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Authors

Chiu, Elizabeth Y
Tran, Lillian N
Hunsaker, Haylee C
et al.

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Effects of Terpenes on the Emissions of Aerosols and Carbonyls from Vaping Δ8- and Δ10-Tetrahydrocannabinol (THC)

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Elizabeth Y. Chiu, Lillian N. Tran, Haylee C. Hunsaker, Sascha C. T. Nicklisch, and Tran B. Nguyen*

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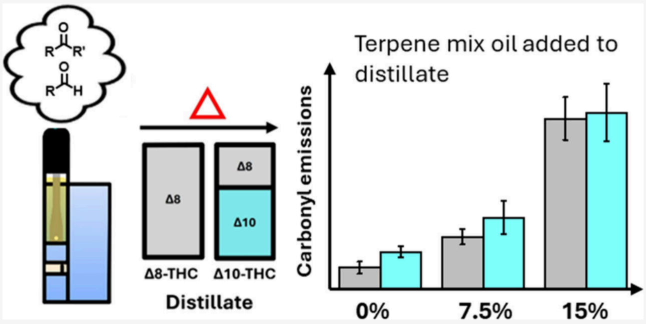
ABSTRACT: Cannabis electronic cigarette (CEC) products, used to "vape" or aerosolize cannabinoids and their mixtures, have proliferated in recent years and are emerging sources of indoor air pollution. This work characterizes the chemical composition and emissions of inhalable aerosol from CEC vaping of the popular synthetic cannabinoids Δ8- and Δ10-tetrahydrocannabinol (Δ8-THC and Δ10-THC, respectively). Commercial Δ8-THC and Δ10-THC distillates were found to have variable purity, with the Δ10-THC distillate comprising roughly one-third of Δ8-THC. The addition of a commercial terpene oil mixture (rich in d-limonene, *β*-caryophyllene, *β*-myrcene, and others) at 0%, 7.5%, and 15% by mass to the Δ8-THC and Δ10-THC distillates significantly increases emissions of carbonyls up to 9-fold, but not those of cannabinoid oxidation products. On average, 6 ± 1 mg of aerosol was produced per puff, which did not significantly vary with added terpenes. Molecular analysis confirmed that a majority of the carbonyl products originate from the chemical oxidation of terpenes during vaping. The most abundantly observed carbonyls were acetone, acetaldehyde, propionaldehyde, and terpene-derived carbonyls. We concluded that high terpene content in CEC products gives rise to more carbonyl emissions in the aerosol due to terpene oxidation, which has adverse implications for inhalation toxicology in an indoor environment.



KEYWORDS: *cannabis vaping, carbonyls, cannabinoids, terpenes, indoor air quality, electronic cigarettes*

1. INTRODUCTION

The 2018 Farm Bill legalized the growth, processing, marketing, and sale of hemp, which contains primarily cannabidiol (CBD) along with lower levels of other cannabinoids and <0.3% of Δ9-tetrahydrocannabinol (Δ9-THC).[1,2] This has led to the introduction of synthetic isomers of Δ9-THC from relatively simple syntheses involving CBD, such as Δ8-tetrahydrocannabinol (Δ8-THC) and Δ10-tetrahydrocannabinol (Δ10-THC), that retain psychoactive properties but have a legal status.[3] Thus, the Δ8 and Δ10 analogues of THC have emerged as popular choices for cannabis use, with Δ8-THC becoming the fastest-growing product in the hemp industry.[4] As "vaping", or aerosolizing liquids, oils, or solids with an electronic (e-) cigarette device, is rapidly emerging as a popular form of intake for cannabinoids among adolescents and young adults, it is increasingly common to find e-cigarette or vaping products that feature Δ8-THC and Δ10-THC as primary ingredients.[5−8] Cannabis electronic cigarettes (CECs), or cannabis vape devices, are similar to nicotine electronic cigarettes, except that they contain a ceramic coil that is optimized to aerosolize a range of different cannabis materials with variable viscosity, including plant/flower material, oily concentrates (e.g., distillate), waxy concentrates (e.g., resin or rosin), or "e-liquids" that contain cannabinoids at various concentrations (milligrams per milliliter) in solvents (e.g., terpenes, propylene glycol (PG)/vegetable glycerin (VG), or polyethylene glycol).[9]

CEC vaping has been shown to be an emerging source of indoor air pollution, which introduces particles, cannabinoids, volatile organic compounds (VOCs), and harmful or potentially harmful components (HPHCs) such as toxic carbonyls into indoor environments.[10−14] For example, cannabis vaping has been shown to emit more fine particles compared to cigarettes in general,[12] and more than cannabis smoking when compared at short distances (<1 m).[15] The chemical composition of cannabis vape emissions is under-characterized, which may be due to regulatory barriers and the

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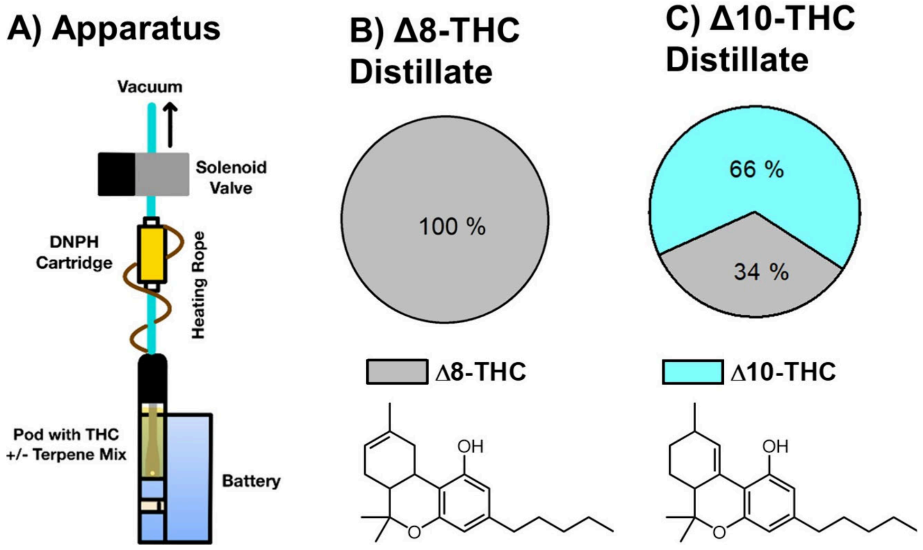


