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Profile of Volatile Organic Compounds (VOCs) from Cold-Processed and Heat-Treated Virgin Coconut Oil (VCO) Samples †

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Abstract: Virgin coconut oil (VCO) can be prepared with or without heat. Fermentation and centrifuge processes can be done without the use of heat (cold process) and expelling involves heat due to friction. Volatile organic compounds (VOCs) from VCO samples prepared with and without the use of heat were collected using solid phase microextraction (SPME) and analyzed using gas chromatography–mass spectrometry (GC-MS). Twenty-seven VCO samples from 9 VCO producers were analyzed. The VOC profile of refined, bleached and deodorized coconut oil (RBDCO) was also obtained for comparison. Fourteen compounds were found to be common in more than 80% of the VCO samples analyzed. These included: acetic acid; C6, C8, C10, C12, and C14 fatty acids, and their corresponding delta-lactones; and C8, C10 and C12 ethyl carboxylates. Fourteen minor VOCs were likewise detected which can be grouped into five types: carboxylic acids (formic acid, butanoic acid, benzoic acid, and pentadecanoic acid), ketones (acetoin, 2-heptanone), alcohol (ethanol), aldehydes (acetaldehyde, hexanal, benzaldehyde), esters (ethyl acetate, methyl tetradecanoate), and hydrocarbons (n-hexane and toluene). Five pyrazines were detected in expeller VCO. Various hydrocarbons from C5 to C14 were noted to be higher in old RBDCO and VCO samples. There were variations in the VOCs within each VCO process as each producer used different processing times, temperatures, and drying procedures. Principal components analysis (PCA) grouped the samples according to the process used, but there were overlaps which may be due to variations in the specific procedures used by the manufacturers. These results may help VCO manufacturers control their production processes.

Keywords: virgin coconut oil (VCO); volatile organic compound (VOC); multivariate analysis; solid-phase microextraction-gas chromatography-mass spectrometry (SPME GC-MS)

1. Introduction

One who is familiar with the aroma imparted by freshly produced virgin coconut oil (VCO) can easily tell how it is produced: fresh and natural characteristic of coconut when cold-processed, or distinctly sweet coconut scent when heat-treated. VCO is obtained from the fresh mature meat of the coconut (*Cocos nucifera* L.) [1]. There are two ways to separate the oil from the coconut kernel: (1) the wet method produces coconut milk, which is subsequently processed using fermentation or centrifugation; and (2) the dry method which entails expelling the oil from coconut meat. In the wet method, heat is not required, hence this is called cold-processed. On the other hand, in the expeller process, heat is generated by friction.

The volatile organic compounds (VOCs) are responsible for the aroma of VCO. The VOCs can come from the coconut meat itself, the process of production, and from degradation processes that occur before, during, and after oil separation.

The coconut meat is the main source of the volatile compounds. For example, seventeen compounds were identified from the unroasted coconut shreds analyzed by gas chromatography (GC) after distillation [2]. It included delta-lactones and ethyl carboxylates of C8, C10, and C12, which were thought to be responsible for the mild, sweet, and pleasant coconut flavor of unroasted coconut shreds.

Some VOCs can also come from various physico-chemical and microbiological processes during oil production and storage [1,3]. Among the notable pathways are the following [4]: (1) formation and cleavage of hydroperoxides by lipoxygenases (LOX); (2) Maillard reaction; and (3) caramelization. LOX pathway reactions can be rapid at certain pH and temperature values. This pathway is responsible for the short-chain aldehydes and alcohols found in cold-pressed seeds. The Maillard reaction and caramelization are major routes for the formation of VOCs in VCO processes that involve heat in the presence of the meat or coconut milk. The Maillard reaction was suggested as the explanation for the formation of six pyrazines, two furans, and two pyrroles which were detected from the roasted coconut shreds [2]; these were thought to contribute to its uniquely strong, sweet, and nut-like scent.

During oven-drying of the coconut meat, oxidation and degradation of unsaturated lipids may contribute to the generation of aroma compounds, such as volatile alcohols, aldehydes, and ketones. Aliphatic methyl ketones and secondary alcohols were isolated from desiccated coconut [5] and were found to be responsible for the rancid off-flavour particularly those with an odd number of carbon atoms, C5-C11. 2-Heptanone and 2-nonanone were present at the highest concentration in rancid coconut. In the VCO fermentation process, the production of acetic acid leads to a rancid odor. Volatile aldehydes, which are significant aroma compounds in vegetable oils, are formed from cleavage of lipid hydroperoxides; this also leads to formation of alcohols, alkanes, alkenes, and alkynes. Fresh coconut oil, subjected to different heat treatments, yielded volatiles of n-alkanes and 1-alkenes, n-alkanals, methyl alkanones, alkenals, gamma- and delta-lactones, methyl and ethyl esters and free fatty acids. The relative amounts of these compounds varied with the type of heat treatment [6].

Previous analysis of VOCs from VCO samples prepared by three different methods detected 14 VOCs: acetic acid, ethyl acetate, the delta-lactones and ethyl carboxylates of C8 and C10, the carboxylic acids of C10 and C12, aldehydes (hexanal, nonanal), ketones (2-pentanone and 2-heptanone), and linear alkanes (octane) and a monoterpene (limonene) [7].

2. Research Objectives

This work sought to identify and quantify the VOCs from VCO samples which were produced using three processes—fermentation, centrifuge, and expeller—as well as refined, bleached and deodorized coconut oil (RBDCO). VCO samples were also collected during on-site observation of each production process. Analysis of the VOC profiles from various stages of VCO production, as well as old VCO samples, will hopefully provide valuable information to VCO producers regarding the production process.

3. Materials and Methods

3.1. VCO and RBDCO Samples

Nine VCO manufacturers were visited to observe the manufacturing process. The VCO sample codes used are as follows: fermentation (F1, F2, and F3), centrifuge (C1, C2, and C3), and expeller (E1, E2, and E3). Each producer utilized different methods and equipment to remove moisture and insoluble particles. Nine samples were submitted by the producers and 18 samples were collected during the observation visits (Table 1). The VCO samples were analyzed for their free fatty acid (%)

FFA as lauric acid) and moisture contents. In addition, five RBDCO (frying oil) samples purchased from commercial stores and one 13-year old RBDCO sample were also analyzed for VOCs.

Table 1. List of VCO samples that were analyzed.

Method of Production	Samples Submitted by Producers	Samples Collected during Production Process
Fermentation	F1-0; F2-0; F3-0	F1-1; F1-2; F2-1; F2-1; F3-1; F3-2
Centrifuge	C1-0; C2-0; C3-0	C1-1; C1-2; C2-1; C2-1; C3-1; C3-2
Expeller	E1-0; E2-0; E3-0	E1-1; E1-2; E2-1; E2-1; E3-1; E3-2

3.2. Headspace Analysis by SPME-GCMS

3.2.1. Standards

Acetic acid (100%, Merck, Darmstadt, Germany); 2-Heptanone (99%, Fluka); Octanoic acid (99%, Sigma, St. Louis, MO, USA); Ethyl octanoate (99%, Aldrich); Decanoic acid (99%, Sigma), Dodecanoic acid (98% Merck). 2-Octanol (97%, Sigma-Aldrich) was used as internal standard for calibration of retention time and quantitation.

