05	1.670	28	Acetophenone	16.408	1.56	6.135
4	3-hexanone	4.895	0.64	1.831	29	cis-4-tridecene	16.791	1.88	6.277
5	2-hexanone	5.004	1.42	1.871	30	Di-n-decylsulfone	18.044	0.49	6.745
6	Hexanal	5.261	4.05	1.967	31	Nonanal	18.307	6.8	6.846
7	Hexamethylcyclotrisiloxane	6.36	2.88	2.378	32	Phenylethyl alcohol	18.817	1.38	7.034
8	p-xylene	7.533	0.91	2.816	33	Ethyl [(trimethylsilyl)oxy] (4-[(trimethylsilyl)oxy]phenyl)acetate	19.206	0.66	7.182
9	2-Methyl-1-nonene	7.67	1.41	2.868	34	Acetoxyacetic acid, nonyl ester	21.592	0.74	8.073
10	3-heptanone	8.277	3.86	3.095	35	Decanal	23.125	3.93	8.650
11	5-methyl-2-hexanone	8.454	1.41	3.162	36	β -cyclocitral	23.755	1.83	8.884
12	5-methyl-3-hexanol	8.683	1.26	3.247	37	2-undecanone	27.171	0.73	10.161
13	Heptanal	8.86	0.66	3.313	38	Carvacrol	27.646	0.52	10.333
14	Methyl N-hydroxybenzenecarboximidoate	9.747	16.76	3.643	39	Geranyl acetone	33.98	0.95	12.707
15	1-ethyl-3-methylbenzene	11.418	1.07	4.270	40	trans- β -ionone	35.33	1.03	13.209
16	Vanillin, tert-butyldimethylsilyl ether	11.601	1.93	4.336	41	Diethyl phthalate	39.559	0.87	14.793
17	1-Benzoylcyclohexanol	12.253	0.94	4.579	42	Hexadecane	39.651	0.92	14.828
18	3-octenol	12.385	1.61	4.630	43	Heptadecane	42.746	0.98	15.984
19	(+)-pinanediol	12.728	1.87	4.759	44	Farnesane	42.912	0.6	16.045
20	3-cyclohexene-1-propanal	12.86	2.43	4.808	45	4-acetyl-2,2,3,7-tetramethyltricyclo(5.2.2.0(1,6))undec-3-ene	46.162	0.71	17.264
21	Decane	13.232	0.68	4.946	46	Phthalic acid, isobutyl octadecyl ester	47.032	0.52	17.585
22	p-cymene	14.353	0.63	5.367	47	Hexadecanoic acid, methyl ester	48.256	0.41	18.041
23	Limonene	14.536	3.43	5.435	48	Hexadecanoic acid	48.88	1.61	18.275
24	2-ethyl-1-hexanol	14.742	1.96	5.510					
25	3-ethyl-3-heptanol	15.166	0.74	5.670					

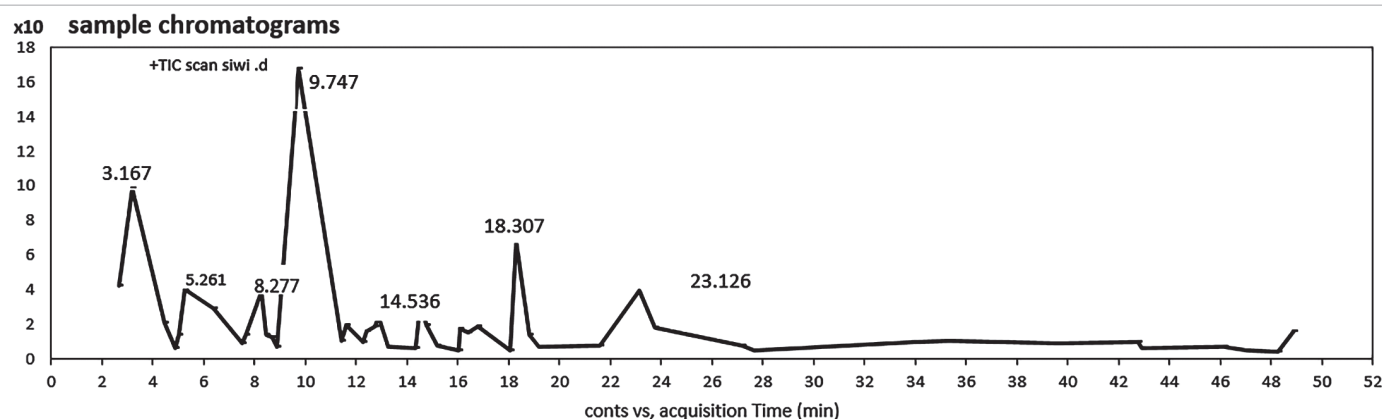


Figure 4. Chromatogram of Siwi date sample obtained by Counts vs. Acquisition Time (min)

