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Volatile aldehyde emissions from “sub-ohm” vaping devices

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Abstract

"Sub-ohm" atomizers with reduced resistance can deliver more power than conventional electronic cigarettes. Typical battery outputs are 100 watts or more. These devices are particularly popular among young users, and can be a significant source of volatile carbonyls in the indoor environment. Emissions from next-generation sub-ohm vaping products were characterized by determining e-liquid consumption and volatile aldehydes emissions for several combinations of popular high-power configurations. Tests explored the effect of dilution air flow (air vent opening), puffing volume, and coil assembly configuration. The mass of liquid consumed per puff increased as the puff volume increased from 50 to 100 mL, then remained relatively constant for larger puff volumes up to 500 mL. This is likely due to mass transfer limitations at the wick and coil assembly, which reduced the vaporization rate at higher puff volumes. Carbonyl emission rates were systematically evaluated using a 0.15 Ω dual coil atomizer as a function of the puffing volume and dilution air flow, adjusted by setting the air vents to either 100% (fully open), 50%, 25%, or 0% (closed). The highest formaldehyde emissions were observed for the lowest puff volume (50 mL) when the vents were closed (48 ng mg^{-1}), opened at 25% (39 ng mg^{-1}) and at 50% (32 ng mg^{-1}). By contrast, 50-mL puffs with 100% open vents, and puff volumes >100 mL for any vent aperture, generated formaldehyde yields of 20 ng mg^{-1} or lower, suggesting that a significant cooling effect resulted in limited carbonyl formation. Considering the effect of the coil resistance when operated at a voltage of 3.8 V, the amount of liquid evaporated per puff decreased as the resistance increased, in the order of 0.15 Ω > 0.25 Ω > 0.6 Ω, consistent with decreasing aerosol temperatures measured at the mouthpiece. Three different configurations of 0.15 Ω coils (dual, quadruple and octuple) were evaluated, observing significant variability. No clear trend was found between carbonyl emission rates and coil resistance or configuration, with

highest emissions corresponding to a 0.25 Ω dual coil atomizer. Carbonyl emission rates were compared with those determined using the same methodology for conventional e-cigarettes (lower power tank systems), observing overall lower yields for the sub-ohm devices.

1. Introduction

Electronic cigarettes continue to grow in popularity, as vaporizer technology evolves rapidly. Adoption of e-cigarettes as an alternative to conventional tobacco has been increasing steadily over the past decade around the world. The US Food and Drug Administration, which regulates these products, is particularly focused on preventing harmful exposures and youth initiation. While there are likely health benefits for long-time smokers who switch from combustion cigarettes to vaping, e-cigarettes may serve as a gateway for a lifetime of nicotine use for vulnerable adolescent and young first-time users (Berry et al., 2019). Despite marketing claims to the contrary, the aerosol generated by these devices contains harmful chemicals at levels that could produce short- and long-term health effects (Goniewicz et al., 2014; Kosmider et al., 2014; Logue et al., 2017; Ratajczak et al., 2018). For that reason, it is critical to investigate new vaping technologies and practices that may lead to exposures to harmful chemicals, and quantify these impacts.

A wide variety of e-cigarettes have become available over time. The first generation of devices ("ciga-likes") closely resembled combustion cigarettes in appearance. Equipped with a rechargeable battery, these e-cigarettes usually have disposable pre-filled cartridges along with built-in atomizers. The second generation or "vape-pens" also have rechargeable batteries, but the atomizer could be replaced and is separated from the e-liquid tank. Users refilled the tank with the liquid of choice, and apply variable voltage/variable power options. MODs (modified e-cigarettes), APVs (advanced personal vaporizers) or "next generation" vaping products appeared in the past few years. The terms MOD or APV apply to a variety of devices that go beyond the simple configuration of the "vape-pens". These devices provide more power and the ability to swap atomizers. Batteries offer outputs of 100 watts or more, large capacities (in the thousands of mAh), and in some cases control of the heating temperature. These upgraded batteries are often combined with reduced resistance "sub-ohm" atomizers (i.e., less than 1 Ω, compared with >2.0 Ω in vape-pens), in order to accelerate heat transfer and to evaporate large volumes of liquid. Sub-ohm atomizers are particularly popular among young users interested in practices like "cloud chasing" or "vape tricks", producing very large and dense exhaled aerosol clouds (Browne & Todd, 2018; Guy et al., 2018; Kim et al., 2016; Measham, O'Brien, & Turnbull, 2016; Pepper et al., 2017).

