Article

Sidr Honeys Physical and Chemical Characterization, a Comprehensive Approach through LC-MS/MS, NMR, and GC-MS Analysis

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Abstract: Honey intake is advantageous to human health due to its antioxidant, anticancer, anti-inflammatory, and antimicrobial properties, all of which are attributed to the rich bioactive compound contents. Moreover, hepatoprotective, wound healing, and gastrointestinal protective properties have been documented. Honey's nutritional value is significantly affected by its chemical composition, which varies depending on botanical and geographical origin. In particular, after Manuka honey, Sidr honey from the Ziziphus species is the most popular. The chemical compositions, physicochemical properties, bioactive compounds, and sensory characteristics of two Sidr honey samples from Egypt and Saudi Arabia were investigated in the current study. Moisture content, electrical conductivity (EC), pH, free acidity (FA), total acidity, lactone hydroxymethylfurfural (HMF) content, and diastase (α-amylase) activity were measured. By using high-performance liquid chromatography (HPLC), mass spectrometry (LC-MS/MS), nuclear magnetic resonance (^{1}HNMR), and solid-phase micro-extraction (SPME) coupled with gas chromatography (GC-MS) analyses, the sugar profile, non-volatile, and volatile compounds were also identified. The physicochemical analysis revealed the following results for Sidr honey from Saudi Arabia and Egypt, respectively: a moisture content of $18.03 \pm 0.05\%$ and $19.03 \pm 0.06\%$, EC values of 1.18 ± 0.05 and 1.16 ± 0.01 mS/cm, pH values of 4.87 ± 0.08 and 5.10 ± 0.01, FA of 37.50 ± 0.05 and 36.50 ± 0.05 meq/kg, total acidity of 41.06 ± 0.05

and 37.50 ± 0.05 meq/kg, lactone of 3.49 ± 0.005 and 1 ± 0.0 meq/kg, HMF of 20.92 ± 0.02 and 11.33 ± 0.01 mg/kg, and diastase of 59.97 ± 0.05 and 8.64 ± 0.06 g/100 g. Honey from Saudi Arabia and Egypt displayed 22.51 ± 0.05 and 26.62 ± 0.16 % glucose, 40.33 ± 0.06 and 35.28 ± 0.01% fructose, 8.94 ± 0.17, and 8.87 ± 0.01% sucrose, and 8.22 ± 0.006 and 8.13 ± 0.01% maltose, respectively. According to the International Honey Commission (IHC) and GCC Standardization Organization (GSO) regulations, the levels of glucose, fructose, sucrose, and maltose were near the standard levels. Flavonoids, sugars, vitamins, and nitrogen contents were additionally measured using LC-MS/MS, whereas GC-MS was employed to identify aldehydes, ketones, phenols, acids, esters, anthraquinone, hydrocarbons, and nitrogenous compounds. The results of a study on the effect of honey's geographic origin on its broad quality are summarized. As a result, knowing its optimal chemical and physical characteristics served as the criterion and indicator of the honey's quality.

1. Introduction

Honey is a delicious viscous fluid food manufactured by *Apis mellifera* L. domestic bees. Honey is a highly appreciated and commonly consumed nutritious food that has been utilized for many centuries as a natural sweetener or flavoring for mixtures [1]. Honey belongs to the most complex natural products because it contains approximately 200 substances, the majority of which are carbohydrates, particularly reducing sugars such as fructose and glucose. Honey contains enzymes, proteins, free amino acids, minerals, vitamins, phenolic acids, flavonoids, organic acids, other organic acids, as well as other phytochemicals in small amounts [2]. Honey's bioactive components have been connected to its health-promoting and nutritional properties. Honey seems to have antioxidant, anticancer, anti-inflammatory, antimicrobials well as antidiabetic, anti-obesity, and wound-healing properties [3–10] and have been used and proposed against viral diseases such as the COVID-19 virus [11,12]. In animal and human models, honey has effects on the cardiovascular system and has been revived as a therapy for gastrointestinal diseases, and asthma [13–15].

Honey's chemical composition is claimed to be influenced by geographical and floral conditions [16], seasonal variations, as well as the manufacturing, handling, and storage processes [17]. Thus, the chemical composition, physical qualities, sensory attributes, and medicinal properties of honey from diverse botanical sources vary widely [18]. Chromatographic and spectroscopic techniques such as high-performance liquid chromatography (HPLC), ultraviolet spectroscopy (UV), near-infrared (NIR), tandem MS (MS/MS), LC-MS/MS, solid-phase micro-extraction (SPME) coupled with gas chromatography (GC-MS), mass spectrometry (MS), nuclear magnetic resonance (NMR) and chemometric analyses were used to identify the distinct characteristics of honey [19–23]. For instance, honey quality is determined by its moisture contents, electrical conductivity (EC), pH, free acid, insoluble matter, ash, carbohydrate level, sucrose-specific rotation, sensory, and microbiological properties [19–21]. The identification of honey components' health-promoting criteria not only helps to maintain the price of honey or increase customer preference, but also helps to facilitates control/verification procedures. Accordingly, monofloral honeys have a higher market value than multifloral honeys. This is due to the distinct aroma profiles of multifloral honeys, which are the result of their unique volatile compounds composition, in addition to the rich medicinal and dietary values [24].

Sidr honey is a type of monofloral honey from the Ziziphus species, gaining popularity after Manuka honey [25]. Sidr, also known as Jujuba, grows mostly in the desert areas of Pakistan, Libya, Saud Arabia, Egypt, and Yemen [26–29]. Because of its scarcity and high price, Sidr honey is frequently adulterated [30]. As a result, determining authenticity is critical for the economy as well as consumer and producer safety. Honey's quality

is determined by its botanical source and chemical composition, which are also used to boost sales through specific labelling, such as monofloral, multifloral, and organic honeys. Researchers were assigned the task to ensure the accurate labelling of the honey type for customers, the honey industry, as well as food law enforcement organizations [31]. Researchers should also suggest techniques and metrics for assessing whether honey complies with legal requirements. In recent decades, a great deal of research has been conducted in an effort to find a suitable chemical marker reflecting the unique characteristics of honey associated with its origin. These investigations focus on the physical and chemical characteristics of honey [18,32–34].

The purpose of this research was to identify Sidr honey samples from Egypt and Saudi Arabia based on physicochemical qualities utilizing modern techniques, i.e., HPLC, LC-MS/MS, NMR, and SPME-GC-MS as part of our ongoing project, with particular emphasis on honeybees and bee products [35–42].

2. Materials and Methods

2.1. Honey Sampling

One kilogram of Saudi Arabian Sidr honey was obtained in 2020 from a regional honey market in Riyadh, Saudi Arabia, and Egyptian Sidr honey sample was collected directly from a beekeeper from Luxor areas of Upper Egypt. The honey samples were kept at 4 °C in glass jars in the dark until they were used for further analysis.