3.2.2. Sample Preparation

The method from an earlier study [6] was used with modifications. About 5.000 g VCO sample was accurately weighed and spiked with 0.125 g of 0.02% 2-Octanol in VCO matrix solution (internal standard, IS), into a 10-mL screw neck, clear vial with screw cap, Butyl/LTPE, 18 mm septum (Supelco, Bellefonte, PA, USA). VCO matrix used in dilution of internal standard was treated with 3 M sodium hydroxide and dried with anhydrous sodium sulfate.

3.2.3. Analysis of %Free Fatty Acids (%FFA) and %Moisture Content

%FFA was quantified based on lauric acid used AOAC Official Method 940.28 (AOAC 1995). Moisture was measured by Karl Fischer titration following AOAC Official Method 984.20 (AOAC 1995).

3.2.4. Headspace SPME and GC-MS Analysis

SPME fiber used for extraction is the divinylbenzene-carboxen-polydimethylsiloxane (DVB/CAR/PDMS) fiber, 50/30 μm , Stableflex, 24 Ga, Manual Holder (Supelco, Bellefonte, PA, USA). A length of 0.8 cm fiber was exposed to the headspace of the oil at 40 $^{\circ}\text{C}$ for 20 min. and immediately desorbed into the injector port operating splitless at 250 $^{\circ}\text{C}$ while a short loop of the GC column was immersed in liquid nitrogen for 2 min. All analyses were carried out using Shimadzu GCMS-QP2010 Plus (Kyoto, Japan) with RTX-5MS[®] column (Restek, 30 m \times 0.25 mm i.d. \times 0.25 μm film thickness, Bellefonte, PA, USA) and ultra-high purity helium as carrier gas at 1.01 mL/min flow rate. The oven temperature program was as follows: initial temperature at 30 $^{\circ}\text{C}$, hold for 10 min, increase to 200 $^{\circ}\text{C}$ at 3 $^{\circ}\text{C}/\text{min}$. The interface and the ion source temperatures were set at 300 $^{\circ}\text{C}$ and 200 $^{\circ}\text{C}$, respectively. MS analysis is carried out by electron ionization at 70 eV, scanning from m/z 40 to 240. Identification of the GC peaks was carried out by comparison with the NIST library using a minimum similarity index of 90. The fiber was cleaned after each use for 60 min at 250 $^{\circ}\text{C}$ under a stream of N₂. Blank fiber analyses were done every after 10–20 runs.

3.3. Principal Component Analysis

Thirty-two samples with 60 variables were selected for principal component analysis (PCA). While most of the 60 variables were the individual major and minor VOCs detected in VCOs and RBDCOs, a few variables were groups of VOCs belonging to the same class or are isomers. No data pretreatment was done prior to analysis. PCA was done using JMP[®] Pro 14.3.0 (SAS Institute, Inc.,

Cary, NC, USA). Sparse estimation was used; it involves single value decomposition of dataset with many zeros.

4. Results

4.1. VCO Samples from Different Production Processes

Table 2 shows the process flow of sample collection from the nine manufacturers visited and the short description of the critical steps: exposure, moisture removal, particles removal and sterilization. Also shown in Table 2 are the %moisture and %free fatty acid (FFA, as lauric acid) content of the finished product.

Table 2. Process flow of VCO samples collected at production sites.

Sample	Exposure Time ^a before Oil Separation	Method of Moisture Removal from Raw VCO	Method of Particles Removal from Raw VCO	Sterilization/Heat Treatment	Analysis of the Finished Product	
					%Moisture Content	%FFA
F1-1	>24 h	Tubular centrifuge	Tubular centrifuge and fine cloth filtration	Hot water for dilution of milk; UV light on oil	0.06	0.25 ^b
F1-2					0.08	0.20
F2-1	<24 h	Tubular centrifuge and vacuum drying	Tubular centrifuge	Hot water for dilution of milk	0.10	0.13
F2-2					0.10	0.13
F3-1	<24 h	Vacuum drying	0.5 to 1.0 micron bag filtration	None	0.09	0.15
F3-2					0.10	0.12
C1-1	<24 h	Disc centrifuge	Paper filter	Hot water to aid in separation	0.09	0.05
C1-2					0.10	0.05
C2-1	<8 h	Disc centrifuge	None	None	0.10	0.09
C2-2					0.11 ^b	0.18
C3-1	<8 h	Settling and vacuum drying	Settling	Blanching of coconut meat	0.08	0.02
C3-2					0.08	0.01
E1-1	<8 h	Settling, disc centrifuge, and demister	Settling and 0.5 to 1.0 micron bag filtration	Oven-drying of ground coconut meat	0.08	0.10
E1-2					0.09	0.38 ^b
E2-1	<8 h	Settling and vacuum drying	Settling, Pressure filter with filter aids	Oven-drying of grated coconut meat	0.13 ^b	0.33 ^b
E2-2					0.12 ^b	0.17
E3-1	<8 h	Vacuum drying	Filter press	Oven-drying of ground coconut meat	0.03	0.08
E3-2					0.04	0.07

^a total time from the opening of coconut to oil separation; ^b value is outside the standard specification for %moisture and %FFA [8].

The %free fatty acid levels of F1-1, E1-2, and E2-1 were exceeded the 0.20% FFA limit and C2-2, E2-1, and E2-2 contained moisture higher than the 0.10% limit set by the Philippine National Standard for Virgin Coconut Oil[8].

4.2. Major VOCs of VCO

Depicted in Figure 1a is a sample GC-MS total ion chromatogram (TIC), and the major VOCs are labeled: hexanoic acid, delta-caprolactone, octanoic acid, ethyl octanoate, delta-octalactone, n-decanoic acid, ethyl decanoate, delta-decalactone, dodecanoic acid, ethyl dodecanoate, delta-dodecalactone, tetradecanoic acid, and delta-tetradecalactone; these are all of C6, C8, C10, C12, and C14 compounds. The structures are drawn in Figure 1b.