Although online guidelines to "sub-ohming" often warn users about potential safety issues associated with battery overheating, fire hazards and explosions, no information is usually given about the risks of inhaling the harmful chemicals that are formed (MistHub, 2015; Vaping360, 2018). At the same time, while most published studies have focused on "ciga-likes" and "vape-pens", vaping technology continues to evolve and larger, more powerful vaporizers present new challenges that have not been fully investigated. Sub-ohm devices have been studied in a few recent articles that focus on specific aspects, such as chemical emissions (El-Hellani et al., 2019; Haddad et al., 2018; Son et al., 2019; Vreeke et al., 2018), particulate matter (Protano et al., 2018) and e-liquid consumption (Korzun et al., 2018; Soulet et al., 2018). Volatile aldehydes in particular have been studied for some sub-ohm devices (Talih et al., 2017), but with puffing regimes that emulate those used in low-power e-cigarettes and combustion cigarettes. The inhaling method for sub-ohm devices, usually mentioned as direct lung inhalation (DLI), differs greatly from those regimens and implies larger puff volume and duration (Farsalinos & Gillman, 2018; Korzun et al., 2018). As aerosol is inhaled directly to the lungs, instead of the mouth, this method is chosen by vapers willing to generate massive "clouds" with sub-ohm devices. In a recent study, where human bronchial epithelial cells were exposed to volatile aldehydes (formaldehyde, acetaldehyde and acrolein) emitted during sub-ohm vaping, results have shown cytotoxicity, increased reactive oxygen species formation and dysregulated gene expression associated with biotransformation, inflammation and oxidative stress (Noël et al., 2020).

In the present study, we characterized emissions from next-generation sub-ohm vaping products that are mostly attractive to young users, a population particularly at risk for long-term effects derived from nicotine and tobacco consumption. We investigated e-liquid consumption and volatile aldehydes emissions for popular high-power configurations of atomizers. This information was used to quantify the potential exposures, and to compare with other types of e-cigarettes.

2. Materials and methods

2.1. E-cigarettes and e-liquid used in this study

A SMOK Stick V8 kit (Smoktech) was purchased from a retail e-cigarette store in Berkeley, CA, USA. The kit included a TFV8 Big Baby tank (24.5 mm diameter and 5 mL capacity), a constant voltage battery (3.8 V nominal value, voltage range 3.4 – 4.2 V) with a capacity of 3,000 mAh, two V8 Baby-M2 Core dual coils of 0.15 Ω and 0.25 Ω respectively (M2), and a USB cable for recharging the battery. Additional V8 Baby-X4 Core 0.15 Ω quadruple coils (X4), V8 Baby-T8 Core 0.15 Ω octuple coils (T8), and V8 Baby-Q2 0.6 Ω dual coils (Q2) were purchased online from an e-cigarette retailer in the USA. All coils used in this study were made of Kanthal® (FeCrAl alloy) wire. The different coil assemblies are specifically designed for sub-ohming operation. By contrast, typical coils used in conventional tank systems have a significantly higher resistance (e.g., 2.0 Ω and 2.6 Ω in those studied in Sleiman et al, 2016).

The e-liquid used in this study was Naked100 Euro Gold tobacco flavored (USA Vape Lab), purchased from the same retailer in Berkeley, CA. Its nicotine concentration was labeled as 6 mg mL^{-1}, with a vegetable glycerin (VG) to propylene glycol (PG) ratio of 65%-35%.

2.2. Experimental setup and sampling

A laboratory-made setup was used to generate consistent emissions from the sub-ohm device. Only stainless-steel Swagelok® connectors were used to collect samples. The e-cigarette was used according to the manufacturer's instructions, by actuating the start button and mechanically drawing emissions with a syringe. Once filled with liquid, the device was allowed to stay in vertical position for 30 minutes to ensure the cotton wicks were wet. The liquid in the tank was refilled after each experiment to avoid "dry puffing" conditions. Before operation, the sub-ohm device was cleaned with paper wipes to remove the excess of e-liquid after refilling the tank. Coils were re-used to maintain consistency in replicate determinations. In average, each coil was only used no more than four times, under what can be considered "initial" conditions. There was no buildup of residues on the surface of the coils after the measurements, which would indicate aging according to our previous experience (Sleiman et al., 2016).

Airflow system vents were used in the following positions: 100% (fully open), 50%, 25% and 0% (closed). The puffing protocol consisted of 4 s duration puffs and inter-puff periods of 30 s. Puff volumes of 50, 100, 250, 350 and 500 mL were generated in different experiments. Each puffing cycle included a total of 21 individual puffs performed over a 12 min period. An AirCon-2 air sampling pump (Gillian) was used to draw air from the device at preset flow rates, except for the 50-mL puff volume experiments, for which a peristaltic pump with #16 tubing (Cole-Parmer MasterFlex L/S) was used instead. In each experiment, samples were collected at flow rates of 12.5, 25, 62.5, 87.5 and 125 mL s^{-1}, for a puff volume of 50, 100, 250, 350 and 500 mL, respectively. The sub-ohm device was weighed on an analytical balance (Mettler) before each puffing test started, after puff #7, after puff #14, and at the end of the puffing cycle (puff # 21). The average mass change per puff was determined for each period. Commercially available 2,4-dinitrophenylhydrazine (DNPH)-impregnated silica gel cartridges (Waters Corp., PN WAT037500) were used to collect volatile carbonyls from 7 consecutive puffs (#8 to #14) in each experiment. Carbonyl emission factors were calculated as the ratio of the mass of each compound emitted to the mass of the e-liquid consumed per puff. Figure S1 (Supporting Information) illustrates the placement of the DNPH cartridge, connected directly to the mouthpiece. Details on the configuration of coils and wicks used in the study can be found in Figure S2 (Supporting Information).