2.2. Standard Physicochemical Parameters and Pollen Analysis

The following parameters were determined for the Sidr honey samples: water content, EC, pH, free acidity (FA), total acidity, lactone, hydroxymethylfurfural (HMF) content, and diastase. All procedures have been carried out according to the International Honey Commission (IHC) regulations [43].

The moisture was determined using a digital refractometer PAL22S (Atago, Tokyo, Japan). In brief, the honey was dissolved in a water bath at 50 °C. The refractive index was measured at 20 °C after 6 min of equilibration [18].

At 20 °C, 10 g of honey was dissolved in 50 mL of distilled water. The EC cell was used for measurement. The EC values were given in mS/cm [44].

A pH meter (Mettler Tolledo, Columbus, OH, USA) was used to determine the pH value. After diluting 25 g of honey in 75 mL of water, the pH value was measured [45].

FA was calculated using the official IHC method. In 75 mL of distilled water, 10 g of honey sample was dissolved. The solution was then titrated with 0.1 M sodium hydroxide to a pH of 8.30. The results were given in meq/kg. Moreover, HMF content was measured using the spectrophotometric technique according to IHC guidelines [43].

Diastase activity was assessed as follows: A 50 mL volumetric flask was filled with 10 g of honey samples, 5 mL of acetate buffer, and 15 mL of water. The solution was diluted to 50 mL with distilled water after adding 3 mL of sodium chloride (0.5 M). An amount of 5 mL of starch solution was added to 10 mL of honey solution after warming the cocktail to 40 °C. Every 5 min, an aliquot was collected and added to 10 mL of iodine solution. The absorbance was determined at 660 nm using a double beam UV–visible spectrophotometer directly against water blank [46].

Pollen analysis was determined via first dissolving 10 g of honey sample in 20 mL of distilled water, then have it in a water bath of 45 °C, prior to centrifugation for 15 min at 1375 g (3500 rpm), after which the supernatant was discarded. The precipitate was immersed in 10 mL of distilled water and centrifuged for another 5 min. The pollen particle deposit was dispersed on a slide. The slides were then placed on a hot plate for 10 min. After drying, a drop of glycerin gelatin was dropped into it, which was then covered with a cover slip for identification [47].

2.3. Sugar Analysis

The reducing sugars (mainly fructose and glucose), as well as the sucrose and maltose content, were all measured using HPLC [48]. Sugar was analyzed at the laboratory of the Plant Protection Research Institute in Egypt. For carbohydrate separation, an APS-2 HypersilTM column (4.6 × 150 mm, particle size 5 μ, Thermo ScientificTM) equipped with a refractive index detector (RID) was used. The injection volume was 20 μL, and the flow ratio was 1 mL/min. In the solvent system, acetonitrile (ACN)/water ratio was 80:20 (*v/v*). The content of each sugar was expressed as g/100 g honey. The peak quantification involved duplicate injections and the use of average peak areas. The sugar content of honey was calculated using standards such as glucose, fructose, sucrose, and maltose.

2.4. Chemical Identification of the Compounds Using LC-LTQ-MS-MS

LC-MS/MS was used to analyze honey samples. A Shimadzu LC-10 HPLC was used, along with a Grace Vydac Everest Narrowbore C-18 column (100 mm × 2.1 mm i.d., 5 μm, 300 Å). Thermo Finnigan's LTQ Linear Ion Trap MS (San Jose, CA, USA) was used with LC-MS/MS, which has a mass range of 100–2000 *m/z*. An autosampler was used to inject a 2 μL sample. The following 40 min method was applied: A gradient was run for 30 min until 95% of ACN and 0.05% of FA were obtained after a 5 min isocratic run, using 5% ACN and 0.05% formic acid (FA). The column was then conditioned for 5 min with 5% ACN and 0.05% FA.

Foundation 3.1 Xcalibur 3.1.6610 was used to process and analyze the data. Additionally, MSConvert from the ProteoWizard suite (https://proteowizard.sourceforge.io/download.html; accessed on 25 December 2022) was used to convert the raw data files to mzXML format. The molecular network was created using the global natural products social molecular networking (GNPS) online workflow [42,49]. The spectra from the network were then verified against GNPS's spectral libraries and literature data.

Cytoscape 3.5 was used to display the networks. The molecular networks were edited and analyzed using the Cytoscape program. Each node had a label that was the parent mass. The sources of the samples are indicated by colors on a pie slice that is proportional to the number of MS/MS spectra for each parent mass.

2.5. ^{1}H-NMR Analysis

On a Jeol EX-600 spectrometer operating at 600 MHz, 1H-NMR spectra were captured [50]. Chemical shifts were referenced to the solvent peak for CD_3OD at δ^1H at 3.3 and 4.8 ppm.

2.6. Sampling of Volatile Compounds

A 100 mL Erlenmeyer flask (E-flask) was filled with approximately 4 g of honey. Aluminum foil was used to seal the E-opening flasks before being further tightened with a rubber band. The honey was equilibrated for 60 min at room temperature (252 °C) before the volatile compounds were collected from it. Using the SPME method, substances released from the honey were collected for 4 h (Supelco, Bellefonte, PA, USA). Immediately following the collection of volatiles, the SPME fiber was retracted, and the SPME needle was injected into a gas chromatography (GC) injector. For 5 min, the GC injector desorption was applied and SPME fiber was cleaned. Each sample was used at least three times under identical circumstances. The phytochemicals sampled from a headspace on SPME fiber were considered as volatile compounds.

2.7. GC-MS Analysis

To separate the volatile components, the samples were injected into a Hewlett Packard GC 6890 N chromatograph (Agilent Technologies Inc., California, CA, USA) fitted with a DB-5 column (30 m length, 0.25 mm internal diameter, and 0.25 μm stationary-phase film thicknesses). The temperature of the GC injector was 250 °C and remained constant isothermally throughout the analysis. The temperature of the GC oven was held isother-

mally at 40 °C for 2 min, then increased by 4 °C/min to 200 °C, and increased again by 10 °C/min to 280 °C, and finally held at 280 °C for 10 min phase with a constant flow of 1 mL/min. The mass spectrometer's ion source was run at 230 °C with a solvent delay of 5 min and an electron ionization energy of 70 eV. By comparing the mass spectra and retention indices of each compound to those in the NIST-2008 MS library, all compounds were identified. A relative percentage of the total peak area is used to express the sample's quantitative composition.

3. Results and Discussions

3.1. Physicochemical Parameters and Palynological Characteristics

Honey physicochemical characteristics are good indicators of its quality and a useful tool for the botanical identification of the honey [21]. Table 1 shows the moisture content, EC, pH, FA, total acidity, lactone, HMF, and diastase activity of the investigated samples. Moisture content is a key factor in yeast fermentation and is accordingly parameter to measure vulnerability or resistance to spoilage; moreover, as stated by the EU Directive (110/2001), it should not be more than 20% following processing and storage conditions [51,52]. In this study, the moisture content of Sidr honeys ranged from 18.03 ± 0.05 to 19.03 ± 0.06% for Saudi Arabia and Egypt, respectively [51,52]. Sidr honey from the Republic of Yemen ranged in moisture content from 13.4 to 16% [18]. Sidr honey from various origins was reported to have a moisture content that ranged from 14 to 17% [18].