Figure 1. (A) Cannabinoid distillate vaping apparatus and GC/MS-quantified THC isomer distribution in (B) commercial Δ8-THC distillate and (C) commercial Δ10-THC distillate.

rapid evolution of the cannabinoid market.[16,17] In particular, the potential health and indoor air quality effects of vaping of the newly introduced synthetic products, Δ8-THC and Δ10-THC, are poorly understood.[18−21] To date, there have been no chemical assessments of Δ8-THC and Δ10-THC vape aerosol emissions in the literature. Furthermore, there are no currently established standards for testing or labeling for these products,[3,22] which prevents consumers and researchers from using the labeled content ingredients to extrapolate risks. The approximately 150 reported cases per year reported to the U.S. National Poison Center involving vaping Δ8-THC, Δ10-THC, and their acetylated analogues,[23] and the aforementioned impacts on indoor air quality, underscore the need to better characterize vape emissions from synthetic cannabinoids.

It has been recently observed that the consumer preference for cannabis vaping products in the United States and Canada is shifting from dry herb to cannabis oil or distillates.[24] Cannabis vape products that use cannabinoid distillates (often >90% purity) are often mixed with an oily mixture of cannabis-derived terpenes (monoterpenes $C_{10}H_{16}$ or sesquiterpenes $C_{15}H_{24}$) for added flavor or aroma and to reach the lower viscosity necessary to vape.[25] A decreased viscosity via the addition of terpenes may affect the aerosol emissions of the distillate. Terpenes are known to have high or moderate volatility. It was found that monoterpenes emitted from cannabis vaping are at comparable or higher concentration compared to cannabis smoking in an indoor environment and may be subject to thirdhand transport to other living spaces.[10] Terpenes are also chemically labile due to double bonds in their chemical structure[26] and known to react quickly[27] with the reactive oxygen species (ROS) that are produced during vaping.[28−30] Thus, the addition of terpenes to the cannabinoid distillates may impact the emission of oxidation products in aerosols such as carbonyls.[31]

There are many commercial cannabis-derived terpene mixtures, each advertising a different aroma that is achieved by a different compositional distribution of terpenes. Common major components are limonene, myrcene, and caryophyllene, alongside several other terpenes as minor components. To date, research on cannabis vaping has primarily focused on the dose transfer of terpenes and cannabinoids to the aerosol from both dabbing and vaping, specifically focusing on Δ9-THC.[32−36] The effects of added terpenes on the emission of air pollutants, such as aerosol particles and toxic carbonyls, from cannabinoid vapes are not well understood. Recently, Meehan-Atrash et al. reported that increasing the percent mass of β-myrcene in CEC vaping of Δ9-THC and cannabinol (CBN) decreased the extent of formation of some VOCs (isoprene, methylbutene, etc.) and isoprene-specific carbonyls (methacrolein, methyl vinyl ketone, etc.) and decreased the rate of degradation of the starting cannabinoid, though the effects on volatile compounds may be the opposite for dabbing.[32,37,38] Our work complements prior studies by characterizing aerosols generated from the CEC vaping of synthetic cannabinoids Δ8-THC and Δ10-THC, with and without the addition of a commercial mixture of terpene oil. Moreover, we examine a large suite of carbonyl emissions with semitargeted analytical techniques.

2. METHODS

2.1. Vape Oil Formulations and Analysis. Distillates of Δ8-THC and Δ10-THC (with a color and viscosity resembling honey) were obtained from commercial vendors without added terpenes and used without further purification. Δ8-THC distillate was obtained from The Hemp Collect (Portland, OR) and was advertised to contain 89.5% Δ8-THC (with minor contributions from cannabinol (0.67%), cannabichromene (0.18%), and cannabidiolic acid (0.03%)) according to the certificate of analysis. Δ10-THC distillate was obtained from Gilded Extracts (San Antonio, TX) and was advertised to contain 87.7% Δ10-THC (with minor contributions from Δ8-THC (0.352%), cannabinol (4.357%), and others) according to the certificate of analysis. An oily mixture of terpenes labeled "Watermelon Splash" was obtained from Abstrax Tech (Tustin, CA). The terpene mixture was advertised to contain d-limonene, β-caryophyllene, and β-myrcene as major components and a "candy" or "melon" flavor and aroma. The available documentation does not provide a specific mass distribution for the terpene mixture. The terpene mix was added to the THC distillates at 0%, 7.5%, and 15% (w/w) to mimic commercially available formulations and those studied in the scientific literature.[37,39]

The chemical composition of cannabinoids and terpenes in vape oils was analyzed by gas chromatography−mass spectrometry (GC-MS, Agilent 5890 GC with 5973 MSD) after dilution with ethyl acetate. Analytes were separated with an HP5-MS capillary column (30 m, 0.25 mm ID, 0.25 μm film, Agilent Technologies Inc., Santa Clara, CA) with the following oven program: 60 °C for 1.5 min, 10 °C/min for 24 min, and 300 °C for 3.5 min. Quantification was performed using certified reference standards of cannabinoids and terpenes: Δ8-THC in methanol and Δ10-THC in methanol (Cerilliant, Round Rock, TX) and Cannabis Terpene Mix A (20-component mixture in methanol) and Cannabis Terpene Mix B (14-component mixture in methanol) (Supelco TraceCERT, Bellefonte, PA).