2.3. Temperature measurements

The temperature profile during the operation of the sub-ohm device was measured by inserting a K-type thermocouple (Marlin Manufacturing Corporation) connected to a HOBO data logger (Onset Corporation). The thermocouple was carefully placed inside the mouthpiece downstream from the coils, avoiding contact with the walls or any other internal part of the devices (see Figure S1 in Supporting Information). Outcoming air temperature measurements were taken every second during the 12 minutes of operation, in the range 20 – 40 °C. Coil temperatures were not measured, and can reach much higher values during heating (e.g., 110 – 334 °C for a wet wick, according to Chen et al, 2018).

2.4. Chemical analysis

The analytical methods have been described previously (Cancelada et al, 2019). DNPH cartridges were extracted with 2 mL of carbonyl-free acetonitrile (Honeywell), and analyzed by High Performance Liquid Chromatography (HPLC) with UV detection (Agilent 1200), following the EPA TO-11 method (U.S.EPA, 1999). Analytes were identified based on the retention time of authentic standards of dinitrophenylhydrazine derivatives. A certified mixture of DNPH (2,4-dinitrophenylhydrazine) derivatives of carbonyls was obtained from Sigma-Aldrich, and was used as quantification standards for the HPLC analysis of formaldehyde, acetaldehyde, acrolein, acetone, propanal, crotonaldehyde, methacrolein, butanal, 2-butanone, benzaldehyde, valeraldehyde, *m*-tolualdehyde and hexaldehyde.

Calibration curves were generated for quantification of each analyte using those standards for thirteen carbonyls. Measurement results of blank samples were subtracted from the values obtained for the samples. Reported values are the average of duplicate determinations. Experimental uncertainties were estimated as the absolute difference of those duplicates.

3. Results and discussion

3.1.Effects of air vents and puffing volume

In an initial set of measurements of the SMOK Stick V8 device with a V8 Baby-M2 Core 0.15 Ω dual coil, we explored the effect of the fraction of airflow system vents open (in the range 0 to 100%), which affects the cooling and evaporation rates at the coils. A higher airflow allows for lower temperatures and it is usually the choice for sub-ohming users, as direct-to-lung inhalation is their preferred vaping method (Korzun et al., 2018). Combustion cigarettes and many electronic vaping devices are commonly used with mouth-to-lung inhalation, which also implies lower puff volumes. While most vaping test regimes for e-cigarettes use a puffing volume that barely exceeds 50 mL, sub-ohming requires a higher inhalation volume, as users attempt to generate very large clouds of exhaled aerosol (Farsalinos & Gillman, 2018). In order to address this distinctive feature, we also explored the effect of the puff volume in the range 50 to 500 mL. The results of these tests are presented in Figures 1 and 2. Figure 1 shows the average puff weight (i.e., the mass of e-liquid consumed per puff) for each experiment. Experimental errors between 15% and 27% in the determination of puff weight were determined from replicates performed in a sub-set of conditions. Except for the 500 mL puff volume, the fraction of the airflow system vents open did not have a major impact in puff weight. For puff volumes between 100 and 350 mL, puff weights were among 40 and 60 mg, with a slightly lower value for the 50 mL puff volume (31 mg in average). Still, these are elevated values compared to low-power devices; Gillman et al. (2016) report a range of 1.5 to 28 mg, while Soulet et al. (2018) report 5 to 14 mg per puff. The higher e-liquid consumption enables the generation of large clouds of aerosols. Figure 1 shows an increase in average puff weight when puff volume changed from 50 mL to 100 mL. Higher flow rates, i.e. higher puff volumes at a fixed puff duration, would increase solvent consumption (Korzun et al., 2018). However, puff volumes greater than 100 mL gave similar puff weights with the airflow vents open at 50% and 100%. A 5-fold increase in puff volume did not affect the amount of e-liquid that was consumed. This value depends on the quantity of liquid in the vicinity of the coil, that is, in the cotton wick that surrounds it. The speed at which this liquid was renewed in the wick, by capillarity, limited the vaporization rate. No matter how high the air flow rate, the quantity of liquid being vaporized was similar.