Table 1. The physicochemical parameters of the Sidr honey samples.

Parameters	Sidr Honey from Saudi Arabia	Sidr Honey from Egypt	Normal Values	References
Moisture (%)	18.03 ± 0.05	19.03 ± 0.06	Up to 20	[18,53]
EC (mS/cm)	1.18 ± 0.05	1.16 ± 0.01	0.8	[18,54]
pH	4.87 ± 0.08	5.10 ± 0.01	3.4–6.1	[18,53]
Free acidity (meq/kg)	37.50 ± 05	36.50 ± 0.05		[18,53]
Total acidity (meq/kg)	41.06 ± 0.05	37.50 ± 0.05	Max. 50	[53]
Lactone acidity (meq/kg)	3.49 ± 0.005	1 ± 0.0		[53]
HMF (mg/kg)	20.92 ± 0.02	11.33 ± 0.01	80	[53,55]
Diastase (g/100 g)	59.97 ± 0.05	8.64 ± 0.06	Up to 80	[53,55]
Glucose (g/100 g)	22.51 ± 0.05	26.62 ± 0.16	25–28	[26,53]
Fructose (g/100 g)	40.33 ± 0.06	35.28 ± 0.01	33–36	[26,53]
Fructose/ glucose	1.79 ± 0.005	1.32 ± 0.01	0.9 to 1.35	[56]
Sucrose (g/100 g)	8.94 ± 0.17	8.87 ± 0.01	Up to 10	[26,53]
Maltose (g/100 g)	8.22 ± 0.006	8.13 ± 0.01	2–16	[53]

EC is frequently used as an alternative to ash content in routine quality control because of its close relationship to its ionic and organic acid content. Two samples of Sidr honey have high EC mS/cm of 1.18±0.05 and 1.16±0.01 for Saudi Arabia and Egypt, respectively. EC values are high compared to other honey samples from different regions of Yemen, ranging between 0.21 and 0.75 mS/cm [18]. The highest accepted value as recommended by the Council of the European Union is 0.8 mS/cm. In favor for our findings, earlier studies have suggested that the higher the EC value, the greater the mineral and acid contents [18,54,57]. EC value is determined for various samples from different regions where the EC value ranges from 0.4 to 1.18 mS/cm [18,26].

The honey's pH values were 4.87±0.08 and 5.10±0.01 for Saudi Arabia and Egypt, respectively, which fall within the standard range established by the Codex Alimentations of 3.7 to 6 [55]. According to geographical variation, Roshan et al. reported that the pH values of various samples of Sidr honey range from 4.8 to 6.96 [18]. Hegazi et al. studied

794 samples of Sidr honey that were imported into the Saudi market from 12 different countries, and found variation of pH values of 3.6 (Egypt), 5.4 (Saudi Arabia), and 7.4 (Yemen) [26]. The pH of the Saudi Arabian Ziziphus honey on the other hand was 6.14 [58].

Honey's acidity is caused by the relevance of organic acids, particularly citric acid, acetic acid, formic acid, oxalic acid, succinic acid, gluconic acid, pyruvic acid, tartaric acid, lactic acid, and maleic acid [59]. The majority of the acid in honey is gluconic acid. Organic acids and the acidity of honey have a very significant positive association [60]. The acidity of honey plays a role in its antimicrobial properties and stability [61]. FA levels in Sidr honey from Egypt and Saudi Arabia were 36.50 ± 0.05 and 37.50 ± 05 meq/kg, respectively, as shown in Table 1. FA exceeded the acceptable limit (50 meq/kg) in Talh honey derived from the Talh tree, Acacia gerrardii Benth, while Romanian acacia honey revealed the lowest value of FA at 8.6 meq/kg. FA is associated with the origin plant's nature and storage conditions. FA is significantly correlated with relative humidity and EC, but not with pH, and it is known to be completely irrelevant to honey quality and maintains the freshness of honey [62–64]. For Egyptian and Saudi Arabian honey, the amounts of free lactone were 1 ± 0.0 and 3.49 ± 0.005 meq/kg, respectively. The significant variation in lactone acidity in honey is primarily caused by the harvest year and botanical origin of secondary nectar. The maximum permitted amount is 50.00 meq/kg [34,55]. Lactones have a sweet aroma with a faintly sour undertone. Lactones contributed significantly to the overall aroma of honey and help to explain some of its exceptional resistance to microbes [65,66].

The HMF is a relative marker of honey freshness and quality. HMF concentrations in honey samples from Egypt and Saudi Arabia were 11.33 ± 0.01 and 20.92 ± 0.02 mg/kg, respectively. The Codex Alimentarius Standard Commission has set the maximum limit for HMF in honey as 40 mg/kg. Similarly, GCC Standardization Organization (GSO) recommends 80 mg/kg for honeys originating from tropical areas. HMF value higher than 40 mg/kg is associated with the hot weather and long harvest time, implying that the sugar contents have been heated and/or processed [67–69], while values lower than 40 mg/kg indicate a relative freshness.

Egyptian and Saudi Arabian honey, respectively, showed a diastase activity of 8.64 ± 0.06 and 59.97 ± 0.05 g/100 g. In the current study, the samples' diastase values fell within the acceptable range [53,55]. One of the key markers of honey freshness is diastase activity. The storage environment and heat treatment of honey affect diastase activity [70,71].

Pollen is a key ingredient in honey analysis. Pollen species are commonly used to identify the floral nectar sources that bees use for producing honey. The melissopalynological investigation of honey samples employing microscopical examination indicated the presence of the typical pollen grains. Sidr honey is distinguished by the presence of Ziziphus pollen [54,72]. In Saudi Arabia and Egypt, Ziziphus sp. (80 vs. 60%) was determined. In addition, 30% *Sesamum indicum* and 10% Trifolium sp. were determined for Egyptian honey, respectively [26,73]. The presence of *Phoenix dactylifera*, *S. indicum*, and Trifolium sp. distinguishes Saudi Arabian Sidr honey [73]. Relative pollen frequency is typically used to verify and label a honey sample according to the primary and minor nectar sources. The diversity of pollen types in honey reflected the broad spectrum of nectar sources present in the region where bees produce honey [73,74].

3.2. Main Sugar Profile

As shown in Table 1, sucrose, maltose, glucose, and fructose concentrations were reasonable, indicating that the honey's total sugar content must be at least 60 g/100 g. The content of sucrose, maltose, glucose, and fructose were 8.13 ± 0.01 vs 8.22 ± 0.006, 8.87 ± 0.01 vs. 8.94 ± 0.17, 26.62 ± 0.16 vs 22.51 ± 0.05, and 35.28 ± 0.01 vs. 40.33 ± 0.06 (g/100 g) of Egyptian and Saudi Arabian honey, respectively [75,76].