2.2. Generation of Aerosol from a Cannabis e-Cigarette Device. The THC-derived vape oils were aerosolized or "vaped" with a representative cannabis electronic cigarette (CEC) device, the CCell Palm Pro with a battery capacity of 500 mAh (Shenzhen Smoore Technology Ltd., Shenzhen, China) (Figure 1A). CCell Kera cartridges (1.4 Ω, 1.0 mL, Shenzhen Smoore Technology Ltd.), hereafter termed the "pod", were selected based on their refillable ability and the specialized Zirconia ceramic coil designed to effectively vaporize viscous fluids. The device was vaped using the 3.6 V setting with the adjustable airflow collar kept closed for the most efficient aerosolization at the same vacuum flow rate and puff duration.

Cannabis vape users engage in longer and deeper puffs compared to nicotine e-cigarette users.[9,40] As a result, parameters were selected at the upper end of the conventional range for e-cigarette users in previous studies.[41] The device was activated with an average applied vacuum flow of 1.8 ± 0.1 L/min for a 4 s puff duration, corresponding to a puff volume of 121 ± 5 mL. The flow rate was measured by a primary flow calibrator (4000 Series Model 4043, TSI Inc., Shoreview, MN), and the puffing regimen was regulated by solenoid valves controlled by a time relay controller (PTR4-SP, Changzhou Xuchuang Info. Tech. Co., Changzhou, China). The puff frequency was 2 puffs/min. The aerosol mass (where the term aerosol includes both gas and particle phase emissions)[42,43] was determined by gravimetric analysis of the postvaped pod minus the prevaped pod using a calibrated Shimadzu microbalance.

2.3. Collection and Analysis of Carbonyls Using HPLC−HRMS. The methods for the collection and analyses of carbonyls used in this work have been described previously.[14,44,45] Briefly, gas and particle phase carbonyls produced from vaping Δ8-THC and Δ10-THC were collected onto 2,4-dinitrophenylhydrazine (DNPH) cartridges (350 mg of DNPH, Supelco Inc., Bellefonte, PA) for conversion into hydrazones. A total of 15 puffs were collected for each analysis. The number of puffs collected was informed by the residual peak of DNPH in mass spectrometry and from visual analysis of the reagent color change. Two milliliters of acetonitrile (LC-MS grade, Fisher Scientific Inc., Hampton, NH) was used to extract the DNPH and hydrazones.[44] The temperature of the pod, connector, and collection media was regulated to ∼35 °C with a fiberglass heating tape (Dwyer Omega, Michigan City, IN) to prevent aerosol condensation and clogging and to mimic body temperature during the transport of the aerosol from source to collection.

Carbonyl quantification was performed utilizing high-performance liquid chromatography−high-resolution mass spectrometry (HPLC−HRMS) using a Thermo Q-Exactive HF (High-field Orbitrap) at 45 000 $m/\Delta m$ mass resolving power at m/z 400. Hydrazones were separated with a Dionex Ultimate 3000 HPLC instrument using an Agilent Poroshell EC-C18 column (2.1 mm × 100 mm, 2.7 μm, 120 Å) with the following gradient program: 40% B (3.33 min), 50% B (14.6 min), 60% B (20 min), 100% B (32 min), and 40% B (37 min). Concentrations of formaldehyde, acetaldehyde, acetone, acrolein, propionaldehyde, methacrolein, hexaldehyde, benzaldehyde, butanone, butyraldehyde, crotonaldehyde, valeraldehyde, glyoxal, and methylglyoxal were quantified using commercially available certified reference standards (AccuStandard, New Haven, CT).[44] Acetic acid, glycolaldehyde, and hydroxyacetone standards were synthesized as described previously.[46] Concentrations of each carbonyl were normalized by the amount of aerosol collected on the cartridge, which was determined by weighing the cartridge before and after aerosol collection using a 51 g × 0.1 mg, 120 V analytical balance (Mettler Toledo, Columbus, OH).

$$\text{normalized carbonyl mass fraction} = \left(\frac{\mu\text{g}}{\text{mL}} \times \text{mL of extraction solvent}\right)\left(\frac{1}{\text{mg of aerosol collected}}\right) \quad (1)$$

Carbonyls for which analytical standards were unavailable were identified using an accurate mass but not quantified. Accurate mass assignments to molecular formulas were performed using MIDAS high-resolution mass spectrometry tools[47] within a mass accuracy tolerance of 3 ppm. These carbonyls are discussed in semiquantitative terms according to their HPLC peak areas. The quantified carbonyl values in this work should be considered to be a lower limit.

3. RESULTS AND DISCUSSION

3.1. Cannabinoid and Terpene Content from Distillate Mixtures. Composition analysis of the cannabinoid content of the distillates (Figure 1B,C) showed that the Δ8-THC distillate contains mostly Δ8-THC (98.7% THC isomer purity by mass) with a minor contribution from cannabidiol (CBD, 0.044%). This result is in rough agreement with the certificate of analysis. In contrast, we found that the Δ10-THC distillate purchased for this research is only two-thirds Δ10-THC with the remaining one-third of the composition comprising Δ8-THC (Figure 1C). This result is in disagreement with the product certificate of analysis, which stated an approximate 90% Δ10-THC content with <1% Δ8-THC. It is not clear if storage-related degradation occurred prior to purchase; however, THC has been reported to degrade via oxidation to cannabinol (CBN) rather than undergoing double bond migration to form other THC isomers.[48] As Δ10-THC is synthesized from other cannabinoids, unintended byproducts[37] and incomplete purification[4] are likely reasons for the high level of impurity of Δ8-THC in the Δ10-THC distillate.