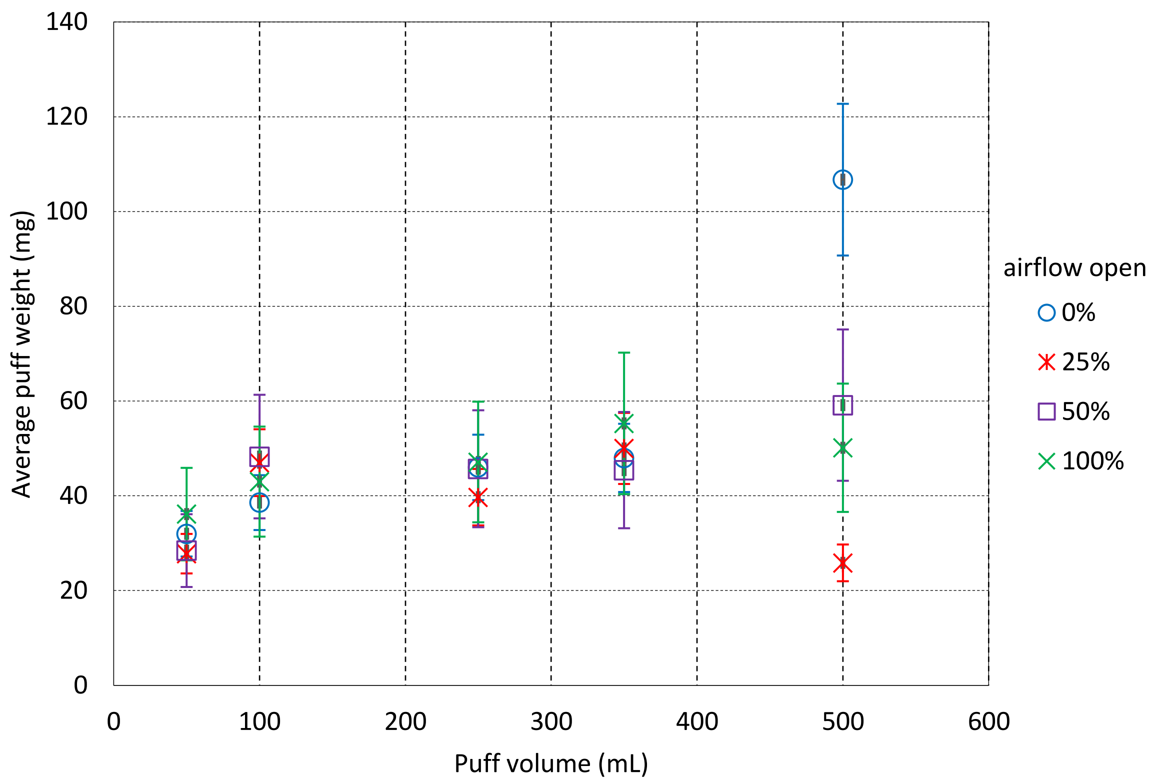

Figure 1. Average puff weight (puffs #8 to #14) versus puff volume for SMOK Stick V8 device with a V8 Baby-M2 Core 0.15 Ω dual coil.

Figure 2 shows the yield in ng per mg of e-liquid consumed for formaldehyde, one of the most prominent by-products. The highest formaldehyde emissions were observed for the lowest puff volume (50 mL): 48 ng mg^{-1}, 39 ng mg^{-1} and 32 ng mg^{-1} for 0%, 25% and 50% of airflow system vents open, respectively. Puff volumes of 100 mL or more generated lower formaldehyde yields (around 20 ng mg^{-1} or less). Although a higher puff volume could potentially imply a higher exposure to harmful compounds, these results show that the increased flow rate can also reduce the degree of decomposition of the e-liquid components, mainly VG and PG, that leads to volatile carbonyls formation (Jensen, Strongin, & Peyton, 2017; Salamanca et al., 2017). At elevated flow rate values, the cooling effect on the coils may be significant, which is also shown by the fact that, at 50 mL puff volume, the formaldehyde yield also falls under 20 ng mg^{-1} when the airflow has no restriction (100% of vents open). Results corresponding to other carbonyls are presented in Table S1 (Supporting Information). Overall, similar trends as those described for formaldehyde were observed for several other carbonyls.