Рисунок 4. Хроматограмма образца финика сорта Siwi, полученная методом подсчета частиц в зависимости от времени их обнаружения (мин)

esters, provides a light, ether-like aroma that is typically perceived as sweet and agreeable. The second significant compound, hexanal, was detected at 5.261 minutes with an area percentage of 4.05%. As an aldehyde, hexanal contributes a robust herbal scent, often associated with the fresh, green aroma of floral material. Following hexanal, 3-heptanone was identified at 8.277 minutes, contributing 3.86% to the total area. This ketone compound is known for its role in flavor and fragrance formulations, adding a subtle, fruity, and somewhat musty note to the aroma profile. Methyl N-hydroxybenzene carboximidoate appeared at 9.747 minutes with a significant area of 16.76%. This ester compound is noted for its ester-like properties, which contribute to the overall aromatic complexity of Siwi dates. Nonanal, detected at 18.307 minutes, represented 6.8% of the area. This aldehyde contributes a citrus-like scent with a strong waxy undertone, reminiscent of orange peel. Similarly, decanal, observed at 23.125 minutes with an area percentage of 3.93%, added to the citrus and floral notes, enhancing the aromatic profile with its distinctive waxy and somewhat sweet aroma. Benzene was detected at 2.675 minutes, representing 4.24 of the area, but did not appear in the chromatogram of the Siwi variety date, it is an aromatic hydrocarbon. Comparison with previous studies by Jado et al. [14] and Reynes et al. [15] revealed that some similarities were observed with our history samples. Results are demon-

strated when determining volatile compounds in the semi-dry date of Siwi cultivar using solid phase microextraction (SPME) and showed that the presence of 48 compounds by gas chromatography-mass spectrometry (GC-MS). Each compound, from the ether-like aroma of methyl formate to the citrusy notes of nonanal and decanal, plays a critical role in defining the sensory characteristics of this date variety. It was [16] found that the ripening stages were a genetically regulated process in molecular and biochemical changes, that lead to change of physical characterize such as colour, softening and maturation of a characteristic aroma and flavour.

3.5. The comparison analysis of volatile compounds from four Egyptian date varieties

A total of 69 specific compounds were identified across these soft varieties (Barhi, Samani and Zaghoul) at khalal stage and semi-dry (Siwi) at tamr stage (Table 5). These volatile substances were classified into eight chemical classes: alcohols, esters, aldehydes, terpenoids, ketones, saturated hydrocarbons and unsaturated hydrocarbons, alkanes, and ethers. These compounds are often associated with date flavor profiles [17]. Identified alcohols: patchouli alcohol, found in all varieties, peaked in Zaghoul (3.6%) and Samani (1.57%). This compound imparts earthy and woody notes to the aroma [18]. Identified esters methyl formate is present in all varieties, and peaked in Siwi (9.84%) and Samani (4.97%) in

Table 5. Volatile compounds in date varieties
Таблица 5. Содержание летучих веществ в сортах фиников