The composition analysis of the terpene mix (Figure 2) used for this research revealed that the major component is d-limonene (56% by mass), with smaller contributions from β-caryophyllene (12%), β-myrcene (11%), linalool (7%), β-pinene (5%), 3-hexenol (5%), and α-pinene (4%). Although the product specifications do not indicate concentrations, it was determined that the major components listed on the

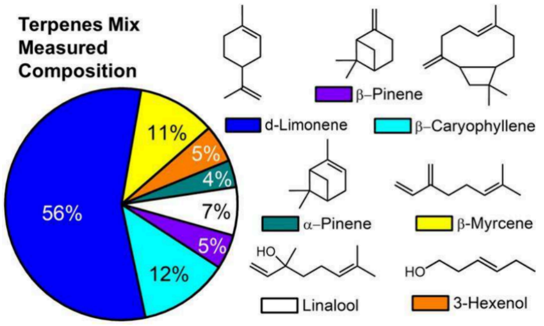


Figure 2. GC/MS-quantified terpene composition from the commercial Watermelon Splash terpene oil mix that was used for this study.

product page (d-limonene, *β*-caryophyllene, and *β*-myrcene) are indeed accurate after validation via GC-MS analysis.

3.2. Aerosol Mass and Carbonyl Mass Fractions That Were Quantified in the Aerosol. It was found that both Δ8-THC and Δ10-THC vape oils produce approximately 6 ± 1 mg/puff of aerosol, which is not significantly different within the uncertainty of the measurement regardless of cannabinoid isomer or added terpene content (Figure 3A). E-cigarette aerosol yields are reflective of both the vape material and the device construction and settings;[45,49] however, we can qualitatively assess that the aerosol yield from distillate vaping using a CEC in this work is comparable to the yields produced from vaping cannabinoid e-liquids using pod type e-cigarettes (50 mg/mL in PG/VG) and extracts of cannabidiol.[50,51] The uncertainties associated with triplicate gravimetric determinations of aerosol mass loss from the device were relatively large for distillate vaping (up to 50% individually), potentially due to the challenges associated with consistently aerosolizing the viscous distillates and their mixtures. Additionally, some vaporized mass may have condensed within the device or tubing upstream of the collection media, further contributing to the variability. It is possible that finer trends in aerosolization efficiency associated with added terpene content are obscured by the large uncertainties. Unfortunately, to the best of our knowledge, reports of aerosol mass yields in cannabinoid distillate vaping with added terpene content are not available in the literature for comparison.

Summed aerosol mass fractions of the quantified carbonyls (Figure 3B) had a smaller standard deviation compared to the quantification of aerosol mass, likely due to the fact that the variations in aerosol mass are accounted for in the mass fraction normalization (eq 1). It was found that an increase in the terpene concentration in the distillate mixture significantly increased the mass fractions of total carbonyls compared to each previous sample ($p < 0.05$, using one-way ANOVA). This trend was observed for both the Δ8-THC and Δ10-THC distillate samples. The 15% terpene sample showed the clearest increase in carbonyl production compared to the 7.5% and 0% terpene samples.

The trend of an increase in carbonyl mass fraction with an increase in terpene content (Figure 3) appears to be driven primarily by the production of acetone due to its relatively high level of production (Figure 4A). Table S1 shows the data for carbonyl mass fractions in each sample, and Table S2 shows one-way ANOVA test results for each carbonyl when comparing the 0% and 15% terpene samples. In the 15% terpene samples, acetone was observed with a mass fraction of 1000 ng/mg of aerosol, or 1 μg/mg. Assuming a density of approximately 1 g/mL for cannabis distillates (range of 0.96–1.05 g/mL),[52] the concentration of acetone itself is approximately 1 mg/mL of the aerosol, similar in concentration to purposefully added ingredients in vape liquids.[53] Propionaldehyde (Figure 4B), acetaldehyde (Figure 4C), and hexaldehyde (Figure 4D) were also observed as significant degradation products; their production also increased with an increase in terpene concentration. Because the pure distillate produced roughly 9-fold fewer carbonyls compared to the 15% terpene sample, this suggests that the degradation of terpenes during the vaping process is an important source of carbonyls during CEC vaping. The aerosol-normalized carbonyl emissions reported in this work (nanograms per milligram) can be converted into a parts-per-million (ppm, by mole) mixing ratio value in air using parameters of the puff (~6 mg of aerosol/puff, 1.8 L/min, 4 s puff) and the molar mass of the carbonyl. At the 15% terpene level, direct emissions of acetone and acetaldehyde equate to approximately 21 and 8 ppm, respectively. The recommended exposure limit (REL) for acetone is 250 ppm, according to the National Institute for Occupational Safety and Health (NIOSH). NIOSH does not list a REL for acetaldehyde, a suspected human carcinogen; however, its threshold limit set by the American Conference of Governmental Industrial Hygienists (ACGIH) is 25 ppm.[54] While these direct emissions from each puff do not violate exposure limits for acetone and acetaldehyde on their own, volatile carbonyls that are not ozone reactive (i.e., compounds

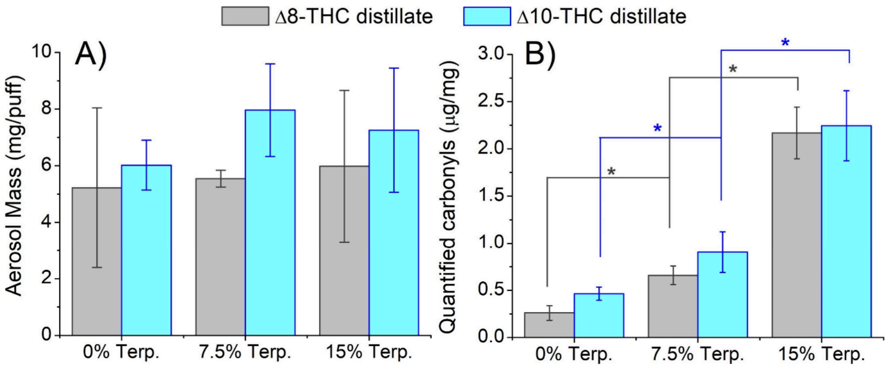


Figure 3. Aerosol mass (milligrams per puff) measurements for Δ8-THC (light gray) and Δ10-THC (light blue) vape oils with 0.0%, 7.5%, and 15.0% terpenes. Asterisks denote statistically significant differences ($p < 0.05$) using one-way ANOVA. Average aerosol masses among the three terpene mix concentrations are not statistically different.