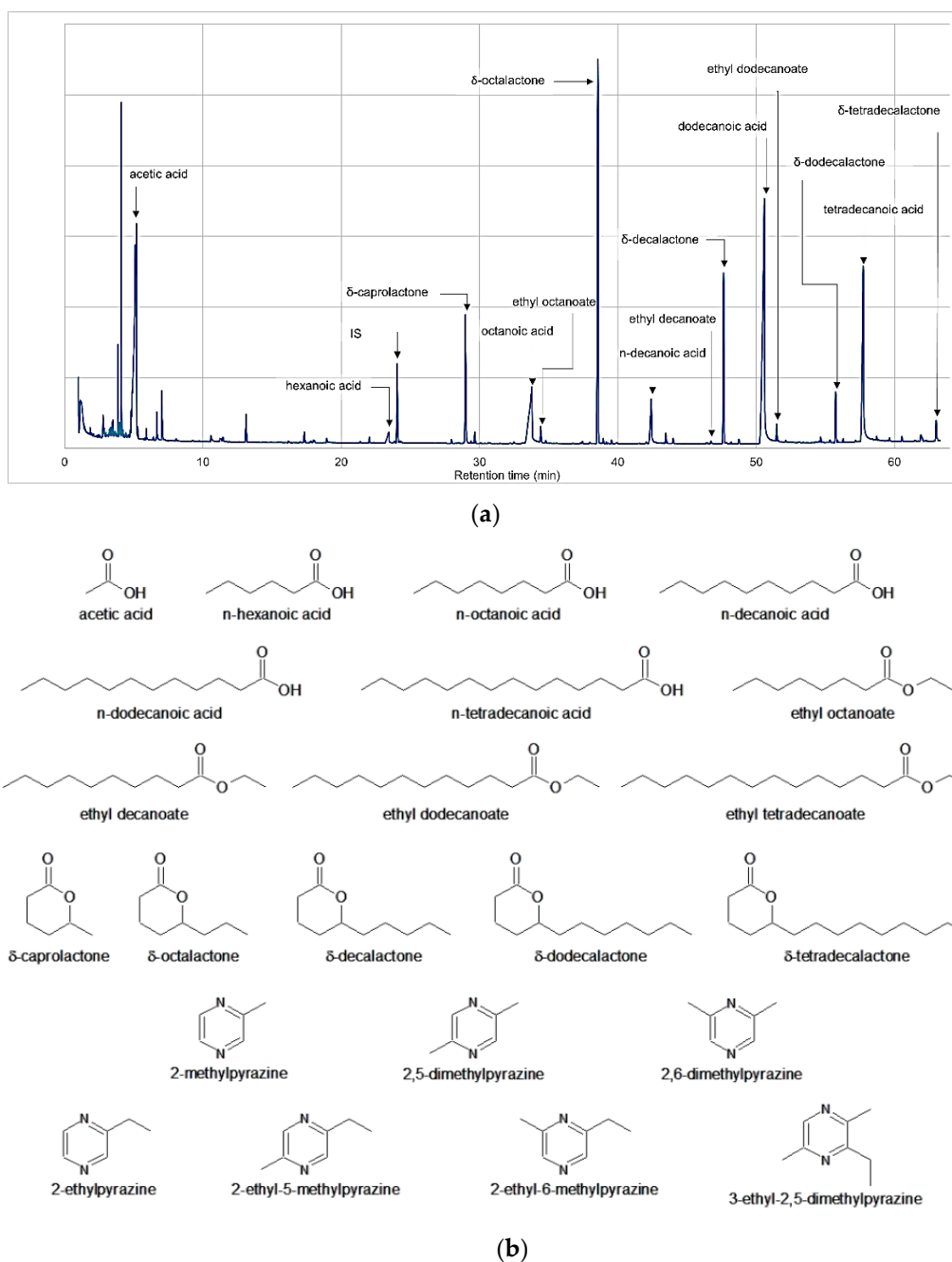


Figure 1. Major VOCs of VCO: (a) Sample GC-MS Total Ion Chromatogram (TIC) of major VOCs (IS used was 2-Octanol) of VCO produced by fermentation; and (b) Chemical structures of VOCs detected from VCO samples.

Figure 2 shows the range of normalized areas of the major VOCs. The full listings of the VOCs in the different VCOs and RBDCOs are in the Supporting Information Tables A1–A4. The C6–C12 delta-lactones, n-octanoic acid, n-dodecanoic acid and acetic acid have high %normalized areas. The rest of the major VOCs have intermediate %normalized areas. It can be observed from Figure 2 that the values even within each process are significantly variable. For some compounds, there were samples with values that are outside the $\pm 1.5 \times \text{IQR}$ (interquartile range, represented by points outside the boxes of the boxplots). Most of the major VOCs were found in more than 80% of the VCO samples analyzed. A notable exception is ethyl tetradecanoate, which was found in only six samples (3 FER and 3 EXP). n-Hexanoic acid was the only major VOC found in the RBDCO samples, and only in two out of five samples.

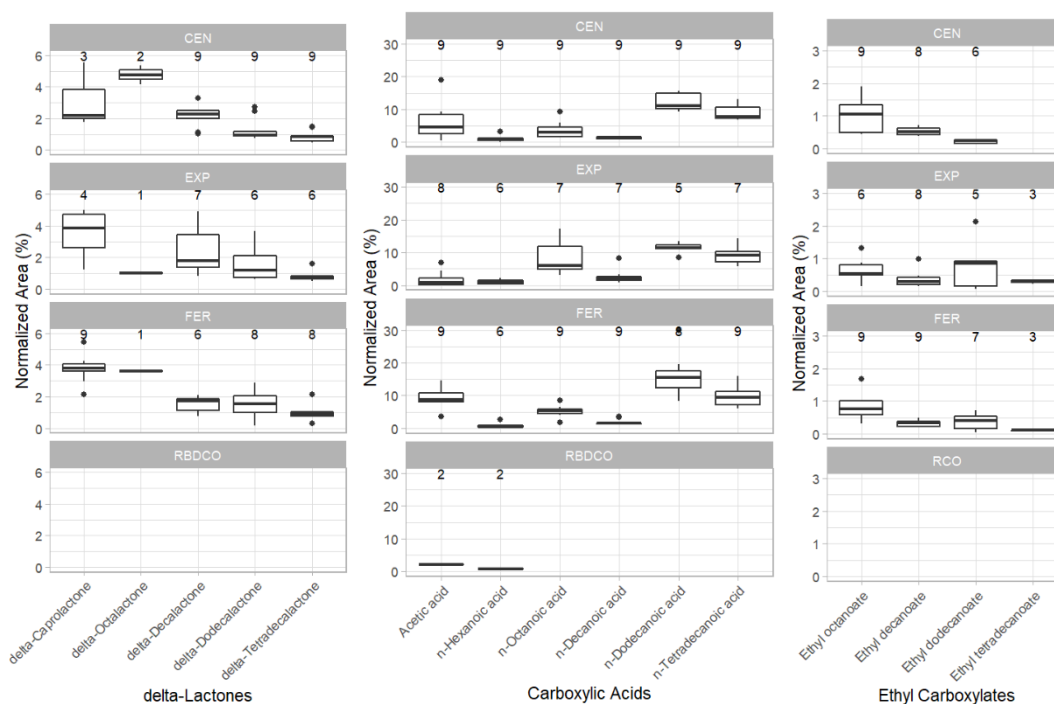


Figure 2. Range of relative amounts (based on normalized areas of the components in the GC-MS TIC) of the of the major VOCs in VCO from different processes (CEN, centrifugation; EXP, expeller; FER, fermentation) and of RBDCO. The numbers on top of every box represent the number of times the particular VOC was detected. (The total number of samples per category are 9 for CEN, EXP and FER, and 5 for RBDCO).

4.3. Minor VOCs of VCOs and RBDCOs

Figure 3 shows select minor VOCs in VCO and RBDCO samples (See also Tables A1–A4). Aldehydes and ketones were detected in the headspaces of VCO and RBDCO samples. Butanal, hexanal and nonanal were found in most of the RBDCOs but not 3-hydroxy-butanone, 2-pentanone and 2-heptanone. Pyrazines were detected in VCO samples produced by expeller method. It must be noted that the %normalized areas are not directly convertible to absolute concentrations. A high %normalized area can be a result of less number of components in a mixture. Hydrocarbons such as n-hexane, toluene and naphthalene were also detected. In addition, it was observed that other alkanes and alkenes were detected in RBDCOs, more so in the old sample, and in some submitted VCO samples (See Table A8).

