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**DETECTION OF TOXIC ALDEHYDES IN AEROSOLS OF
ELECTRONIC CIGARETTES**

By

Ellie Bess Reed

B.S. University of Louisville, 2022

A Thesis

Submitted to the faculty of the

University of Louisville J.B. Speed School of Engineering

In fulfillment of the requirements

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In Chemical Engineering

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DETECTION OF TOXIC ALDEHYDES IN AEROSOLS OF
ELECTRONIC CIGARETTES

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A Thesis Approved on

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ABSTRACT

Over the past decade, use of electronic cigarettes (e-cigarettes) has increased in the younger generations of the United States. With the broad range of flavors and devices distributed on the market, American youth are prime marketing targets for the e-cigarette industry. To create a more regulated market, research of this thesis has been conducted on newer generations of e-cigarette “MOD” devices to examine how e-cigarette battery power output and coil temperature, concentrations of propylene glycol and vegetable glycerin, added flavorings (strawberry, mango, and menthol), and the presence of nicotine affect generation of aerosol particles and aldehydes in aerosols emitted by later versions of e-cigarettes. A 50 mL syringe and Tedlar bags were used to standardize and collect the vapor produced by the e-cigarette. Gas chromatography-mass spectrometry (GC-MS) was utilized to analyze the amounts and concentrations of aldehydes in the collected e-cigarette aerosols. Various tests were run using different e-liquid flavors, nicotine concentrations, and power-temperature settings. Tests were conducted on two separate heating coils with resistances of 1.4 Ω and 0.6 Ω . The results indicate that an increase in power and a decrease in resistance of the heating coil generated more aldehydes. Given the wide variety of e-cigarette device structures, flavor types, and nicotine concentrations on the market, it is likely that e-cigarettes produce broad ranges of toxic aldehydes, like formaldehyde, acrolein, glyoxal, and methylglyoxal, that react with proteins linked to respiratory diseases such as cardiovascular disease, chronic obstructive pulmonary disease (COPD), and early onset cancers.

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CHAPTER 1. INTRODUCTION

E-cigarettes are battery-powered devices that are used to vaporize e-liquids. The battery power of these devices can be controlled by wattage and heating coil resistance changes which dictate the coil temperatures of the atomizer and contribute to the aerosol size and evaporation rates delivered to the consumer's lungs [1]. The wire behavior according to the supplied power could be separated into three regimes: under-heating (insufficient power to generate an aerosol), optimal vaporization characterized by a linear trend (vaporization of the e-liquid proportional to the supplied energy) and over-heating (dry-burn occurs). Using a controllable and repeatable battery power supply, the reproducibility of the quantity of vaporized e-liquid can be verified for each of the series of 20 puffs programmed for all the atomizers. As the e-cigarette industry continues to develop lower (sub-ohm) resistant coils and higher battery-power outputs, the number of aerosol particles will continue to increase.

1.1 Literature Review

Newer generations of e-cigarette devices use a battery power output of 9 Watts and above. The emission of these devices indicates risks of use in both American adult and youth populations. Lower molecular weight organic compounds in e-cigarette aerosols are proven to be the most toxic constituents of tobacco products and tobacco smoke [2]. To alleviate the intake of e-cigarette aerosols in the American population and

mitigate the health risk, it is necessary to detect and accurately measure the amounts of aerosol and carbonyl compounds produced by the devices and newer generations of e-cigarettes that will be added to the market. The emission of these compounds has raised the concern that these devices could contribute to early pulmonary diseases and cancers that can contribute to active and secondary exposure to the device [3]. Low molecular carbonyl compounds (acetaldehyde, acetone, acrolein, crotonaldehyde, formaldehyde, methylethylketone) are on the list of chemicals and chemical compounds identified by the FDA as harmful and potentially harmful constituents (HPHCs) in tobacco products and tobacco smoke. All belong to the respiratory toxicant group. Acetaldehyde, crotonaldehyde and formaldehyde are carcinogens. In addition, acrolein is a cardiovascular toxicant, and acetaldehyde has addictive properties. Hence, accurate measurements of these compounds' concentration in the aerosol, and consequently the estimation of the electronic cigarettes impact on health are important for users, especially adolescents that are fascinated and experimental with these devices [4].