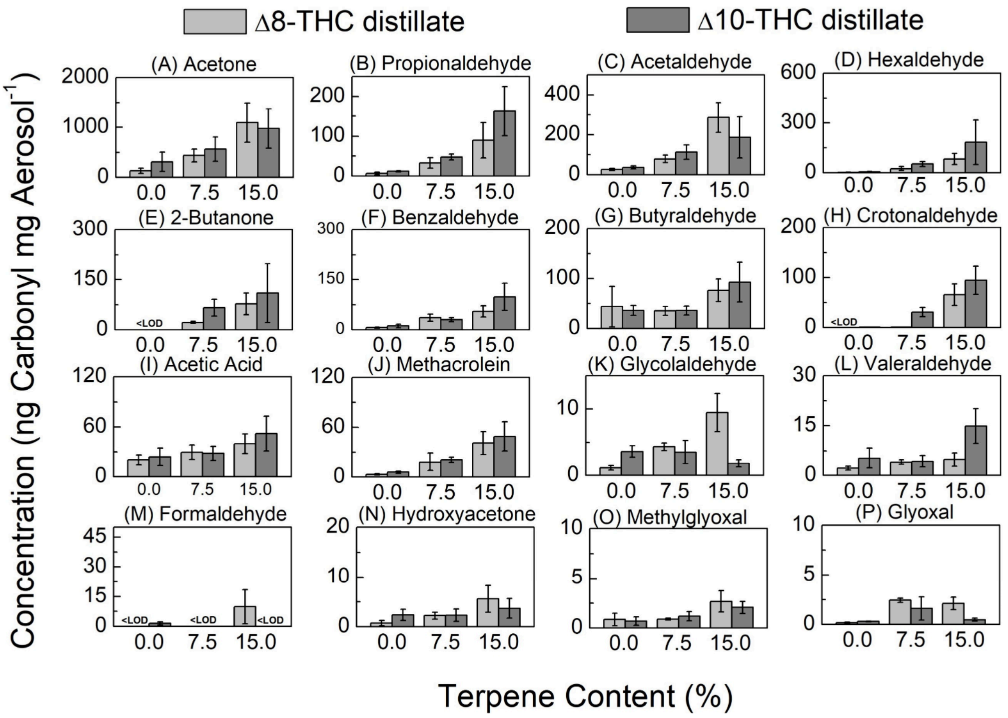


Figure 4. Concentrations of select carbonyls (nanograms per milligram of aerosol = micrograms per milliliter of aerosol assuming the density is 1 g/mL) that were quantified in Δ8-THC distillate samples (light gray) and Δ10-THC distillate samples (dark gray) at 0%, 7.5%, and 15% terpene levels.

with no C═C bonds, including acetone and acetaldehyde) may accumulate in indoor environments to increase exposure.[55] Furthermore, fugitive terpene emissions[10] from cannabinoid-containing vapes are highly ozone reactive and may be "aged" to further produce carbonyls and other toxicants in an indoor environment.[56] Interestingly, formaldehyde, which is usually a major carbonyl product for nicotine-containing e-cigarettes that contain propylene glycol (PG) and glycerol (or vegetable glycerin (VG)),[44,57,58] is found in very low mass fractions in cannabis vape distillates. The relatively high emission of acetone and the low emission of formaldehyde in the vaped Δ8-THC and Δ10-THC aerosol are consistent with the findings of Li et al.,[14] who studied Δ9-THC distillate with a third-generation e-cigarette device. It is possible that these results reflect the different chemical characteristics of vaping lower-viscosity e-liquid compared to higher-viscosity distillate or differences in coil types of the nicotine devices and the CEC devices. However, sampling differences cannot be ruled out as a source of the discrepancy as the DNPH-impregnated silica cartridges have not been validated for oily aerosol. Acrolein mass fractions in our study were either low or below the detection limit, consistent with the results reported by Meehan-Atrash et al.[32] and Li et al.[14] Acrolein is a well-studied dehydration product from glycerol (or VG).[59] VG is a widely used solvent for e-cigarette vaping, primarily with nicotine but also some modalities of cannabinoid vaping;[39,50] Li et al. showed that acrolein mass fractions are zero when VG is absent and increase with an increase in VG fraction in e-cigarette vaping.[44] It should be noted that acrolein is readily observable in the vaped aerosol of cannabinoids mixed with VG;[50] thus, its low mass fractions in this study and the aforementioned studies likely reflect the lack of VG (or other acrolein precursors) compared to other types of vaping.

The data presented here show that methacrolein mass fractions increase with a higher terpene concentration during CEC vaping. This differs from the report of lower HPHC emissions (specifically methacrolein, methyl vinyl ketone, and isoprene) with higher mass percentages of β-myrcene during CEC vaping of Δ9-THC vape oil.[37] The discrepancy could be due to the fact that this study examined a terpene mixture instead of a single terpene; thus, the chemical source of methacrolein in this work might be different compared to that from Meehan-Atrash et al.[37] It is also possible that the normalization with aerosol mass done in our work may reveal trends that are absent when the data are not normalized due to the high uncertainties related to aerosolization from viscous THC distillates (Figure 3A). Unfortunately, direct comparison of other products is not possible between this study and those in the literature.