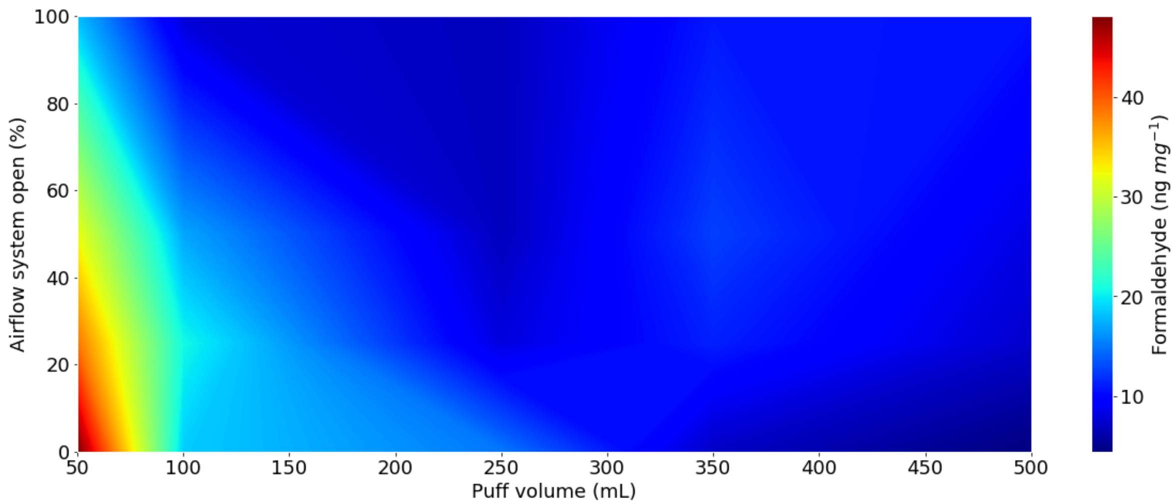

Figure 2. Effect of the fraction of airflow system vents open (y axis) and puff volume (x axis) on the yield of formaldehyde, expressed in ng of formaldehyde per mg of e-liquid consumed (V8 Baby-M2 Core 0.15 Ω dual coil).

3.2 Effects of different resistance and coil configurations

In subsequent tests, the effect of using different coil assemblies was studied for 50 mL puff volume and vent positions corresponding to 50% and 25% of total airflow. Figure 3 illustrates the puff weight determined in each case as the average of duplicate determinations, showing very similar results for both vent settings. The experimental error corresponds to the absolute difference between each pair of duplicate measurements. Experiments performed with a resistance of 0.15 Ω using three different coil configurations showed that the system with 4 coils was able to emit a larger mass per puff, compared with systems with 2 and 8 coils. Using the same type of device, Talih et al. (2017) established that, at constant power, an increase in coil surface area resulted in a decrease in e-liquid consumption, as the temperature in the coil is proportional to the power input per unit area. We verified this relationship with the results for the 0.15 Ω V8 Baby-X4 quadruple and 0.15 Ω V8 Baby-T8 octuple coils. However, the 0.15 Ω dual coil did not respond to the same trend, even if it had a lower coil surface area. Other factors might be affecting e-liquid vaporization in this case; for example, the smaller surface in the dual coil was surrounded by less liquid available for evaporation. After it was evaporated, the remaining heat was used to increase the temperature of the coil and the surrounding aerosol. Since these experiments were not able to establish the temperature at the coil surface, we used the temperature of the aerosol recorded at the mouthpiece as a proxy for coil temperature. The temperature measured in the mouthpiece after 12 minutes was the highest for the 0.15 Ω dual coil, followed by the 0.15 Ω quadruple and octuple coils, as shown in Figure 4-A.

When experiments were carried out at higher resistances, we observed that the amount of liquid evaporated per puff decreased as the resistance increased, in the order 0.15 Ω > 0.25 Ω > 0.6 Ω (Figure 3). As expected, this result confirmed that the temperature achieved at the coil surface is proportional to the delivered power at a constant voltage of 3.8 V. All other parameters being equal, the estimated delivered power was 96 W, 58 W and 24 W for the 0.15 Ω, 0.25 Ω and 0.6 Ω dual coils, respectively. Figure 4-A also shows the decrease in temperature when increasing resistance in the coil.

Figure 3 compares the data from our study with the average puff weight for two conventional tank-type devices from our group's previous work (Sleiman et al., 2016). These low-power devices were the eGO CE4 (version 2) with single coil and resistance of 2.6 Ω, and the Kangertech Aerotank Mini with dual coil and resistance of 2.0 Ω. Both devices were used at 3.8 V, with a delivered power estimated around 6 W. Average puff weight for these devices was between 5 and 8 mg, as it is shown in Figure 3 (shadowed area). Only the highest resistance used in our study (0.6 Ω) presented a similar behavior. Figure 4-B shows that the mouthpiece temperature in these low-power devices was in the same range than the results found for the sub-ohm device.