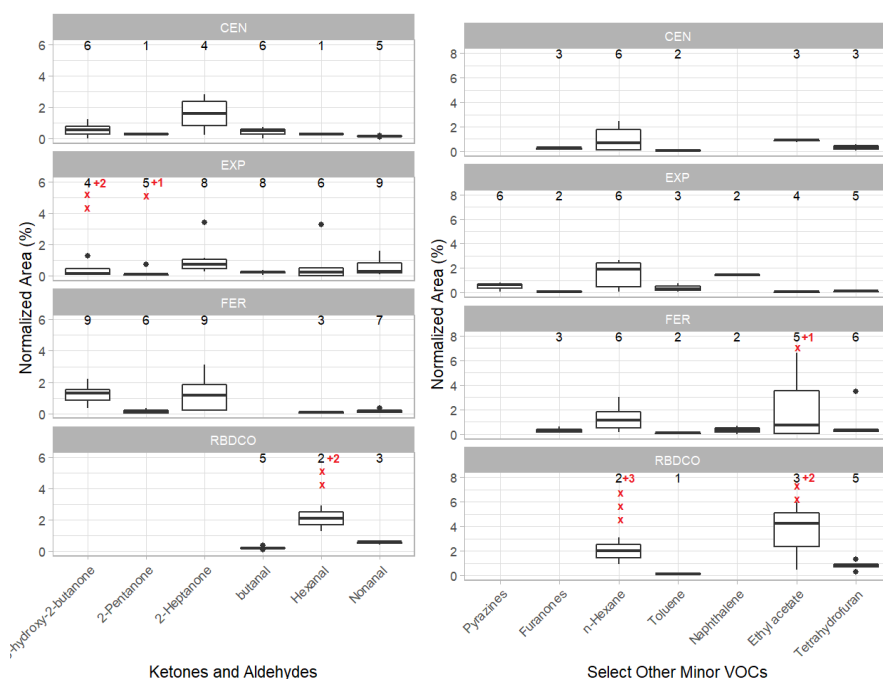


Figure 3. Range of relative amounts (based on normalized areas of the components in the GC-MS TIC) of the of select minor VOCs in VCO from different processes (CEN, centrifugation; EXP, expeller; FER, fermentation) and of RBDCO. The numbers on top of every box represent the number of times the particular VOC was detected. The total number of samples per category are 9 for CEN, EXP and FER, and 5 for RBDCO. The x mark is a datapoint beyond the scale of the graph.

4.4. PCA

Principal component analysis (PCA) was done to examine the relationship between the method of VCO production and the detected VOCs. Figure 4 shows the scores plot of the first two components. The RBDCO are clearly separated from the VCO samples. VCOs prepared by centrifugation are less variable and are more closely clustered together than those prepared by fermentation and expeller. The VCO from the expeller method showed high variability between methods used while the VCO from the fermentation method showed high variability between methods and within method (between batches).

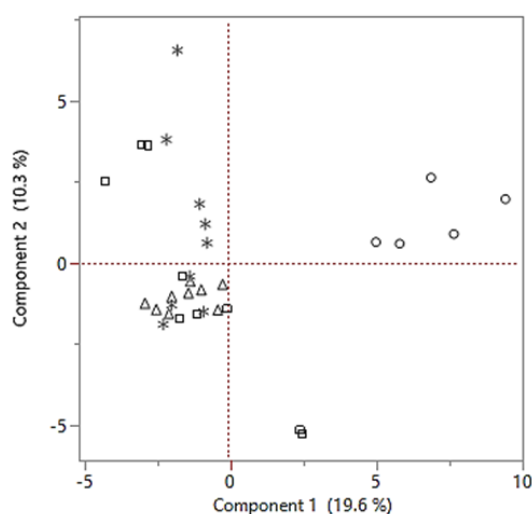


Figure 4. Scores plot of the first two components of the PCA of VOCs of VCO (Δ centrifugation; \square expeller; $*$ fermentation) and RBDCO (\circ) samples.

5. Discussion

Among the major VOCs detected in this study, the C6 and C14 delta-lactones were not observed in earlier studies; there are now five delta-lactones that are known to be present in VCO. Five fatty acids were observed, from C6 to C14. Only three of the ethyl carboxylates of these acids, C8, C10, and C12 were detected in very small quantities.

The major VOCs found in VCO samples originate from the coconut kernel. The different processes of VCO production can affect the amounts of these compounds in the VOC. In the wet methods, the amount of the major VOCs in oil is partly due to the partitioning behavior of the compounds between the aqueous and the oil layer. The partitioning of free fatty acids is further affected by the pH of the aqueous layer. The complexity of the fermentation process makes it difficult to control the final state of the aqueous layer, which results in the large variation in the VOC profiles even in samples prepared from the same method from different batches. This may explain the overlaps in the PCA profile. On the other hand, the centrifugation process produced more consistent VOC results with the least variability between methods and between batches. In the dry method, the variability in the VOC profile can be attributed to the differences in the drying methods of different VCO manufacturers.

Fourteen major compounds were consistently present in freshly prepared VCO samples but absent in RBDCOs, with the exception of hexanoic acid. Some of the minor VOCs may have developed during the production process. In the fermentation method, microbial action can lead to a wide range of organic products. Acetic acid is present in all samples but can be as low as 0.5% in abundance. The fermentation method produces more acetic acid up to 12% abundance; the amount of acetic acid also affects the free fatty acid level. Formic acid was also detected at 0–0.4% but as much as 2.7% in the old RBDCO (See Table A4). Because fermentation can also occur in coconut milk, fermentation products may also be detected in VCO produced from centrifugation. Minor VOCs detected in fermentation and centrifuge VCO included benzoic acid, butanoic acid and pentadecanoic acid. On the other hand, the drying step makes the expeller method less prone to microbial and enzymatic actions. However, the expeller method produces pyrazines from heating of the coconut meat. Ketones, in particular 3-hydroxy-2,3-butanone (acetoin) and 2-heptanone were detected at significant amounts in expeller VCO, along trace quantities of 2-pentadecanone and 2-undecanone. Hydrocarbons were also found in the samples, but more prominently in RBDCO. The 13 yr. old RBDCO sample contained high concentrations of hydrocarbons indicating that these compounds were produced during storage.

6. Conclusions

Fourteen major VOCs were detected in the headspace of VCO: acetic acid; the fatty acids and the corresponding delta-lactones of C6, C8, C10, C12, and C14, and the ethyl carboxylates of C8, C10, and C12. Fourteen minor VOCs were likewise detected. These can be grouped into five types: carboxylic acids (formic acid, butanoic acid, benzoic acid, and pentadecanoic acid), ketones (acetoin, 2-heptanone), alcohol (ethanol), aldehydes (acetaldehyde, hexanal, benzaldehyde), esters (ethyl acetate, methyl tetradecanoate), and hydrocarbons (n-hexane and toluene). Five pyrazines were detected in expeller VCO. Various hydrocarbons from C5 to C14 were noted to be higher in old RBDCO and VCO samples.

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Appendix A

Table A1. Volatile organic compounds (VOCs) in VCO produced by fermentation method.