Chemical classes	Volatile compound	Barhy	Zaghloul	Samani	Siwi
Area summation (%)					
Alcohol	Patchouli alcohol	1.83	3.6	1.57	1.38
	Citronellol hydrate	0.00	1.38	0.00	0.00
	β -acoreanol	0.00	0.87	0.00	0.00
	Benzyl carbazate	0.00	0.00	0.00	2.05
	5,7-dodecadiyn-1,12-diol	0.61	0.00	0.00	0.00
Esters	5-methyl-3-hexanol	0.00	0.00	0.00	1.26
	Isopropyl myristate	0.00	0.95	0.68	0.00
	Methyl formate	0.00	1.39	4.97	9.84
	Methyl N-hydroxybenzenecarboximidoate	0.00	0.00	0.00	16.76
	Ethyl [(trimethylsilyl)oxy](4-[(trimethylsilyl)oxy]phenyl)acetate	0.00	0.00	0.00	0.66
Aldehyde	Acetoxyacetic acid, nonyl ester	0.00	0.00	0.00	0.74
	Hexadecanoic acid, methyl ester	0.00	0.00	0.00	0.41
	Phthalic acid, isobutyl octyl ester	2.9	00	3.33	0.52
	β -cyclocitral	0.00	0.00	0.00	1.83
	3-Cyclohexene-1-propanal	0.00	0.00	0.00	2.43
	(Z)-2-butenal	46.46	10.6	38.64	0.00
	Hexanal	1.62	1.66	0.69	4.05
	(E)-2-hexenal	3.09	0.85	0.62	0.00
	Heptanal	0.78	1.82	0.49	0.66
	Octanal	1.48	2.1	0.87	1.61
	Nonanal	7.22	12.77	2.53	6.8
	Decanal	6.13	4.32	0.62	3.93
	p-hydroxybenzaldehyde	0.00	1.47	0.00	0.00
	α -hexylcinnamaldehyde	1.95	5.36	4.74	0.00
	2-(4a,8-dimethyl-6-oxo-1,2,3,4,4a,5,6,8a-octahydro-2-naphthalenyl)propanal	0.00	1.69	0.00	0.00
Terpenoids	3-carene	0.00	0.00	0.00	0.46
Ketones	Pulegone	1.45	0.95	1.05	0.00
	β -methyl ionone	5.26	10.31	23.67	0.00
	Cycloheptatrienone	0.00	1.21	0.00	0.00
	-(1,3-dimethylbuta-1,3-dienyl)-1,6,6-trimethyl-3,8 dioxatricyclo[5.1.0.0(2,4)]octane	0.96	0.00	0.00	0.00
	Isomenthone	0.75	0.00	0.00	0.00
	3-hexanone	0.00	0.00	0.00	0.64
	2-hexanone	0.00	0.00	0.00	1.42
	3-heptanone	0.00	0.00	0.00	3.86
	5-methyl-2-hexanone	0.00	0.00	0.00	1.41
	Isophorone	0.00	0.00	0.00	1.75
	Acetophenone	0.00	0.00	0.00	1.56
	2-undecanone	0.00	0.00	0.00	0.73
	Geranyl acetone	0.00	0.00	0.00	0.95
	trans- β -ionone	0.00	0.00	0.00	1.03
	p-menthan-3-one	3.02	3.35	0.78	0.00
Hydrocarbon	Hexadecane	0.00	0.00	0.00	0.92
	Benzene	0.00	0.00	0.00	4.24
	p-xylene	0.00	0.00	0.00	0.91
	p-cymene	0.00	0.00	0.00	0.63
	Limonene	0.00	0.00	0.00	3.43
	Okta methylcyclotetrasiloxan	5.15	0.00	0.00	0.00
	1-Ethyl-3-methylbenzene	0.00	0.00	0.00	1.07
Hydroxyl	Farnesane	0.00	0.00	0.00	0.6
	Hexamethylcyclotrisiloxane	0.00	0.00	0.00	2.88
	Vanillin, tert-butyl dimethylsilyl ether	0.00	0.00	0.00	1.93
	1-Benzoylcyclohexanol	0.00	0.00	0.00	0.94
	(+)-pinanediol	0.00	0.00	0.00	1.87
Alkene	3-ethyl-3-heptanol	0.00	0.00	0.00	0.74
	Carvacrol	0.00	0.00	0.00	0.52
	4-acetyl-2,2,3,7-tetramethyltricyclo(5.2.2.0(1,6))undec-3-ene	0.00	0.00	0.00	0.71
	Decane	0.00	0.00	0.00	0.68
	cis-4-tridecene	0.00	0.00	0.00	1.88
Alkane	2-Methyl-1-nonene	0.00	0.00	0.00	1.41
	Heptadecane	0.00	0.00	0.00	0.98
Sraight-chain alkane	1-propanol, 2,2'-oxybis-	0.00	5.58	5.58	0.00
Ether	2-(2-Hydroxypropoxy)-1-propanol	0.00	6.7	0.00	0.00
	Diethyl phthalate	6.04	14.28	0.48	0.87
	β -terpinyl acetate	0.91	2.2	7.08	0.00
	Benzyl isopentyl ether	0.00	0.57	0.00	0.00
	Hexadecanoic acid	0.00	0.00	0.00	1.61
Saturated fatty acid	2-ethyl-1-hexanol	0.00	0.00	0.00	1.96
Ethyl	Di-n-decyl sulfone	0.00	0.00	0.00	0.49
Sulfone	Galaxolide	2.38	4.03	1.63	0.00
Other compounds					

Zaghloul 1.39. No results were shown in Barhi variety. The table shows that Barhi does not contain a single ester compound, which is phthalic acid, isobutyl octyl ester, representing 2.9%.