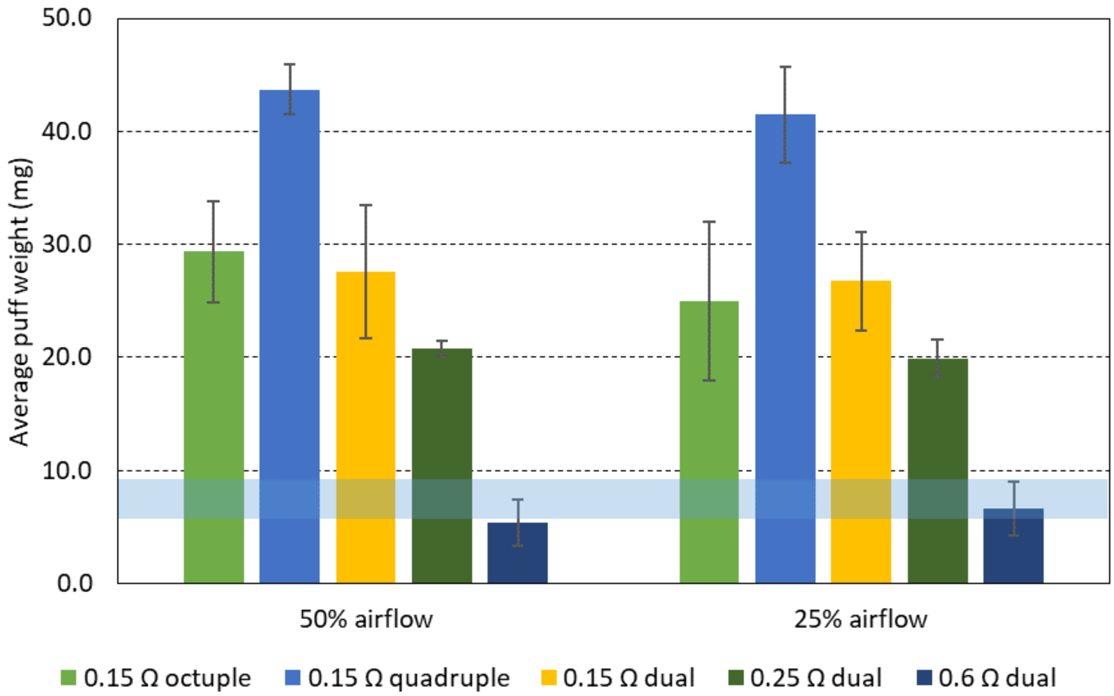

Figure 3. Effect of the coil resistance and configuration on average puff weight (puff volume = 50 mL). Shadowed area corresponds to average puff weights for low-power e-cigarettes from Sleiman et al. (2016).