The average fructose and glucose contents of Algerian jujube (*Ziziphus lotus* Lam) honey were 40.8% and 30.7%, respectively [72]. Omani Sidr honey contained 17.0–27.5% and 23.9–38.9%, respectively, of glucose and fructose [69]. The fructose to glucose ratio in

Egyptian and Saudi Arabian honey was 1.32 and 1.79, respectively, whereas the optimal range is known to be between 0.9 and 1.35 [75,76]. Fructose-to-glucose ratios were less than 1.0, resulting in a faster honey crystallization, as the fructose/glucose ratio and humidity are indicators of honey's tendency to crystallize [48,77,78]. The crystal with the highest van der Waals and electrostatic interactions formed when the fructose/glucose ratio was 1.1 [79]. Maltose and sucrose were regarded as minor sugars in samples of honey. Honey's sucrose and maltose contents may reveal information about adulteration and the honey's botanical source [80,81]. The upper limits for sucrose and maltose were 10% and 2–16%, respectively, according to GSO [26,53]. For Sidr honey from Oman, the range of sucrose content was 0.1–17.5% [69]. Sidr honey from Saudi Arabia had maltose content values ranging from less than 1% to 3% [82,83].

3.3. Metabolites Profile Using LC-MS/MS Analysis

　　Utilizing GNPS networking and MS-MS data in positive ionization mode, the metabolomic mass profiles of honey were determined (Table 2 and Figures 1 and 2). The elution gradient of ACN and acidified water proved effective in 40 min. MS/MS experiments in positive ionization mode, as well as comparisons with data from the literature and databases such as the Natural Products Dictionary, PubMed, and GNPS [19], aided in the identification of 10 chemical constituents, as reported in Table 2, where the corresponding retention time, molecular formula, molecular ion $[M+H]^+$, and MS/MS fragment ions were also enlisted. The compounds are classified into flavonoids (4), carbohydrates (3), vitamins (2), and prenol lipids (1). Compounds no. 1, 2, 4,5, 6, 8, and 9 have already been reported in the literature [68,84–88]. Compound 1 showed ion $[M+H]^+$ at m/z 290.27 corresponding to the molecular formula $C_{15}H_{14}O_6$, was identified as catechin, and was found in both Sidr honey from Egypt and Saudi Arabia. Catechin is a flavonoid found in many natural sources, including honey, and possesses a variety of biological properties such as antioxidant, anticancer, and anti-inflammatory properties [89–91]. Eriodictyol is another flavonoid with m/z of 288.25 Da, MF $C_{15}H_{12}O_6$, and a fragment ion at m/z 270.9870 [M-18], 253.0110 [M-36], 162.9830 [M-124], 144.9750 [M-143], and 116.9910 [M-172] due to the loss of OH^{-1}, $H_2O_2{}^{2-}$, $C_6H_4O_3{}^{2-}$, $C_6H_7O_4{}^{2-}$, and $C_7H_8O_5$, respectively [88].

Table 2. The parent masses and fragments of the identified metabolites from the raw mass spectrum of honey samples from Egypt and Saudi Arabia.

No.	Compound Name	R_t	$(M+H)^+$	MF	MS-MS	ESh	SSh	Reference
Flavonoids								
1	Catechin	14.43	291.09	$C_{15}H_{14}O_6$	206.8420, 147.0610, 122.0800	+	+	[84,85]
2	5,6-Dihydroxy-7,3′,4′-Trimethoxyflavone	14.81	345.45	$C_{18}H_{16}O_7$	327.1430, 278.9170, 245.1730, 183.0340, 165.0750, 137.0500	+	+	[86]
3	Isovitexin	31.11	430.91	$C_{21}H_{20}O_{10}$	403.292, 371.3540, 311.3090	–	+	[93] https://bit.ly/3IqYxik (accessed on 22 March 2023)
4	Eriodictyol	39.98	289.23	$C_{15}H_{12}O_6$	270.9870, 253.0110, 162.9830, 144.9750, 116.9910,	–	+	[87]

Table 2. *Cont.*

No.	Compound Name	R_t	$(M+H)^+$	MF	MS-MS	ESh	SSh	Reference
Sugars								
5	Sucrose	1.76	342.40	$C_{12}H_{22}O_{11}$	180.0360, 162.0558, 144.0044	+	+	[68]
6	Maltotetraose	2.26	667.34	$C_{24}H_{42}O_{21}$	667.090, 648.40, 505.030, 486.3220, 342.3010, 325.0140, 223.0410, 180.0300	+	+	[68]
7	Maltose	3.57	325.19 $(M+H-H_2O)$	$C_{20}H_{22}O_4$	288.942, 271.0200, 258.9580, 253.0100, 241.1143, 229.0660, 162.9790, 144.9800, 135.0000, 126.9980, 108.8764, 96.860	+	+	[68] https://bit.ly/3lKLL67 (accessed on 22 March 2023)
Vitamins								
8	Biotin (B8 or H)	16.01	243.40	$C_{10}H_{16}N_2O_3S$	243.0419, 228.0072, 165.9573, 164.9543	–	+	[68]
9	Vitamin E	31.29	430.33	$C_{29}H_{50}O_2$	401.3180, 387.3050, 219.0980, 205.0260, 164.9960, 149.0590	+	-	[88] https://bit.ly/3FQzTXd (accessed on 22 March 2023)
Prenol lipid								
10	(2S,3S,4S,5R,6R)-6-[[(3S,4S,6aR,6bS,8aR,9R,12aS,14bR)-9-hydroxy-4-(hydroxymethyl)-4,6a,6b,8a,11,11,14b-heptamethyl-1,2,3,4a,5,6,7,8,9,10,12,12a,14,14a-tetradecahydropicen-3-yl]oxy]-5-[(2S,3R,4S,5R,6R)-4,5-dihydroxy-6-(hydroxymethyl)-3-[(2S,3R,4R,5R,6S)-3,4,5-trihydroxy-6-methyloxan-2-yl]oxyoxan-2-yl]oxy-3,4-dihydroxyoxane-2-carboxylic acid	21.14	943.83	$C_{48}H_{78}O_{18}$	797.3400, 781.4667, 763.1130, 635.1310, 599.3470, 581.4260, 459.1030, 441.3610, 423.3690, 405.3520, 383.4170, 323.13500, 271.2820	–	+	https://bit.ly/40BfLA9 (accessed on 22 March 2023)

R_t: Retention time, MF: molecular formula, ESh: Egyptian Sidr honey, SSh: Saudi Arabian Sidr honey, (–): No detected.