	Volatile Compound	Retention Time	%Area (Abundance)								
			F1-0	F1-1	F1-2	F2-0	F2-1	F2-2	F3-0	F3-1	F3-2
1	Acetaldehyde	3.9–4.1	-	-	0.14	-	-	0.06	-	-	-
2	Ethanol	3.9–4.7	1.18	1.36	2.94	0.50	0.48	2.65	3.18	2.36	1.10
3	Formic acid and esters	4.2–16.0	0.26	0.37	-	-	0.21	0.30	0.24	0.26	0.34
4	Acetic acid	4.8–5.8	8.85	8.03	10.69	10.26	8.22	12.31	14.46	7.85	3.58
5	n-Hexane	4.7–5.3	-	3.01	1.25	0.15	0.97	0.35	-	-	1.99
6	2-Butanone, 3-hydroxy-	4.9–7.6	1.68	0.84	1.27	0.58	2.21	1.44	1.29	1.51	0.36
7	Ethyl Acetate	5.0–5.3	0.72	6.64	11.11	-	-	3.53	-	0.04	0.07
8	Tetrahydrofuran	5.2–5.9	0.46	0.21	0.28	-	0.13	3.50	-	0.26	-
9	1-Butanol + isomer	5.8–6.5	0.21	0.11	0.21	-	0.13	0.26	-	0.23	-
10	Butanal isomers	5.6–5.8	-	-	-	-	-	-	-	-	-
11	2-Pentanone	6.3–7.0	0.35	0.01	0.04	-	0.08	0.03	-	0.26	-
12	Toluene	9.1–9.3	-	0.03	-	-	0.13	-	-	-	-
13	2,3-Butanediol	10.2–11.7	-	0.09	0.13	-	0.80	0.40	0.57	1.06	0.38
14	Hexanal	11.3–11.5	-	-	0.05	-	0.17	-	-	-	0.06
15	Pyrazines	12.7–23.9	-	-	-	-	-	-	-	-	-
16	2-Heptanone	16.6–17.5	3.13	0.21	0.28	1.22	1.84	0.23	0.15	3.00	1.15
17	Butanoic acid, 4-hydroxy-	18.6	-	-	-	-	0.13	0.11	-	-	-
18	Benzaldehyde	21.3–21.4	0.12	-	0.03	-	0.84	0.15	-	0.08	0.20
19	Hexanoic acid	23.3–23.8	-	-	0.49	0.56	0.24	-	0.41	2.57	0.87
20	2-Octanol (internal standard)	23.9–24.1	3.23	1.72	2.24	3.80	5.76	2.12	1.91	2.53	1.75
21	δ-Caprolactone	28.9–29.0	4.26	2.98	4.07	3.79	2.12	3.61	3.77	5.46	3.94
22	Nonanal	29.6–29.7	0.04	0.06	0.17	0.20	0.19	0.14	-	-	0.35
23	2(3H)-Furanones	21.1–37.3	-	-	-	-	0.60	0.19	-	-	0.16
24	Benzoic acid + isomers	33.1–49.9	-	-	-	-	0.81	3.21	-	0.45	0.53
25	Naphthalene & derivatives	33.4–46.6	-	-	-	-	0.66	-	-	-	-
26	Octanoic acid	33.4–34.5	4.95	4.44	5.43	8.42	1.88	4.08	5.25	6.5	5.27
27	Octanoic acid, ethyl ester	34.4–34.5	0.58	0.31	0.50	1.67	0.75	1.00	0.82	1.01	0.64
28	δ-Octalactone	38.4–38.7	15.14	11.61	14.42	8.96	3.58	9.61	7.31	11.60	10.78
29	2-Undecanone	38.9–39.0	-	0.07	0.12	0.33	0.33	0.23	-	0.28	0.26
30	n-Decanoic acid	42.2–42.8	1.38	1.59	1.58	3.67	1.28	1.21	3.30	1.19	1.64
31	Decanoic acid, ethyl ester	43.4–43.5	0.34	0.20	0.29	0.49	0.21	0.40	0.23	0.37	0.33
32	δ-Decalactone	47.5–47.7	8.31	6.31	6.67	2.09	0.76	1.72	0.93	1.71	1.89
33	Dodecanoic acid, methyl ester	48.7–48.8	-	0.10	-	-	0.07	-	-	0.36	-
34	Dodecanoic acid	50.3–51.0	13.73	19.65	15.53	29.89	15.52	8.57	33.41	8.40	16.68
35	Dodecanoic acid, ethyl ester	51.4–51.5	0.71	0.53	0.53	0.41	0.04	0.17	-	0.14	-
36	γ-Dodecalactone	54.2–54.7	-	-	-	0.15	3.33	1.75	-	1.38	1.23
37	δ-Dodecalactone	55.6–55.8	2.39	1.93	1.84	0.66	2.86	-	0.29	1.20	1.08
38	Methyl tetradecanoate	56.3	-	0.10	-	-	-	-	-	-	-
39	Tetradecanoic acid	57.5–57.9	7.51	15.91	10.45	9.32	7.13	5.99	11.22	6.09	11.25
40	Tetradecanoic acid, ethyl ester	58.7–58.8	0.10	0.12	0.08	-	-	-	-	-	-
41	Pentadecanoic acid	61.0–61.1	-	-	-	-	-	-	-	3.74	0.90
42	δ-Tetradecalactone	62.9–63.1	0.75	0.82	0.73	0.30	2.12	0.95	-	1.09	0.99

Table A2. VOCs in VCO produced by centrifuge method.