Identified aldehydes: (Z)-2-butenal, an aldehyde, was not detected in Siwi dates but had a notable presence in Barhy (46.46%) and Samani (38.64%) dates, with a moderate amount in Zaghloul (10.6%). Hexanal is another aldehyde observed in Siwi (4.05%), but is present in much smaller quantities in the other varieties. Hexanal contributes to the grassy and green aroma typical of many fruits [19]. Nonanal, a prominent aldehyde, showed the highest peak in Siwi (6.8%) and Barhy (7.22%) is associated with citrusy and floral aromas [20]. Zaghloul had a higher concentration (12.77%) than Samani (2.53%), thus suggesting a more pronounced citrus note in this variety.

Identified terpenoids group, it appears only in the Siwi variety through the 3-carene compound and represents a small amount, not exceeding 0.46%. Identified ketones: (E)-2-hexenal and heptanal, both aldehydes, were detected at lower levels across varieties, except for heptanal in Zaghloul variety (1.82%). Heptanal adds a fatty and waxy character to the aroma [21]. β -methyl ionone, a ketone, was notably higher in Samani (23.67%) compared to other varieties. This compound is known for its floral and woody notes and is often used in perfumery [22]. Pulegone, another ketone, was present in moderate amounts in Barhy (1.45%) and Samani (1.05%). It contributes minty and refreshing notes [22,23].

Identified hydrocarbons: benzene and limonene, both hydrocarbons, were detected in Siwi (4.24% and 3.43%, respectively). Limonene, in particular, is known for its citrus aroma and is commonly used in cleaning products and perfumes [24,25]. Vanillin, an aromatic compound, appeared in Siwi (1.93%) and it adds a sweet vanilla aroma, which is often valued in the flavour and fragrance industries [26].

Identified hydroxyl: hexamethylcyclotrisiloxane, vanillin, tert-butyl dimethylsilyl ether 1 benzoylcyclohexanol Identified (+)-pinanediol, 3-ethyl-3-heptanol, carvacrol. It appears only in the Siwi variety, and its

percentage does not exceed 3%. Identified diethyl phthalate, an ether, was prominent in Zaghloul (14.28%) and Barhy (6.04%), and it is known for its use as a plasticizer and its sweet, floral aroma [27,28]. Esters: methyl formate, found predominantly in Siwi (9.84%), it contributes a fruity aroma and is often used as a flavoring agent [29]. Identified phthalic acid, isobutyl octyl ester was notably present in Zaghloul (14.28%) but it is absent in other varieties. This ester typically has a sweet, fruity scent [30].

The data indicate distinct aroma profiles across the date varieties, while Siwi dates features higher concentrations of certain aldehydes and esters, contributing to their unique flavor profile. Barhy dates show a diverse range of compounds, including prominent aldehydes and ethers, while Samani dates are characterized by higher levels of ketones and alcohols. Zaghloul dates stand out with higher concentrations of certain esters and aldehydes, potentially offering a more complex aroma [30]. Identified profiling in 4 Egyptian date palm fruit varieties via SPME GC-MS showed that Siwi varieties exhibited the most distinct aroma among the studied date varieties. Understanding these volatile profiles is crucial for both flavor development and quality control in date production and processing, as they contribute significantly to the sensory attributes of the fruit.

4. Conclusion

A total of 69 compounds were identified, that included aldehydes, alcohols, esters, terpenoids, ketones, hydrocarbons, alkanes, and ethers in four Egyptian date fruit cultivars. The Siwi variety contained the highest number of flavor compounds (48), followed by Zaghloul (25), Barhy (20) and Samani (19). Notable compounds such as β -(Z)-2-butenal and β -methyl ionone were the significant contributors to the unique aroma of each date variety. The varied volatile profiles reveal complex aromatic characteristics that could facilitate the development of date-derived food products, improve quality control and optimize marketing strategies in the date industry.

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