Our results may also be compared to those of Tang et al.,[60] who studied the heating of light terpenes at a range of temperatures (25−500 °C) and various terpene mixtures (light, heavy, full, and complex) at 500 °C. Tang et al. also found that a full terpenoid mixtures, comparable to this work, produced the highest levels of acetone accompanied by lower levels of other carbonyls such as acetaldehyde, methacrolein, hexaldehyde, etc. However, our study reports lower mass fractions of formaldehyde and acrolein than those of Tang et al. Regardless, caution is advised for the intercomparison, as significant differences exist between our study and that of Tang et al. that may influence the temperature distribution, viscosity, and other factors impacting carbonyl emissions. For example, while both studies used DNPH cartridges to analyze carbonyls, our study uses a cannabis e-cigarette (CEC) with ceramic coil and a distillate/terpene mixture to simulate the user vaping experience, while Tang et al. heated terpenes directly on a Petri

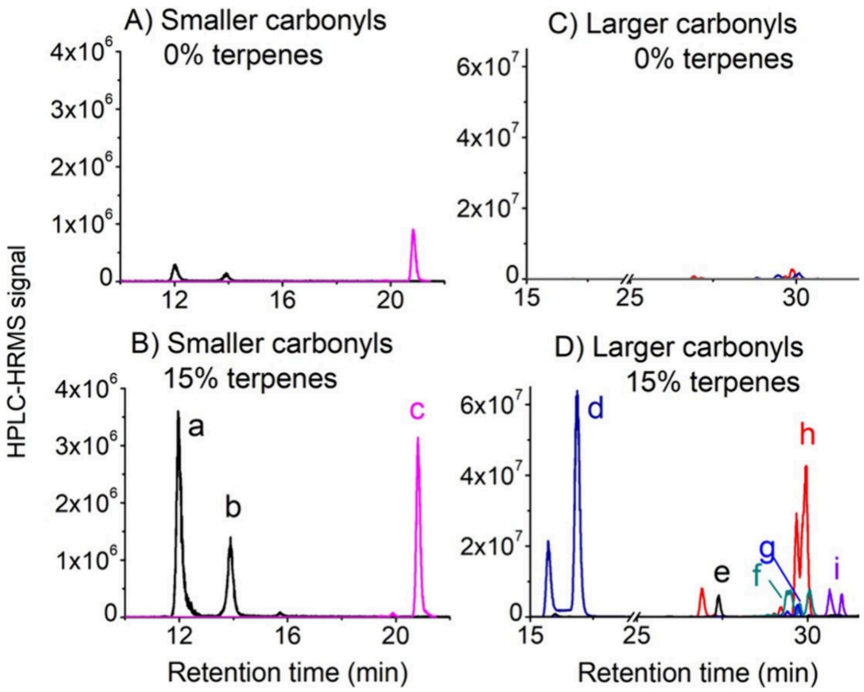


Figure 5. Extracted ion chromatograms for representative smaller (<C_6) and larger (C_9–C_{10}) carbonyl products, detected as their DNPH hydrazone negative ion ($m/z = M + C_6H_6N_4O_4 - H_2O - H$), from Δ8-THC distillate with no terpenes added (A, smaller; C, larger) and for Δ8-THC distillate with 15% terpenes by mass (B, smaller; D, larger). Chemical key, with assigned identities in parentheses: (a) C_3H_6O (acetone), (b) C_3H_6O (propionaldehyde), (c) C_5H_8O (3-methylbut-2-enal), (d) $C_{10}H_{12}O_2$ (two isomers), (e) $C_9H_{16}O_3$, (f) $C_{10}H_{16}O$ (three isomers), (g) $C_9H_{14}O$ (two isomers), (h) $C_9H_{16}O$ (three isomers), and (i) $C_{10}H_{16}O$ (two isomers).

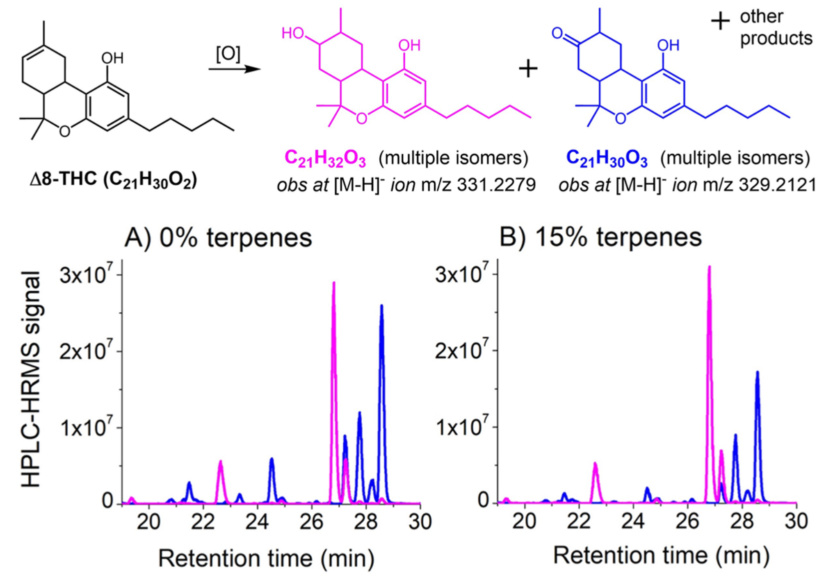


Figure 6. Extracted ion chromatograms for two representative oxidation products from Δ8-THC, $C_{21}H_{32}O_3$ (magenta) and $C_{21}H_{30}O_3$ (blue), from the following vaped aerosol samples: (A) Δ8-THC distillate (no terpenes added) and (B) Δ8-THC distillate + 15.0% terpenes by mass. One proposed chemical structure for each of the cannabinoid oxidation products is shown, although others may exist.