Carbohydrates constitute roughly 95% of the dry weight of honey. Sucrose and maltose (disaccharides), melezitos (trisaccharides), and maltotetraose (tetrasaccharides) were determined using LC-MS/MS analysis [68,92]. Finally, Vitamin E and maltose were identified utilizing the literature data and the GNPS library [68,88].
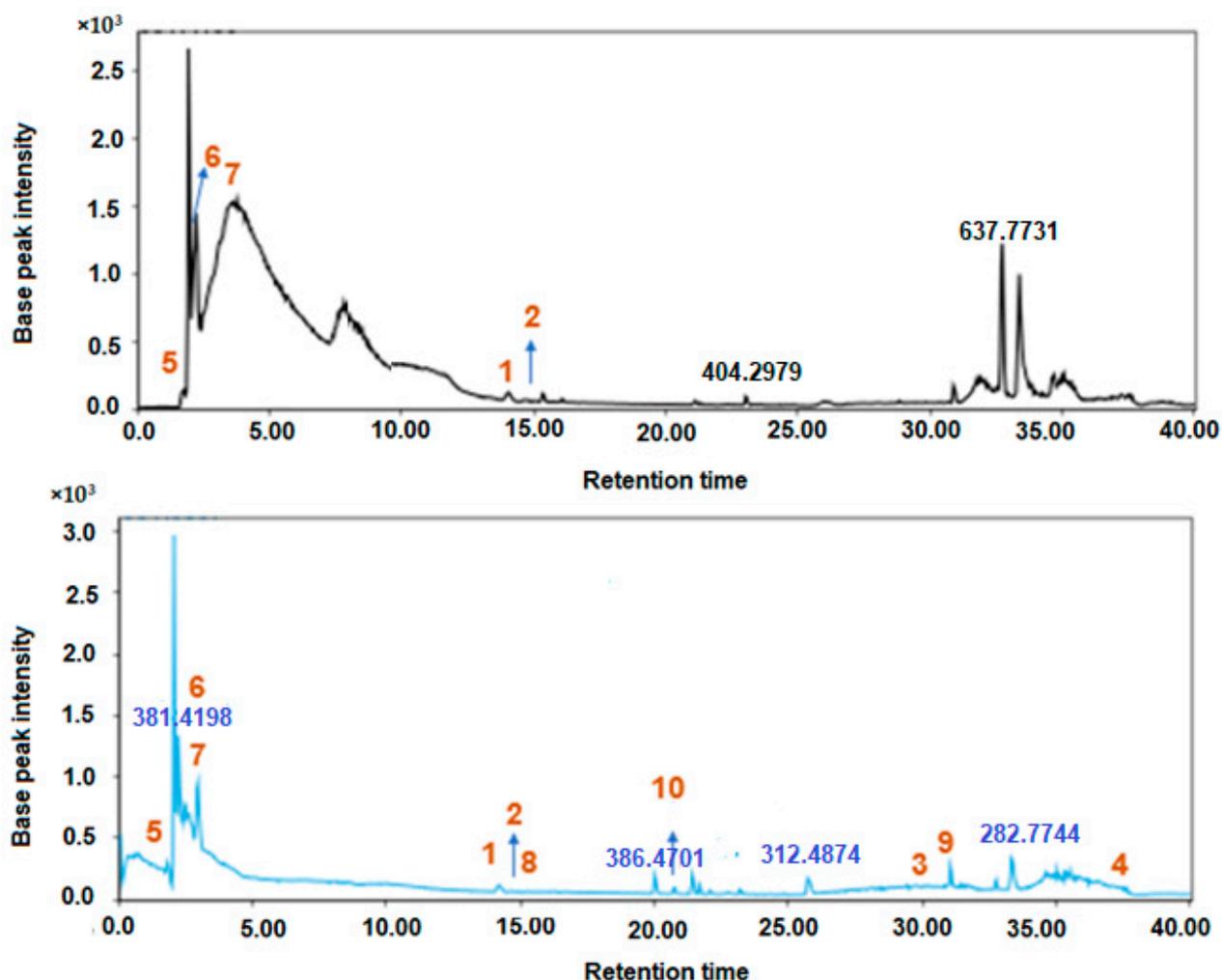
Figure 1. LC-MS/MS chromatography of identified compounds from Sidr honey from Egypt (**A**) and Saudi Arabia (**B**). 