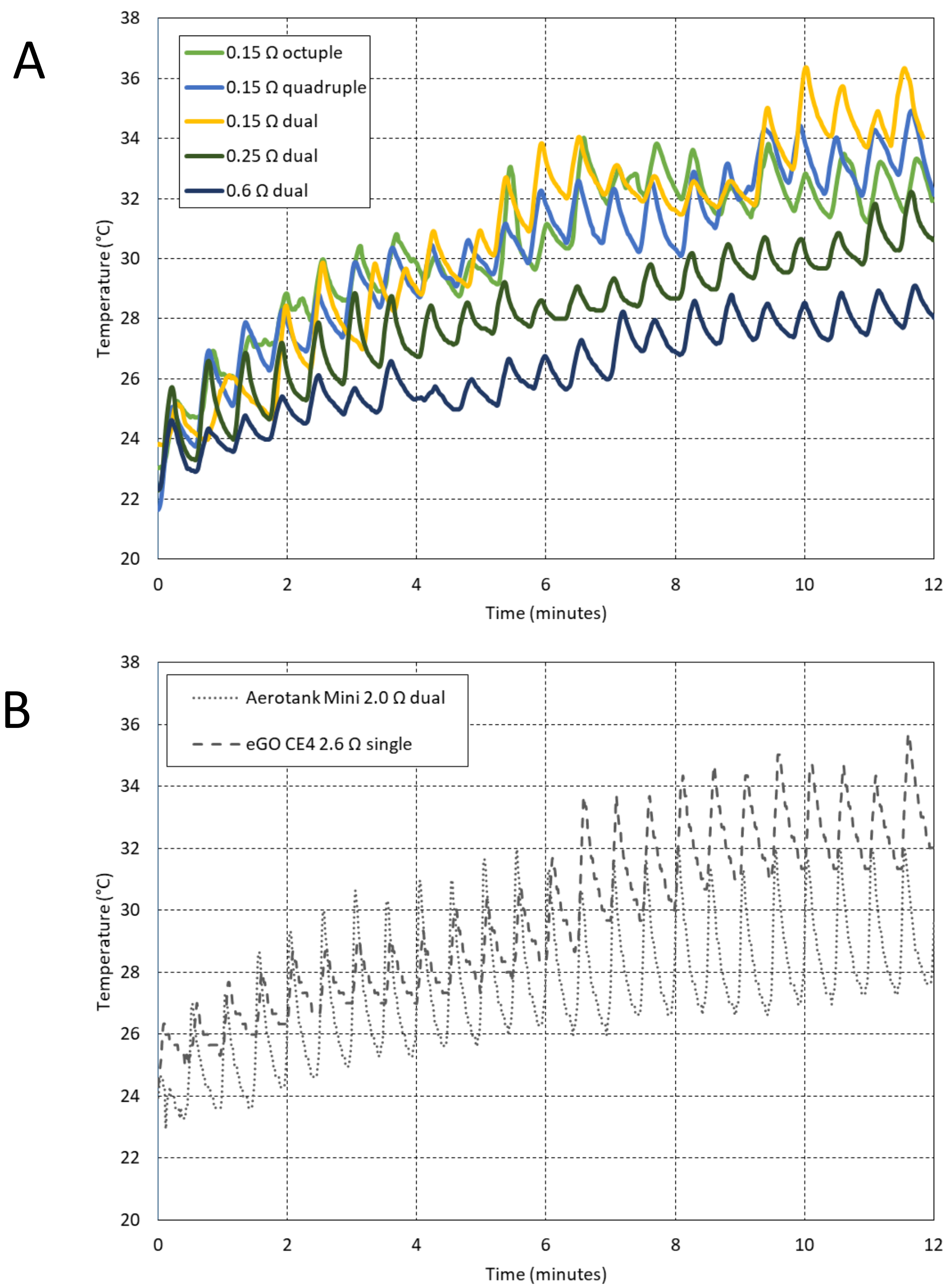

Figure 4. Mouthpiece temperature profiles during puffing (A) using the SMOK Stick V8 device with different coil resistances and configurations; (B) using two low-power tank-style devices, described in Sleiman et al. (2016).

For each of these tests performed with five different coils using 50 mL puff volume and vent positions corresponding to 50% and 25% of total airflow, we analyzed the yields of volatile carbonyls. The emission factors in ng of compound per mg of e-liquid consumed are reported in Figure 5, corresponding to the average of two replicates. Experimental error represents the absolute difference between each pair of duplicate determinations. Formaldehyde was the most prominent byproduct, followed by acetaldehyde, consistent with previous findings on e-cigarette emissions (Goniewicz et al., 2014; Kosmider et al., 2014; Logue et al., 2017; Noël et al., 2020). Other analytes found in all tests were acrolein, methacrolein and hexaldehyde. A summary of the results corresponding to twelve carbonyls (valeraldehyde was not detected in these experiments) is presented in Table S2 (Supporting Information). No clear trend was found among coil resistance or configuration in this case. Surprisingly, the 0.25 Ω dual coil showed emission factors that were one order of magnitude higher than the rest of the atomizers, while those for 0.15 Ω and 0.6 Ω coils were consistently similar. As it was pointed out by Jensen et al. (2017), multiple coils can be more efficient at heat dissipation, minimizing solvent degradation. Therefore, a lower carbonyl production was expected from the quadruple and octuple coils than the dual coils. The higher emission factors for the octuple coil compared to the quadruple coil are consistent with the results shown by Talih et al. (2017) using the same type of device. In that study, authors have shown that, counterintuitively, high power devices do not necessarily produce high volatile carbonyls emissions. In fact, the degree of by-product generation may be affected by device design, coil construction and coil materials, all of which influence the coil resistance and temperature. The increased variability shown in Figure 5 for both the 0.15 Ω and 0.25 Ω dual coils (triplicate determinations were performed in both cases), indicates a major influence of variations in coil construction in aldehydes yields, as it was noted by Jensen et al. (2017).