	Volatile Compound	Retention Time	%Area (Abundance)								
			C1-0	C1-1	C1-2	C2-0	C2-1	C2-2	C3-0	C3-1	C3-2
1	Acetaldehyde	3.9–4.1	-	-	-	-	-	-	-	-	-
2	Ethanol	3.9–4.7	0.68	1.15	0.86	0.54	3.94	1.10	0.95	3.49	3.41
3	Formic acid	4.2–5.0	-	0.29	-	-	0.15	0.26	0.36	0.23	0.18
4	Acetic acid	4.8–5.8	8.43	4.08	-	2.52	9.33	4.43	18.94	0.67	0.46
5	n-Hexane	4.7–5.3	-	1.98	-	2.45	1.13	0.09	-	0.10	0.22
6	2-Butanone, 3-hydroxy-	4.9–7.6	-	0.48	0.24	-	1.25	0.81	0.64	0.02	-
7	Ethyl Acetate	5.0–5.3	-	0.76	0.95	-	-	0.93	-	-	-
8	Tetrahydrofuran	5.2–5.9	-	0.58	0.45	0.73	0.59	-	0.24	-	0.03
9	1-Butanol	5.8–6.5	0.48	0.09	0.16	-	0.26	0.09	-	0.16	0.15
10	Butanal isomers	5.6–5.8	-	-	-	-	0.06	-	-	-	0.02
11	2-Pentanone	6.3–7.0	-	-	-	-	0.28	-	-	-	-
12	Toluene	9.1–9.3	0.57	0.06	-	0.30	-	-	-	-	-
13	2,3-Butanediol	10.2–11.7	-	-	-	0.57	0.17	0.12	1.23	0.10	-
14	Hexanal	11.3–11.5	-	-	-	-	-	0.27	-	-	-
15	Pyrazines	12.7–23.9	-	-	-	-	-	-	-	-	-
16	2-Heptanone	16.6–17.5	-	-	-	2.83	2.19	1.01	0.24	-	-
17	Butanoic acid, 4-hydroxy-	18.6	-	-	-	0.24	-	-	-	0.19	0.19
18	Benzaldehyde	21.3–21.4	0.37	0.32	0.28	0.30	0.25	0.25	0.24	0.19	0.18
19	Hexanoic acid	23.3–23.8	0.88	0.54	0.83	3.28	0.83	1.07	1.21	0.14	0.18
20	2-Octanol (internal standard)	23.9–24.1	2.19	2.71	3.71	3.26	3.05	3.09	2.23	2.53	2.41
21	δ-Caprolactone	28.9–29.0	9.80	5.51	6.74	9.75	7.94	7.69	7.70	1.77	2.19
22	Nonanal	29.6–29.7	0.17	0.21	-	-	-	-	0.11	0.15	0.16
23	2(3H)-Furanones	21.1–37.3	-	-	-	-	-	0.09	-	0.29	0.26
24	Benzoic acid + isomers	33.1–49.9	4.71	5.14	5.98	3.95	3.14	3.85	2.94	4.40	2.92
25	Naphthalene	33.4	-	-	-	-	-	-	-	-	-
26	Octanoic acid	33.4–34.5	2.86	1.77	1.71	9.32	4.08	5.80	4.51	1.38	1.49
27	Octanoic acid, ethyl ester	34.4–34.5	0.48	0.44	0.51	0.50	1.83	1.91	1.04	1.14	1.33
28	δ-Octalactone	38.4–38.7	23.57	18.35	19.77	18.89	17.90	16.64	15.35	4.16	5.35
29	2-Undecanone	38.9–39.0	-	0.13	-	0.53	0.77	0.40	-	0.27	0.24
30	n-Decanoic acid	42.2–42.8	1.01	1.11	1.14	1.51	1.01	1.6	0.96	1.67	1.67
31	Decanoic acid, ethyl ester	43.4–43.5	0.37	0.39	0.43	-	0.58	0.63	0.42	0.61	0.72
32	δ-Decalactone	47.5–47.7	3.26	2.39	2.27	2.49	2.23	2.49	2.01	1.05	1.13
33	Dodecanoic acid, methyl ester	48.7–48.8	-	-	-	-	-	-	-	0.10	0.09
34	Dodecanoic acid	50.3–51.0	11.13	14.25	15.47	9.92	10.83	10.26	9.37	15.10	14.90
35	Dodecanoic acid, ethyl ester	51.4–51.5	-	0.14	0.13	-	0.23	0.25	-	0.27	0.28
36	γ-Dodecalactone	54.2–54.7	1.10	1.43	1.54	1.09	1.18	1.19	1.10	3.66	2.76
37	δ-Dodecalactone	55.6–55.8	0.88	1.17	1.10	0.75	0.89	0.93	0.90	2.73	2.45
38	Methyl tetradecanoate	56.3	-	-	-	-	-	-	-	0.28	-
39	Tetradecanoic acid	57.5–57.9	7.53	9.75	10.70	6.83	7.34	7.63	7.06	13.05	11.31
40	Tetradecanoic acid, ethyl ester	58.7–58.8	-	-	-	-	-	-	-	-	-
41	Pentadecanoic acid	61.0–61.1	-	-	0.36	0.34	0.29	0.47	0.25	1.12	1.29
42	δ-Tetradecalactone	62.9–63.1	0.54	0.84	0.88	0.50	0.61	0.55	0.89	1.45	1.51

Table A3. VOCs in VCO produced by expeller method.

	Volatile Compound	Retention Time	%Area (Abundance)								
			E1-0	E1-1	E1-2	E2-0	E2-1	E2-2	E3-0	E3-1	E3-2
1	Acetaldehyde	3.9–4.1	0.14	0.09	-	-	0.06	0.20	-	0.13	0.25
2	Ethanol	3.9–4.7	0.51	0.17	0.02	0.43	0.25	0.28	-	2.46	0.91
3	Formic acid	4.2–5.0	0.24	0.12	-	2.14	0.07	-	-	-	-
4	Acetic acid	4.8–5.8	0.38	0.26	0.05	4.31	0.08	-	6.82	1.22	1.39
5	n-Hexane	4.7–5.3	-	0.09	0.05	2.68	1.58	-	-	2.46	2.21
6	2-Butanone, 3-hydroxy-	4.9–7.6	-	0.10	-	1.30	0.06	0.18	-	6.53	23.19
7	Ethyl Acetate	5.0–5.3	-	0.03	0.07	-	-	-	-	0.13	0.02
8	Tetrahydrofuran	5.2–5.9	0.07	0.27	0.10	0.24	0.30	0.37	-	0.27	0.22
9	1-Butanol	5.8–6.5	-	0.02	-	-	-	0.06	-	-	0.71
10	Butanal isomers	5.6–5.8	-	0.05	-	-	-	-	-	0.79	0.29
11	2-Pentanone	6.3–7.0	0.06	0.09	0.03	-	0.75	0.09	-	-	-
12	Toluene	9.1–9.3	0.15	0.07	0.01	-	-	-	-	0.24	0.12
13	2,3-Butanediol	10.2–11.7	-	-	-	1.24	0.80	3.40	8.31	20.23	23.79
14	Hexanal	11.3–11.5	0.05	0.04	0.03	3.29	-	-	-	0.43	0.55
15	Pyrazines	12.7–23.9	0.10	0.74	-	-	0.69	0.29	-	0.83	0.63

16	2-Heptanone	16.6–17.5	0.80	1.04	0.29	3.41	12.06	1.15	0.71	0.27	0.55
17	Butanoic acid, 4-hydroxy-	18.6	-	-	-	0.22	0.07	0.14	-	0.30	0.04
18	Benzaldehyde	21.3–21.4	0.10	0.09	-	-	0.12	0.25	-	0.21	0.16
19	Hexanoic acid	23.3–23.8	0.34	0.08	0.63	2.06	1.70	1.21	-	-	-
20	2-Octanol (internal standard)	23.9–24.1	1.84	5.72	0.99	1.99	1.92	3.13	4.82	7.36	8.07
21	δ-Caprolactone	28.9–29.0	3.07	4.66	1.22	6.89	5.01	9.59	17.14	10.04	8.50
22	Nonanal	29.6–29.7	0.27	0.29	0.09	0.41	0.14	0.21	0.82	1.60	1.61
23	2(3H)-Furanones	21.1–37.3	0.07	-	-	-	0.07	-	-	-	-
24	Benzoic acid + isomers	33.1–49.9	-	-	-	-	-	-	-	-	-
25	Naphthalene	33.4	-	-	-	1.39	-	1.51	-	-	-
26	Octanoic acid	33.4–34.5	6.00	3.05	17.27	8.34	15.49	5.70	4.14	-	-
27	Octanoic acid, ethyl ester	34.4–34.5	0.50	1.31	-	0.48	0.16	0.57	-	0.87	0.99
28	δ-Octalactone	38.4–38.7	14.06	22.77	7.67	12.31	10.59	20.47	30.55	14.43	13.32
29	2-Undecanone	38.9–39.0	0.32	0.53	0.11	0.49	0.43	0.67	0.83	-	-
30	n-Decanoic acid	42.2–42.8	3.21	1.82	8.30	1.52	1.89	1.38	0.85	-	-
31	Decanoic acid, ethyl ester	43.4–43.5	0.33	0.98	0.41	0.25	0.15	0.47	-	0.17	0.21
32	δ-Decalactone	47.5–47.7	7.86	13.90	4.93	1.80	1.67	2.76	4.10	1.06	0.82
33	Dodecanoic acid, methyl ester	48.7–48.8	0.44	0.25	0.04	-	0.53	-	-	0.17	0.16
34	Dodecanoic acid	50.3–51.0	30.56	13.31	37.25	11.18	11.40	12.32	8.35	-	-
35	Dodecanoic acid, ethyl ester	51.4–51.5	0.91	2.13	0.84	-	0.06	0.16	-	-	-
36	γ-Dodecalactone	54.2–54.7	0.25	0.17	0.03	0.86	0.79	0.62	-	-	-
37	δ-Dodecalactone	55.6–55.8	2.31	3.65	1.58	0.74	0.66	0.80	-	-	-
38	Methyl tetradecanoate	56.3	0.49	0.21	0.03	-	-	-	-	-	-
39	Tetradecanoic acid	57.5–57.9	10.95	6.10	14.39	9.06	8.30	9.50	5.71	-	-
40	Tetradecanoic acid, ethyl ester	58.7–58.8	0.22	0.35	0.30	-	-	-	-	-	-
41	Pentadecanoic acid	61.0–61.1	0.09	-	-	1.11	0.89	0.75	-	-	-
42	δ-Tetradecalactone	62.9–63.1	0.86	1.60	0.69	0.73	0.49	0.62	-	-	-

Table A4. VOCs in refined, bleached and deodorized coconut oil (RBDCO).