Flavors increase product attractiveness among all types of users, that is, among youth and adults and among current smokers, dual users, exclusive vapers, as well as non-users. For smokers, switching to e-cigarettes may be beneficial, as e-cigarette use is considered less harmful than regular cigarette smoking. In line with this, the use and marketing of e-liquid flavors that are appealing to smokers may contribute to public health benefits. However, flavors may also stimulate vaping among non-users, young people [5]. This is concerning, as e-cigarettes are not safe. That is, chemicals in e-cigarette emissions (tobacco specific nitrosamines, trace metal nanoparticles, aldehydes, and other flavorings) can be toxic and thus harmful to consumers' health. In addition, e-

cigarettes may facilitate smoking initiation among nonsmokers. Consequently, e-liquid flavors are considered an important target in tobacco control to decrease e-cigarette attractiveness and exposure to potentially toxic emissions [6]. Using this data, the three flavor profiles that were selected for this study were strawberry, mango, and menthol.

The newer generations of e-liquid products have added salts. E-liquid salts use benzoic acid to increase the amount of nicotine, increasing the nicotine content from a standard 3 mg/ml to a staggering 35 mg/ml [7]. Even though the salts product will appeal more to the adult population, allowing the consumer to smoke less e-liquid while still receiving the same nicotine fix as they would with a standard tobacco cigarette, the product is still available to the youth population. More regulations and consumer guidance are necessary to create a safer product.

To regulate the newer generations of e-cigarettes and e-liquids, advanced research will be required to ensure the health and safety of the American consumer market. In addition to increased power output and lower coil resistance, new brands of e-liquids and additives are posing a threat on the e-cigarette market. The use of flavorings in e-cigarette fluids has become a central focus for those marketing e-cigarettes and for those demanding regulatory control [8]. An estimated 4.1 million high school students and 1.2 million middle school students currently use e-cigarettes, an estimated 1.6 million students reported frequent use of e-cigarettes, an estimated 970,000 students use e-cigarettes daily, and an estimated 2.4 million exclusive e-cigarette users use flavored e-cigarettes. The data also would suggest that among these exclusive e-cigarette users, an estimated 1.6 million high school and middle school students use fruit-flavored e-

cigarettes, an estimated 1.2 million use menthol or mint-flavored e-cigarettes, and an estimated 830,000 use candy, dessert, or other sweet-flavored e-cigarette e-liquids [9].

1.2 Definition of the problem and current research methods

This thesis examines the amount of carbonyl compounds produced by various electronic cigarette coil resistances and concentrations of pure and mixed raw materials found in the e-liquids sold to the American consumer. Gas chromatography-mass spectrometry (GC-MS) is the most effective and common software used to rationalize sample capture. However, because of the high reactivity of aldehydes in e-cigarette aerosols, it is difficult to quantify the amounts of individual aldehydes and other carbonyl compounds. Also, aldehydes are reactive compounds and tend to decompose or react during sample preparation or storage. Additional analytical problems arise from their low concentrations [10]. Current methods have used 2,4-dinitrophenylhydrazine (DNPH) silica gel cartridges to capture aerosols produced by e-cigarettes as well as analysis using liquid-liquid extraction (LLE) methods [11-13]. However, the high reactivity and volatility of low-molecular-mass carbonyl compounds impose the need for their derivatization prior to detection by a spectroscopic or chromatographic technique [14-15]. Few studies have measured both free aldehydes and aldehyde-hemiacetals in aerosols generated from various e-liquid mixtures using enhanced carbonyl trapping agents and microfabricated silicon microreactors. Using advanced microfabricated silicon technology allows for microfluidic devices to capture carbonyl compounds with higher efficiencies utilizing chemical reactions. These reactions will allow for compounds in the aerosol to be extracted from the vapor produced by the e-cigarette liquids.