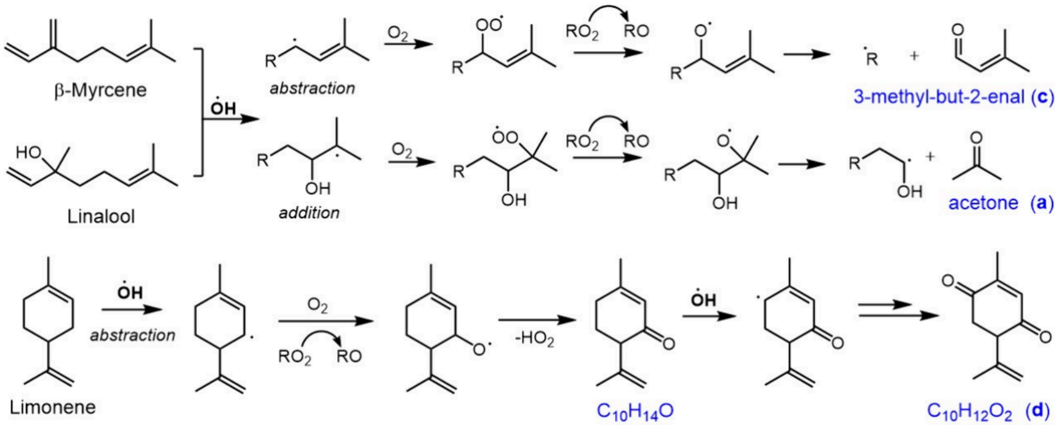
dish; these differences may contribute to the variability in carbonyl emissions observed across studies.

When comparing Δ10-THC vape oil and Δ8-THC vape oil, there are no significant differences in carbonyl formations, either pure or with varying terpene mix concentrations. Glycolaldehyde (Figure 4K) and valeraldehyde (Figure 4L) do show variations between the Δ8- and Δ10-THC samples at the 15% terpene level; however, they were observed with low mass fractions and thus are more susceptible to quantification inconsistencies. Generally, the trends of speciated carbonyls support the overall trend in quantified carbonyls, which increases with terpene mix percentage.

3.3. Oxidation Products of Cannabinoids and Terpenes.

Extracted ion chromatograms of major carbonyls observed in this work (Figure 5) visually demonstrate that mass fractions of carbonyls in the distillate vape aerosol increase substantially with the addition of terpene oil, regardless of whether those carbonyls were quantified with

Scheme 1. Proposed Reaction Mechanism for the Production of Carbonyl Compounds Associated with the Three Largest Peaks of Products Shown in Figure 5 (a, c, and d)[a]



[a] Proposed chemical mechanisms are based on textbook radical-initiated chemistry occurring in air.

standards or observed semiquantitatively (i.e., the peak areas correlate to concentration, but the data were not converted to concentration values). Most of the unquantified carbonyls are higher-molecular weight or complex carbonyls with the intact or nearly intact carbon backbone of the monoterpenes (C_9–C_{10}); the magnitude of their observed chromatographic peak areas was substantial. For example, the most abundant quantified carbonyl, acetone (Figure 4A), has a chromatographic peak area that is up to 20 times smaller compared with some unquantified carbonyls (Figure 5). It is understood that the range of electrospray ionization sensitivities for carbonyl-DNPH hydrazones generally vary by less than an order of magnitude.[61] Thus, the unquantified carbonyls may have yields exceeding that of acetone, suggesting that the quantification of targeted carbonyls in complex samples, such as cannabis vape oils and liquids, only tells a small fraction of the full story. Both the raw and processed carbonyl data collectively suggest that the added terpenes are being chemically transformed into carbonyls and that they are responsible for the majority of the carbonyl emissions.

In contrast, observations of the cannabinoid oxidation products show that the degradation of the parent cannabinoid either decreased slightly or remained the same with an increase in terpene content (Figure 6). While minor changes in the precursor THC content are challenging to quantify within uncertainty due to the fact that distillate contains nearly pure THC, we can monitor the peak areas of specific oxidation products of THC more precisely. Figure 6 shows that $C_{21}H_{32}O_3$ and $C_{21}H_{30}O_3$, two select oxidation products of Δ8-THC ($C_{21}H_{30}O_2$) with strong signals, had similar or smaller chromatographic peak areas in the samples with 0% and 15% added terpene content.

As these THC oxidation products were observed at the accurate mass of their deprotonated ions, their molecular formulas are accurately known and chemical structures (Figure 6, inset) may be proposed assuming established chemical mechanisms. These cannabinoid-derived ions have the exact molecular formula of Δ8-THC, but with the addition of O ($C_{21}H_{30}O_3$, signifying the addition of a carbonyl) or H_2O ($C_{21}H_{32}O_3$, signifying the addition of a hydroxyl), they most likely result from oxidation at the C═C bond in the ring structure of Δ8-THC. Multiple isomers exist for each oxidation product, suggesting that the location of the added carbonyl or hydroxyl moieties can vary along the structure of Δ8-THC, which is reasonable given that Δ8-THC has two allylic H abstraction sites adjacent to the endocyclic double bond, one tertiary benzyl H abstraction site, two alkenyl radical addition sites (one on each carbon of the alkene), two ring hydroxylation sites, and multiple chain oxidation sites.

Given the high chemical lability of terpenes[62] and the increasing carbonyl emissions (specifically those with a terpenoid backbone) with an increase in terpene content in the vape oil, it can be inferred that the terpenes in vape oil suffer substantial degradation during cannabis vaping; this was also observed by Meehan-Atrash et al.[32,37] As the levels of some cannabinoid oxidation products decreased with increasing terpene content, this may suggest that terpenes are shielding the cannabinoid from degradation, again in agreement with the works of Meehan-Atrash et al. With prior evidence that hydroxyl radicals (OH) are formed during the heated coil vaping process,[28−30,63] established mechanisms of radical-initiated chemical reactions in air[64−66] can be used to rationalize the formation of major carbonyl products from some abundant terpenoids in the terpene oil mixture: d-limonene, linalool, and *β*-myrcene (Figure 2). Scheme 1 shows proposed reaction mechanisms for the three most abundant carbonyl products according to chromatographic peak areas observed in this work (Figure 5): acetone, 3-methyl-but-2-enal, and terpene oxidation product $C_{10}H_{12}O_2$. Note that there is likely more than one reaction source for these products, and multiple isomers exist for $C_{10}H_{12}O_2$; thus, the reactions in Scheme 1 should be regarded as demonstrative only. Furthermore, after radical initiation via OH reaction or other processes, intermolecular reactions such as autoxidation may propagate radicals and lead to a different distribution of products; these autoxidation reactions are known to be favored at the higher temperatures present during vaping.[67,68] Future work may explore the role of autoxidation in producing hydroperoxides that are pro-oxidant.