1: catechin, 2: 5,6-dihydroxy-7,3′,4′-trimethoxyflavone, 3: isovitexin, 4: eriodictyol, 5: sucrose, 6: maltotetraose, 7: maltose, 8: biotin, 9: vitamin E, 10: (2S,3S,4S,5R,6R)-6-[[(3S,4S,6aR,6bS,8aR,9R,12aS,14bR)-9-hydroxy-4-(hydroxymethyl)-4,6a,6b,8a,11,11,14b-heptamethyl-1,2,3,4a,5,6,7,8,9,10,12,12a,14,14a-tetradecahydropicen-3-yl]oxy]-5-[(2S,3R,4S,5R,6R)-4,5-dihydroxy-6-(hydroxymethyl)-3-[(2S,3R,4R,5R,6S)-3,4,5-trihydroxy-6-methyloxan-2-yl]oxyoxan-2-yl]oxy-3,4-dihydroxyoxane-2-carboxylic acid.
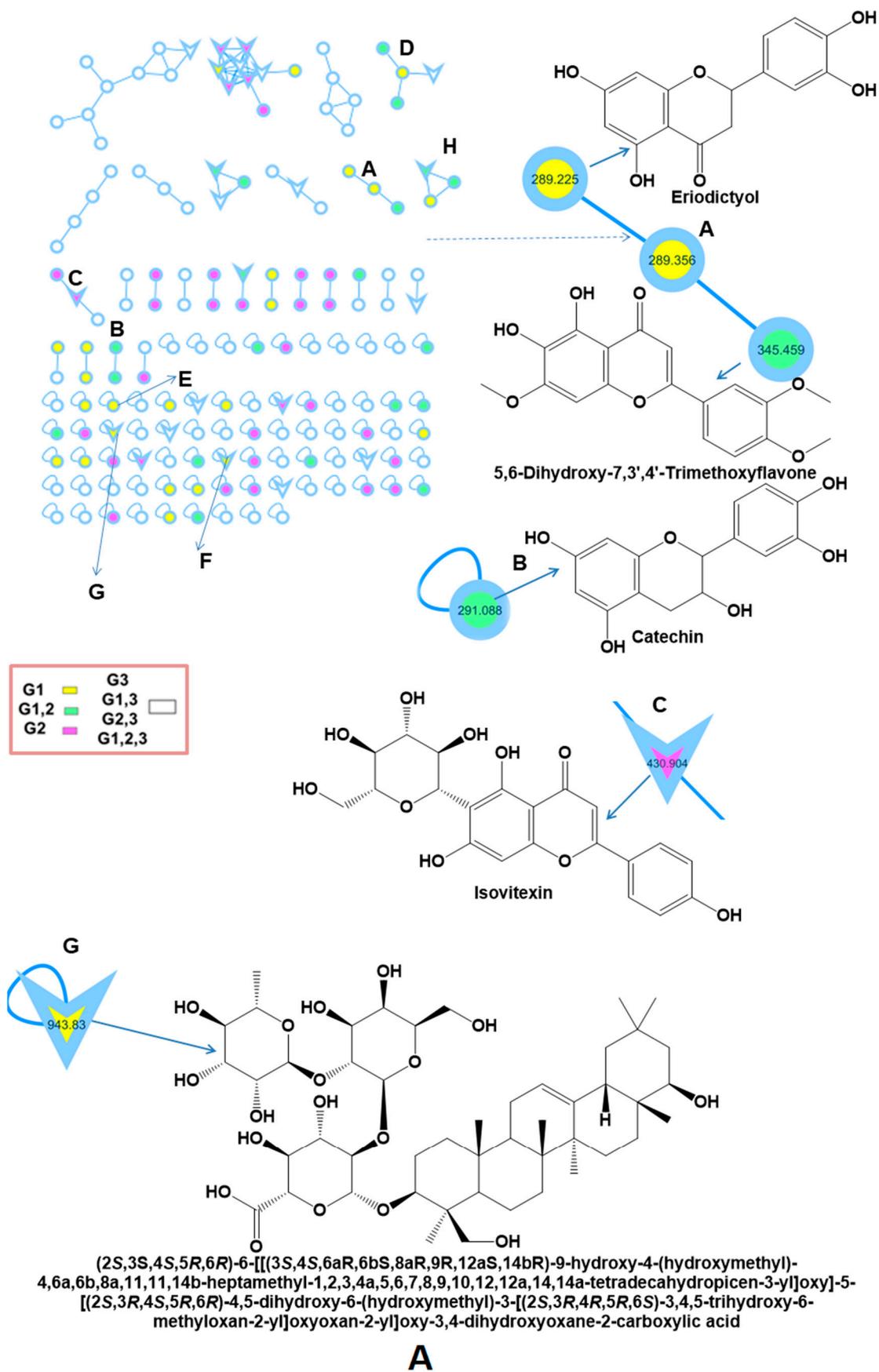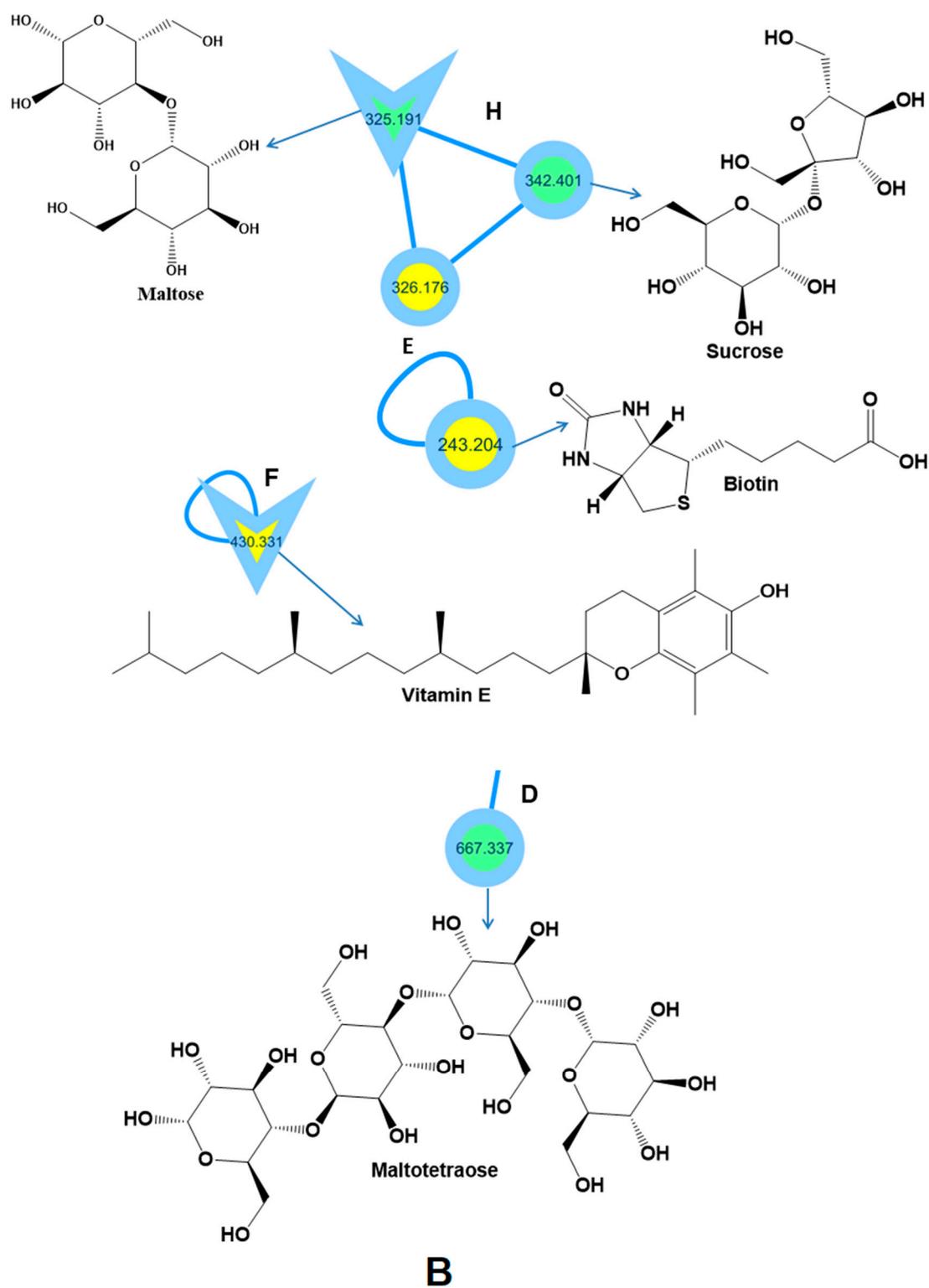
Figure 2. *Cont.*