	Volatiles	Retention Time	% Area (Abundance)					
			RCO-1	RCO-2	RCO-3	RCO-4	RCO-5	Old RCO
1	Acetaldehyde	3.9–4.1	0.18	0.57	0.66	2.25	1.47	0.13
2	Ethanol	3.9–4.7	0.33	0.86	1.18	2.26	0.45	0.21
3	Formic acid	4.2–5.0	-	-	-	-	-	2.73
4	Acetic acid	4.8–5.8	4.16	2.10	3.06	0.84	2.18	1.22
5	n-Hexane	4.7–5.3	0.91	9.41	16.20	12.34	3.10	3.17
6	2-Butanone, 3-hydroxy-	4.9–7.6	-	-	-	-	-	-
7	Ethyl Acetate	5.0–5.3	5.94	14.99	26.82	4.25	0.44	0.56
8	Tetrahydrofuran	5.2–5.9	0.37	0.21	0.11	0.18	0.18	0.24
9	1-Butanol	5.8–6.5	0.14	0.08	-	-	0.20	-
10	Butanal isomers	5.6–5.8	-	0.13	-	-	-	-
11	2-Pentanone	6.3–7.0	-	-	-	-	-	0.05
12	Toluene	9.1–9.3	0.88	0.72	1.32	0.93	0.28	3.39
13	2,3-Butanediol	10.2–11.7	-	-	-	-	-	-
14	Hexanal	11.3–11.5	1.28	2.91	9.95	11.27	12.56	50.99
15	Pyrazines	12.7–23.9	-	-	-	-	-	-
16	2-Heptanone	16.6–17.5	-	-	-	-	-	0.34
17	Butanoic acid isomers	18.6	0.03	-	-	-	-	-
18	Benzaldehyde	21.3–21.4	0.42	-	-	-	-	-
19	Hexanoic acid	23.3–23.8	-	0.75	-	-	0.66	2.22
20	2-Octanol (internal standard)	23.9–24.1	4.65	24.67	15.73	22.42	22.13	7.99
21	δ-Caprolactone	28.9–29.0	-	-	-	-	-	-
22	Nonanal	29.6–29.7	0.61	-	-	0.40	0.59	0.66
23	2(3H)-Furanones	21.1–37.3	-	-	-	-	-	-
24	Benzoic acid + isomers	33.1–49.9	-	-	-	-	-	-
25	Naphthalene	33.4	-	-	-	-	-	-
26	Octanoic acid	33.4–34.5	-	-	-	-	-	2.58
27	Octanoic acid, ethyl ester	34.4–34.5	-	-	-	-	-	-
28	δ-Octalactone	38.4–38.7	-	-	-	-	-	-
29	2-Undecanone	38.9–39.0	-	-	-	-	-	-
30	n-Decanoic acid	42.2–42.8	-	-	-	-	-	-
31	Decanoic acid, ethyl ester	43.4–43.5	-	-	-	-	-	-
32	δ-Decalactone	47.5–47.7	-	-	-	-	-	-
33	Dodecanoic acid, methyl ester	48.7–48.8	0.37	-	-	-	-	0.20
34	Dodecanoic acid	50.3–51.0	-	-	-	-	-	-

35	Dodecanoic acid, ethyl ester	51.4–51.5	-	-	-	-	-	-	-	-
36	γ-Dodecalactone	54.2–54.7	-	-	-	-	-	-	-	-
37	δ-Dodecalactone	55.6–55.8	-	-	-	-	-	-	-	-
38	Methyl tetradecanoate	56.3	-	-	-	-	-	-	-	-
39	Tetradecanoic acid	57.5–57.9	-	-	-	-	-	-	-	-
40	Tetradecanoic acid, ethyl ester	58.7–58.8	-	-	-	-	-	-	-	-
41	Pentadecanoic acid	61.0–61.1	-	-	-	-	-	-	-	-
42	δ-Tetradecalactone	62.9–63.1	-	-	-	-	-	-	-	-

Table A5. Hydrocarbon VOCs in VCOs produced by fermentation methods.

	Hydrocarbon	Retention Time	F1-0	F1-1	F1-2	F2-0	F2-1	F2-2	F3-0	F3-1	F3-2
1	Butane + isomers	4.2	-	-	-	-	-	-	-	-	-
2	Pentane + isomers	4.6–6.1	-	-	-	-	0.01	-	-	-	-
3	Hexane + isomers	4.7–12.6	1.80	3.01	1.25	0.15	0.97	0.36	-	-	1.99
4	Cyclopentane, methyl-	5.3	-	-	-	-	-	-	-	-	-
5	Benzene + isomers	5.7–47.2	5.56	0.02	0.02	-	0.04	-	-	-	-
6	Heptane/heptene + isomers	6.3–6.8	0.64	0.04	-	-	0.02	-	-	-	-
7	Toluene	9.11–9.3	-	-	-	-	0.13	-	-	-	0.06
8	1-Octene/octane + isomers	10.7–21.6	0.97	-	-	-	-	-	-	-	-
9	p-Xylene	15.6–17.1	-	-	-	-	-	-	-	-	-
10	Styrene	16.9	-	-	-	-	-	-	-	-	-
11	Undecane + isomers	21.7–23.8	0.87	0.02	0.04	0.39	-	0.15	-	-	-
12	Nonane + isomers	22.4–38.3	3.13	-	-	1.35	0.55	-	-	-	-
13	2-Octanol (Internal Standard)	24.0	3.23	1.72	2.24	3.8	5.76	2.12	1.91	2.53	1.75
14	Decane + isomers	25.1–37.5	-	-	-	0.79	0.03	-	-	-	-
15	D-Limonene	25.3	-	-	-	-	-	-	-	-	-
16	Dodecane + isomers	27.0–35.5	2.40	-	-	0.14	0.10	-	-	-	-
17	Naphthalene & derivatives	33.4–53.6	-	-	-	-	0.15	-	-	-	0.42
18	Dodecane	34.4	-	-	-	-	-	-	-	-	-
19	Hexadecane	34.6	-	-	-	0.08	0.08	0.02	-	-	-
20	Tetradecane + isomers	36.5–43.6	0.05	-	0.04	0.32	-	-	-	-	-
21	Eicosane	38.2–47.3	0.27	0.03	-	0.46	-	-	-	-	0.70
22	Heneicosane	47.1–49.2	-	-	-	-	0.18	0.03	-	-	0.16
Total %Normalized Area			15.69	3.12	1.35	3.68	2.26	0.56	0.00	0.00	3.33

Table A6. Hydrocarbon VOCs in VCOs produced by centrifuge methods.