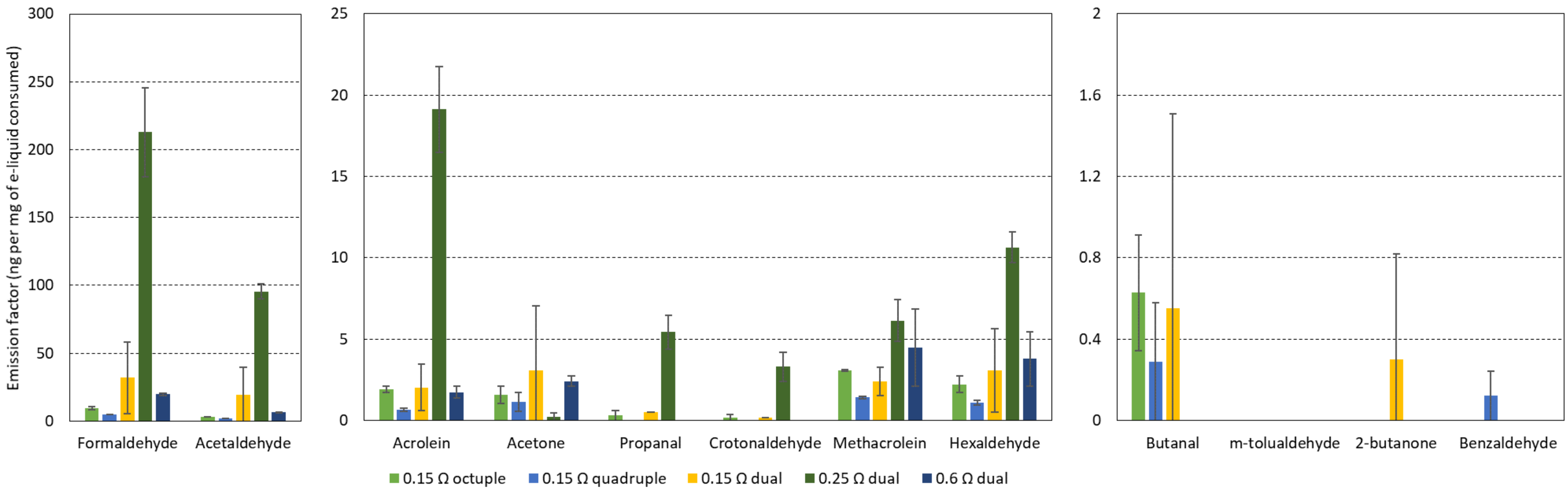

Figure 5. Emission factors for volatile carbonyls (puff volume = 50 mL, 50% airflow system vents open).

4. Conclusions

In order to assess the potential impact on users and on indoor concentrations of volatile aldehydes, we compared the results from this study with those produced by our group using low-power vape-pen devices (Sleiman et al., 2016). Table 1 shows the values determined here for e-liquid consumption and volatile aldehydes intake for the atomizers studied with the SMOK Stick V8 device and Naked100 Euro Gold e-liquid (nicotine concentration 6 mg mL^{-1}). The last two columns show results from two low-power e-cigarettes used in our previous study, which used an eGO CE4 version 2 (single coil, 2.6 Ω, operated at 3.8 V) and Kangertech Aerotank Mini (dual coil, 2.0 Ω, operated at 3.8 V), with Apollo Classic Tobacco e-liquid (nicotine concentration 20.4 mg mL^{-1}). In all cases, emissions were analyzed using similar methods and instrumentation. Table 1 shows that while the estimated nicotine content per puff remained in the same order of magnitude for all devices, the difference in volatile aldehyde emissions was significant. The sub-ohming practice, due to higher puff volumes and solvent consumption, requires low nicotine concentration e-liquids to avoid a harsh taste, thus using more liquid to reach the desired blood levels (Etter, 2016). However, this compensatory behavior did not necessarily translate into higher exposure to harmful compounds. Values for formaldehyde, acetaldehyde and acrolein intake rate, calculated as the average content per puff, are presented in Table 1. Similar values for formaldehyde and acetaldehyde from a 0.15 Ω and 0.5 Ω sub-ohming device operated at 3.8 V were reported in a recent study (Noël et al., 2020). Our results were slightly lower than those obtained with a “mod” device in another recent study (Son et al, 2020a). Differences between the sub-ohm results presented here and vape-pen devices studied by our group several years ago are between one and two orders of magnitude, and may reflect in part recent improvements in e-cigarette technologies. Moreover, if users' intake values are expressed per mg of nicotine, these differences are even more significant. The expected impact of such relatively low aldehyde emission rates on indoor air quality are expected to be minor, but not negligible, particularly in settings where several users are present (Son et al, 2020b).

Acknowledgement

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Table 1. Solvent consumption and volatile aldehydes intake for the SMOK V8 Stick device and low-power e-cigarettes (eGO stands for eGO CE4 version 2 and Aero, for Kangertech Aerotank Mini); airflow system vents 50% open.

Atomizer	**This study**									**Sleiman et al. (2016)**	
	SMOK V8 Stick									**eGO**	**Aero**
	T8	**X4**	**M2**						**Q2**		
Resistance (Ω)	0.15	0.15	0.15					0.25	0.6	2.6	2.0
Number of coils	8	4	2					2	2	1	2
Puff volume (mL)	50	50	50	100	250	350	500	50	50	50	50
E-liquid consumed per puff (mg)	29.3	43.7	27.6	45.6	44.1	45.7	54.7	20.8	5.4	5.1	8.2
Estimated nicotine content (µg per puff)	0.15	0.22	0.14	0.24	0.23	0.24	0.28	0.11	0.03	0.09	0.14
Formaldehyde emission rate											
µg per puff	0.35	0.23	1.0	0.82	0.31	0.57	0.49	4.4	0.12	30	15
µg per mg of nicotine	2.3	1.0	7.3	3.5	1.4	2.4	1.7	41.3	4.3	337	106
Acetaldehyde emission rate											
µg per puff	0.11	0.09	0.64	0.42	0.14	0.12	0.20	2.0	0.04	5.2	2.2
µg per mg of nicotine	0.2	0.4	0.4	1.8	0.6	0.5	0.7	0.2	0.04	58	15
Acrolein emission rate											
µg per puff	0.07	0.03	0.07	0.07	0.03	0.04	0.05	0.4	0.01	4.6	0.7
µg per mg of nicotine	0.5	0.3	0.1	0.3	0.1	0.2	0.2	0.1	0.03	51	4.5

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