Figure 2. Molecular networking was created for honey samples using MS/MS data in positive ionization mode. The parent mass is assigned to all nodes. The network is represented as a pie chart, with yellow (G1), green (G1,2), pink (G2), and white colors (G3, G1,2, G1,3, C1,2,3) representing the distribution of precursor ion intensity in honey from Saudi Arabia (G1), Egypt, shared compounds between honey samples, and blank solvent, respectively. The parent ions identified in the GNPS molecular network are represented by the triangle nodes. **A**: Flavonoids and prenol lipids have been detected. **B**: Sugars and vitamins were determined.

3.4. NMR Analysis

As shown in Figure 3, the ^{1}H-NMR profiles of honey samples are divided into three regions based on the chemical shift, namely the aliphatic region (δ 0–3 ppm), the carbohydrate region (δ 3–6 ppm), and the aromatic region (δ 6-10 ppm). At δ 2.50 (s), the presence of CH_2 adjusted to the carboxylic group could indicate the presence of succinic acid in the aliphatic region. As expected, the most intense and dominant signals in ^{1}H-NMR spectra of honey are in the sugar regions (δ 3.0–5.5 ppm), and typically glucose and fructose are the most intense and dominant signals. The presence of anomeric proton at δ 4.59 ppm (d) and δ 5.22 ppm (d), respectively, supported the occurrence of β- glucose and α-glucose. The signals at δ 4.20 (m) and 3.65 ppm (m) indicate the presence of fructose in both samples, whereas the peak at δ 5.45 ppm (d) indicates sucrose. The two honey samples showed signals in the region δ 8.40 (s) of formic acid. In Egyptian honey, the signal at δ 6.85 (d) indicated the presence of HMF [94].
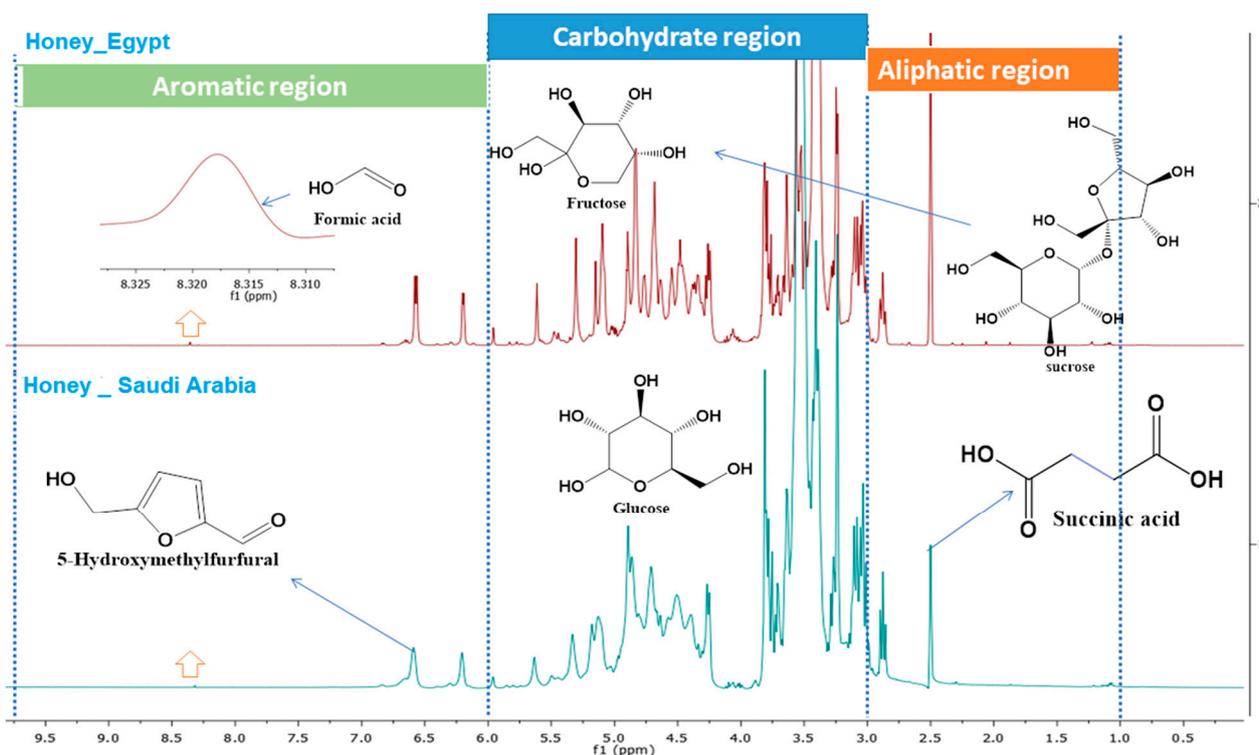

Figure 3. Selected characteristic signals of the ^{1}H-NMR as seen in the Egyptian and Saudi Arabian honey samples.

3.5. GC-MS Analysis

After performing a concise pretreatment, SPME was used to analyze the volatile and semi-volatile compounds found in samples of honey. A combination of GC-MS and SPME offers more information, including retention time (R_t), mass fragments for the volatile compounds, and more precise qualitative and quantitative results. The analysis of volatile compounds in foods, including fruits, vegetables, tea, wine, and honey, has been extensively conducted using this technique [47]. In the current study, 37 volatile compounds were identified and grouped into aldehydes (64.67 vs. 32.8%), ketones (16.54 vs. 32.04%), phenols (10.31 vs. 4.45%), acids and esters (7.46 vs. 23.01%), anthraquinone (0 vs. 6.38%), hydrocarbon (0.59 vs. 0%), and nitrogenous compounds (0.37 vs. 1.06%) for honey from Saudi Arabia and Egypt, respectively (Table 3).

Table 3. Identified volatile metabolites of honey samples using SPME coupled with GC-MS analysis.

No.	Compounds	R_t	MW	MF	MS-MS	SSH (%)	ESH (%)
				Aldehydes			
1	2-Furaldehyde	6.28	96	$C_5H_4O_2$	97, 96, 95, 67, 50, 42, 40, 39,38, 37	30.19	–
2	Benzaldehyde	10.53	106	C_7H_6O	106, 105, 77, 78, 74	5.16	–
3	2-Furaldehyde, 5-methyl-	10.65	110	$C_6H_6O_2$	111, 110, 109, 81, 53, 52, 51, 50, 39,43	22.91	–
4	Benzeneacetaldehyde	13.62	120	C_8H_8O	120, 92, 91, 89, 65, 51, 39, 63	6.19	32.06
5	Nonanal	15.78	142	$C_9H_{18}O$	124, 95, 57, 56, 55, 44, 43, 41, 39, 32	0.25	0.77
Total						**64.67**	**32.83**
Acids and esters							
6	Carbonic acid, heptyl phenyl ester	4.61	236	$C_{14}H_{20}O3$	94, 92, 91, 66, 65, 57, 50, 40, 38, 31	–	0.24
7	Hexanoic acid	5.16	116	$C_6H_{12}O_2$	73, 60, 57, 55, 45, 43, 41, 42, 32, 39	4.86	–
8	Tetronic acid	5.52	100	$C_4H_4O_3$	100, 72, 43	1.10	–
9	Isovaleric acid	6.88	102	$C_5H_{10}O_2$	87, 69, 61, 60, 45, 43, 42, 41, 39	1.50	10.48
10	Ethylmethylacetic acid	7.80	102	$C_5H_{10}O_2$	87, 74, 73, 69, 57, 56, 55, 45, 41, 39	–	11.84
11	Hexanoic acid, 3,5,5-trimethyl-	17.28	156	$C_9H_{18}O_2$	103, 83, 60, 57, 4341	–	0.69
Total						**7.46**	**23.01**
Ketones							
12	Tetrahydrofuran	3.34	72	C_4H_8O	72, 71, 42, 41	–	0.15
13	Dihydro-2-methyl-3-furanone	5.53	100	$C_5H_8O_2$	100, 72, 55, 45, 44, 42, 43	–	0.81
14	Furfural	6.63	96	$C_5H_4O_2$	97, 96,95, 67, 50, 42, 40, 39, 38, 37	–	5.54
15	1,2-Cyclopentanedione	7.11	98	$C_5H_6O_2$	98, 70, 69, 61, 56, 45, 43, 40, 39, 31	–	0.57
16	Furfural, 5-methyl-	10.65	110	$C_6H_6O_2$	111, 110, 109, 81, 53, 52, 51, 50, 43, 39	–	21.21