β-Myrcene and linalool have similar chemical backbones, with linalool having one less degree of unsaturation and one more H_2O unit compared to *β*-myrcene at carbon 3. H abstraction at carbon 5 and OH addition at carbon 6 form the two most thermodynamically favorable alkyl radicals (an allylic radical and a tertiary radical, respectively).[69] Further alkylperoxy (RO_2) chemistry,[70] reducing to the alkoxy radical

(RO), may fragment[71] to acetone and 3-methyl-but-2-enal (Scheme 1, top). Acetone has also been quantified as a major OH oxidation product of myrcene previously, with yields of approximately 36%.[72] Product $C_{10}H_{12}O_2$ can be rationalized from d-limonene, β-myrcene, or potentially other monoterpenes (shown in Scheme 1 for only d-limonene) via allylic H abstraction and the aforementioned chemistry. The secondary alkoxy radical that is produced may lose H upon collision with oxygen ($-HO_2$) or other abundant species, forming the carbonyl; this is an established fate of secondary alkoxy radicals in air.[71] Oxidation at both allylic sites of d-limonene affords $C_{10}H_{12}O_2$. Not all coproducts of terpene oxidation, including the aforementioned formaldehyde,[72] are observed in high mass fractions in this work. This could signify a deviation of the vaping chemistry compared to more dilute chemistry in air but, again, could also represent the challenge of detecting all carbonyl products when viscous aerosol is produced from thick and dense distillates.

4. CONCLUSIONS

It was found that commercial sources of Δ8-THC and Δ10-THC had varying purity levels for the major cannabinoid, which may be different than the advertised information and may not necessarily be used for modeling, research, or risk assessment in the absence of chemical characterization. The terpene oil composition analysis matched the top three advertised components, although the specific composition again required chemical analysis. The addition of a commercial terpene oil mixture to distillates of Δ8-THC and Δ10-THC substantially increased the formation of harmful or potentially harmful carbonyls but had no discernible (within the uncertainty of this work) effects on aerosolization efficiency of the distillate mixture when vaped using a commercial CEC device. The data suggest that the degradation processes of terpenoid compounds in the mixture were chemically responsible for the majority of carbonyls in the aerosol. The most abundant quantified carbonyl was acetone; however, substantial chromatographic peaks for terpene-derived oxidation products were observed. Although these larger terpene-derived carbonyls were not quantified in this work, they are likely the major fraction of carbonyls in the aerosol. The value of total carbonyls reported from the cannabinoid vape mixtures in this study is a lower limit. The major carbonyl products can be rationalized from the known air oxidation chemistry of the major terpene components with OH radicals. The data suggest that oxidation of the cannabinoid occurred but did not increase due to the addition of terpenes and may decrease for some products. This work may be used to support an increasing body of knowledge on how cannabis vaping impacts health risks and indoor air quality. Overall, the popular practice of adding high levels of terpene oils to cannabinoid distillates may have adverse effects on HPHC formation, which may increase toxicity to the users and those in the immediate vicinity compared with vaping distillate alone.

While this work revealed important insights, there are a number of limitations to note. First, one terpene mixture was chosen for study due to the complexity of vape oil combinations and carbonyl product analyses. While terpene mixtures lend a high degree of relevance to real world exposures, terpene-specific origins of carbonyls cannot be elucidated. Studies of different terpenes or terpene mixtures yield different distributions of carbonyls. Future work may study individual terpenes in comparison with complex mixtures to supplement the understanding of terpene oxidation in cannabis vaping. Second, we were not able to vary the coil temperature or other device conditions in this study, as the vape oil composition was the main variable in the experimental design. One important next step is to investigate the role of temperature, vape regimen, or air flow on carbonyl emissions from cannabis vaping using one representative vape oil composition. Third, several larger carbonyls remained unquantified in this study despite having peak areas exceeding that of acetone, highlighting the need for further efforts in the quantification of terpene-derived carbonyls or nonspeciated total carbonyls. Together, these efforts will help clarify the chemical landscape of cannabis vaping aerosols and their potential health implications.

■ ASSOCIATED CONTENT

Data Availability Statement

The data supporting this article have been included as part of the Supporting Information.

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsestair.5c00169.

Tabulated aerosol mass yields and carbonyl mass fractions; carbonyl-specific ANOVA results (PDF)

■ AUTHOR INFORMATION

Corresponding Author

Tran B. Nguyen − *Department of Environmental Toxicology, University of California, Davis, Davis, California 95616, United States*; orcid.org/0000-0001-9206-4359; Email: tbn@ucdavis.edu

Authors

Elizabeth Y. Chiu − *Department of Environmental Toxicology, University of California, Davis, Davis, California 95616, United States*

Lillian N. Tran − *Department of Environmental Toxicology, University of California, Davis, Davis, California 95616, United States*; Present Address: L.N.T.: Environmental Toxicology Graduate Program, University of California, Riverside, Riverside, CA 92521; orcid.org/0009-0006-8882-5016

Haylee C. Hunsaker − *Department of Chemistry, University of California, Davis, Davis, California 95616, United States*

Sascha C. T. Nicklisch − *Department of Environmental Toxicology, University of California, Davis, Davis, California 95616, United States*; orcid.org/0000-0003-3120-6485

Complete contact information is available at: https://pubs.acs.org/10.1021/acsestair.5c00169

Author Contributions

E.Y.C. and T.B.N. designed the experiment. E.Y.C. developed the sampling protocols. E.Y.C., L.N.T., and H.C.H. developed the data analysis protocols. E.Y.C., L.N.T., and H.C.H. collected and analyzed the data. S.C.T.N. and T.B.N. secured the funding. E.Y.C., L.N.T., and T.B.N. wrote the manuscript. All authors reviewed the manuscript and made intellectual contributions to the project.

Notes

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