	Hydrocarbon	Retention Time	C1-0	C1-1	C1-2	C2-0	C2-1	C2-2	C3-0	C3-1	C3-2
1	Butane + isomers	4.2	-	-	-	0.38	-	-	-	-	-
2	Pentane + isomers	4.6–6.1	-	-	-	-	0.05	0.38	-	-	-
3	Hexane + isomers	4.7–12.6	-	1.98	-	2.45	1.13	0.09	-	0.10	0.22
4	Cyclopentane, methyl-	5.3	-	-	-	-	-	-	-	-	-
5	Benzene + isomers	5.7–47.2	-	-	-	-	-	-	-	-	-
6	Heptane/heptene + isomers	6.3–6.8	0.76	-	-	1.14	-	-	-	-	-
7	Toluene	9.11–9.3	0.57	-	-	0.3	-	-	-	-	-
8	1-Octene/octane + isomers	10.7–21.6	-	-	-	-	-	-	-	-	-
9	p-Xylene	15.6–17.1	-	-	-	-	-	-	-	-	-
10	Styrene	16.9	-	-	-	-	-	-	-	-	-
11	Undecane + isomers	21.7–23.8	-	-	-	-	-	-	-	-	-
12	Nonane + isomers	22.4–38.3	0.83	-	-	0.31	-	-	0.59	-	-
13	2-Octanol (Internal Standard)	24.0	2.19	2.71	3.71	3.26	3.05	3.09	2.23	2.53	2.43
14	Decane + isomers	25.1–37.5	-	-	-	-	-	-	-	-	-
15	D-Limonene	25.3	-	-	-	-	-	-	-	-	-
16	Dodecane + isomers	27.0–35.5	-	-	-	-	-	-	-	-	-
17	Naphthalene & derivatives	33.4–53.6	-	-	-	-	-	-	-	-	-
18	Dodecane	34.4	-	-	-	-	-	-	-	-	-
19	Hexadecane	34.6	-	-	-	-	-	-	-	-	-
20	Tetradecane + isomers	36.5–43.6	-	-	-	-	-	-	-	-	-
21	Eicosane	38.2–47.3	0.48	-	-	0.4	-	-	0.33	-	-
22	Heneicosane	47.1–49.2	-	-	-	-	-	-	-	-	-
Total %Normalized Area			2.64	1.98	0.00	4.98	1.18	0.47	0.92	0.10	0.22

Table A7. Hydrocarbon VOCs detected in VCOs produced by expeller method.

	Hydrocarbon	Retention Time	E1-0	E1-1	E1-2	E2-0	E2-1	E2-2	E3-0	E3-1	E3-2
1	Butane + isomers	4.2	-	-	-	-	-	-	-	-	-
2	Pentane + isomers	4.6–6.1	-	-	-	-	-	0.04	-	-	-
3	Hexane + isomers	4.7–12.6	0.09	-	-	2.68	1.58	-	-	2.46	2.21
4	Cyclopentane, methyl-	5.3	-	-	-	-	-	-	-	-	-
5	Benzene + isomers	5.7–47.2	0.14	-	-	-	0.02	0.04	-	0.13	-
6	Heptane/heptene + isomers	6.3–6.8	0.58	-	-	-	-	-	0.91	0.39	-
7	Toluene	9.11–9.3	0.15	-	-	-	-	-	-	0.24	0.12
8	1-Octene/octane + isomers	10.7–21.6	-	-	-	-	-	-	-	-	0.05
9	p-Xylene	15.6–17.1	-	-	-	-	-	-	-	-	-
10	Styrene	16.9	-	-	-	-	-	-	-	0.13	-
11	Undecane + isomers	21.7–23.8	0.14	-	-	-	-	-	-	-	-
12	Nonane + isomers	22.4–38.3	0.19	-	-	-	-	-	-	-	-
13	2-Octanol (Internal Standard)	24.0	1.84	5.72	0.99	1.99	1.92	3.13	4.82	7.36	8.07
14	Decane + isomers	25.1–37.5	0.26	-	-	-	-	-	-	-	-
15	D-Limonene	25.3	-	-	-	-	-	-	-	-	-
16	Dodecane + isomers	27.0–35.5	-	-	-	-	-	-	-	-	-
17	Naphthalene & derivatives	33.4–53.6	-	-	-	1.39	-	1.51	-	-	-
18	Dodecane	34.4	-	-	-	-	-	-	-	-	-
19	Hexadecane	34.6	-	-	-	-	-	-	-	-	-
20	Tetradecane + isomers	36.5–43.6	0.12	-	-	-	-	-	-	-	-
21	Eicosane	38.2–47.3	-	-	-	-	-	-	-	-	-
22	Heneicosane	47.1–49.2	-	-	-	-	-	-	-	-	-
Total %Normalized Areas			1.67	0.00	0.00	4.07	1.60	1.59	0.91	3.35	2.38

Table A8. Hydrocarbon VOCs detected in RBDCOs.

	Hydrocarbon	Retention Time	RBD CO-1	RBD CO-2	RBD CO-3	RBD CO-4	RBD CO-5	RBD CO Old
1	Butane + isomers	4.2	0.05	-	-	0.13	-	2.94
2	Pentane + isomers	4.6–6.1	0.83	1.14	1.99	0.76	6.41	-
3	Hexane + isomers	4.7–12.6	1.12	9.49	16.43	13.23	3.28	3.17
4	Cyclopentane, methyl-	5.3	-	0.22	0.72	0.23	-	-
5	Benzene + isomers	5.7–47.2	0.05	-	-	-	-	0.91
6	Heptane/heptene + isomers	6.3–6.8	-	0.24	0.04	-	0.69	-
7	Toluene	9.11–9.3	0.88	0.72	1.32	0.93	0.28	3.39
8	1-Octene/octane + isomers	10.7–21.6	-	-	0.21	-	1.93	-
9	p-Xylene	15.6–17.1	0.40	-	-	-	-	1.05
10	Styrene	16.9	0.91	-	-	-	-	0.10
11	Undecane + isomers	21.7–23.8	0.09	-	-	-	0.47	1.40
12	Nonane + isomers	22.4–38.3	0.34	-	-	-	-	-
13	2-Octanol (Internal Standard)	24.0	4.65	24.67	15.73	22.42	22.13	7.99
14	Decane + isomers	25.1–37.5	0.16	-	-	-	-	-
15	D-Limonene	25.3	0.12	-	-	-	0.25	0.14
16	Dodecane + isomers	27.0–35.5	0.10	-	-	-	-	-
17	Naphthalene & derivatives	33.4–53.6	0.38	-	-	-	-	-
18	Dodecane	34.4	-	-	-	-	-	0.56
19	Hexadecane	34.6	-	-	-	-	-	-
20	Tetradecane + isomers	36.5–43.6	0.10	-	-	-	-	0.16
21	Eicosane	38.2–47.3	0.28	-	-	-	-	-
22	Heneicosane	47.1–49.2	-	-	-	-	-	-
Total %Normalized Areas			5.81	11.81	20.71	15.28	13.31	13.82

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