Table 3. *Cont.*

No.	Compounds	R$_t$	MW	MF	MS-MS	SSH (%)	ESH (%)
17	Pantolactone	13.25	130	C$_6$H$_{10}$O$_3$	72, 71, 68, 57, 56, 55, 53, 43, 41, 39	2.87	–
18	5-formylfurfural	14.90	124	C$_6$H$_4$O$_3$	125, 124, 123,95, 67, 53, 39, 38, 37	13.28	–
19	Diglycolic anhydride	15.34	116	C$_4$H$_4$O$_4$	59	–	0.03
20	Isophorone	16.46	138	C$_9$H$_{14}$O	138, 95, 83, 82, 67, 54, 53, 41, 39, 32	–	3.73
21	2,6,6-Trimethyl-2-cyclohexene-1,4-dione	17.33	152	C$_9$H$_{12}$O$_2$	152, 122, 96, 93, 73, 69, 68, 44, 43, 39	0.39	–
Total						**16.54**	**32.04**
Phenols							
22	Acetylmethylcarbinol	3.44	88	C$_4$H$_8$O$_2$	45, 43, 73, 77, 46, 41, 39	6.99	–
23	1-Butanol, 2-methyl-	4.33	99	C$_5$H$_{12}$O	41, 55, 70,57, 32, 56, 45, 42, 45, 77	+	–
24	Phenyl pentofuranoside	4.68	226	C$_{11}$H$_{14}$O$_5$	94, 73, 65, 66, 57, 51, 50, 42, 39, 31	–	1.19
25	3-Pentanol	5.36	88	C$_5$H$_{12}$O	149, 133, 74, 59, 58, 55, 41, 39, 31	1.48	–
26	4-Penten-2-ol	6.80	86	C$_5$H$_{10}$O	67, 45, 42, 39, 37	0.94	–
27	3-Hydroxymethylfuran	7.10	98	C$_5$H$_6$O$_2$	98, 97, 95, 87, 81, 69, 53, 51, 42, 41	–	1.29
28	Phenol	11.56	94	C$_6$H$_6$O	94, 66, 65	–	0.37
29	Benzyl alcohol	13.39	108	C$_7$H$_8$O	108, 107, 79, 77	+	1.21
30	*Cis*-Linaloloxide	14.75	170	C$_{10}$H$_{18}$O$_2$	111, 97, 94, 93, 83, 81, 67, 55, 59, 43	0.90	–
31	Linalol	15.78	154	C$_{10}$H$_{18}$O	93, 83, 80, 71, 69, 55	–	0.39
Total						**10.31**	**4.45**
Nitrogenous compounds							
32	Ammonium acetate	2.36	77	C$_2$H$_7$NO$_2$	75, 61, 60, 55, 45, 43, 33	–	0.66
33	Ethanamine, 2-methoxy-	5.02	75	C$_3$H$_9$NO	77, 75, 70,45	0.37	–
34	Ethanamine, N-butylidene-	11.34	99	C$_6$H$_{13}$N	99	–	0.05
35	Cyprodinil	34.18	225	C$_{14}$H$_{15}$N$_3$	225, 224, 179, 194, 85	–	0.35

Table 3. *Cont.*

No.	Compounds	R_t	MW	MF	MS-MS	SSH (%)	ESH (%)
Total						**0.37**	**1.06**
Anthraquinone							
36	Anthraquinone, 1-(o-chlorophenyl)-	12.17	318	$C_{20}H_{11}ClO_2$	284, 283, 267, 207, 193, 191, 177, 133, 125, 73	–	6.38
Total							**6.38**
Hydrocarbon							
37	Cyclohexene, 3,5,5-trimethyl-	17.32	124	C_9H_{16}	32, 40, 124,109, 69, 68, 56, 55, 53, 41	0.59	–
Total						**0.59**	–

R_t: Retention time, MW: molecular weight, MF: molecular formula, SSh: Saudi Arabian Sidr honey, ESh: Sidr honey from Egypt, (–): No detected.

Both honey samples yielded benzeneacetaldehyde, nonanal, and isovaleric acid. The major constituents were: benzeneacetaldehyde (6.19 vs. 32.06%), 2-furaldehyde, 5-methyl- (22.91 vs. 0%), furfural 5-methyl- (0 vs. 21.21%), 5-formylfurfural (13.28 vs. 0%), isovaleric acid (1.50 vs. 10.48%), and ethylmethylacetic acid (0 vs. 11.84%) for honey from Saudi Arabia and Egypt, respectively. Benzeneacetaldehyde was identified previously in chestnut, heather, honeydew, orange blossom, citrus, eucalyptus, and thyme honeys [32,95], as well as from Sidr honey [96]. Isovaleric acid was also identified in the marmeleiro and in buckwheat honey [65,97]. Both furfural, 5-methyl-, and 2-furaldehyde were extracted from honey [98,99]. Heat processing during SPME fractionation of honey volatiles could result in the presence of furan derivatives (furfural 5-methyl- and 5-formylfurfural). Carbohydrates are also responsible for the formation of furan derivatives such as HMF. Pentoses and hexoses in honey degrade in a slow enolization and a fast elimination of three molecules of water to form unfavorable compounds such as furans when they are heated or kept for a long time [100,101]. The two main recognized furans are furfural, which is derived from pentoses, and 5-HMF, which is derived from hexoses, such as glucose and fructose. These furans are used as indicators of food heat treatment [102].

4. Conclusions

Sidr honey has become increasingly popular due to its diversity in chemical composition. The physical and chemical properties of Sidr honey are determined using a combination of chromatographic and spectroscopic techniques such as high-performance liquid chromatography (HPLC), mass spectrometry (LC-MS/MS), nuclear magnetic resonance (1H-NMR), and solid-phase micro-extraction (SPME) coupled with gas chromatography (GC-MS). Based on the International Honey Commission (IHC) and GCC Standardization Organization (GSO) regulations, the values of moisture, electrical conductivity (EC), pH, free acidity (FA), total acidity, lactone, hydroxymethylfurfural (HMF), diastase, and sugars (glucose, fructose, sucrose, and maltose) are perfectly detected within the recommended levels [43]. The honey samples contained a significant number of phytoconstituents that were identified. Ten compounds from various classes of compounds, mainly flavonoids, were recognized by LC-MS/MS analysis. GC-MS analysis revealed 37 volatile compounds, the most abundant of which was benzeneacetaldehyde in Egyptian honey and furfural, 5-methyl- in Saudi Arabian honey. Overall, our research has aided in the assessment of the Sidr honey quality criteria of two geographical locations, namely Egypt and Saudi Arabia. The work offers basic knowledge about the ingredients for distinguishing the honeys and

may serve as a helpful basis for future research. Further studies using more specimens of Sidr honey from different locations may clearly show how climate and region affect the quality of the honey.

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