Further, few studies have directly compared evaporation rates to amounts of extracted compounds as well as comparisons of pure, mixed, and flavored e-liquids. In this study, a total of six compounds were included to compare interactions and amounts of each compound produced by a puff of the e-cigarette. The six compounds selected for this study include, formaldehyde, acetaldehyde, acetone, propanal, acrolein, and butanone [16]. Additional compounds were detected in this study; however, these are the compounds that were chosen because they presented the largest peak areas on GC-MS and are the compounds connected to early pulmonary diseases. The compounds examined in the study were utilized to develop calibration curves and determine their retention times.

In addition to public health questions, the factors influencing e-cigarette performance must also be investigated. They are complex and include but are not limited to heat and mass transfers in a cylindrical fibrous medium impregnated with a multi-component e-liquid, vaporization of multi-component systems. Therefore, the systematic analysis of the devices and the e-liquid vaporization is challenging. Thus, understanding how e-cigarettes work and the influence of the key parameters influencing their performance have become major issues in this sector. Indeed, e-liquid consumption informs e-cigarette performance and the optimal use conditions. Currently, there are three categories of parameters that influence e-liquid consumption: parameters related to the design of the atomizer (coil design, supplied power), parameters related to e-liquids (composition), and parameters related to the user (inhalation profile) [17].

1.3 Purpose of research

The purpose of this research is to aid in the analysis of carbonyl compounds produced by newer generations of e-cigarettes. Coil power and temperature, concentrations of propylene glycol and vegetable glycerin, presence of nicotine, and added flavorings will be analyzed to find the effects of these variables on the size and concentration of aerosol particles emitted by newer generations of e-cigarettes. These analyses serve to better understand how use of e-cigarette devices among American adults and youth can potentially lead to pulmonary disease or early forms of cancer.

This study has also examined two toxic compounds, glyoxal and methylglyoxal in e-cigarette aerosols, that are commonly linked to cancers as carcinogenic substances. Glyoxal (GO), and methylglyoxal (MGO) are among the most toxic compounds emitted by electronic cigarettes and regular tobacco cigarette smoke. Airway diseases presented mucus over production as their major pathophysiologic feature [18]. However, the amounts of GO and MGO have not been measured in e-cigarettes and few studies have been conducted on the total generation of these compounds. Reportedly, there are more than 13 million e-cigarette users in the US. When heated, as in e-cigarettes, propylene glycol can generate secondary products. This potential for secondary product formation from heated propylene glycol was first raised around the issue of formaldehyde in e-cigarettes. The potential for secondary product generation from propylene glycol extends beyond formaldehyde. Propylene glycol can generate methylglyoxal and other toxic chemicals such as acetaldehyde and acrolein. In total, this body of research demonstrates that glycol, methylglyoxal, and other toxic carbonyl can be generated from e-cigarettes under typical heating coil temperatures. Methylglyoxal is a major cell-permeant precursor

of advanced glycation end-products (AGEs), which are associated with several pathologies including diabetes, aging, and neurodegenerative diseases [19].

The two main components that make up any e-liquid are propylene glycol (PG) and vegetable glycerin (VG). Pure samples of each PG and VG were tested on GC-MS using both 1.4 Ω and 0.6 Ω resistors. Initial and final weights of each pure sample were taken to gather evaporation rates of each pure substance. Next, PG and VG were mixed to form a broad range of collection data and evaporation rates for all potential e-liquid mixtures that could be purchased on the American market. Using a 50/50 PG/VG mixture, flavor extracts were added to examine carbonyl compound amounts collected from each flavor profile mixture (4.75 mL PG, 4.75 mL VG, and 0.5 mL pure flavor extract). Lastly, nicotine samples were taken from store bought e-liquids with similar flavor profiles as the lab formulated sample. The e-liquids, both commercial and lab formulated, contain a 30/70 PG/VG mixture with strawberry and mango flavor extract and 3 mg/ml of tobacco-free nicotine added to the liquids. Using the calibration curves and sample peak areas, amounts of aldehydes were calculated per puff of e-liquid and were compared to gather a large array of data for all carbonyl compounds detected in newer generations of e-cigarettes.

CHAPTER 2. MATERIALS AND METHODS

In this study, various power and resistance testing was conducted on different strengths of PG/VG components, flavoring components, and nicotine strength components. The following procedures and conditions were used in the collection of all samples using the Stick N18 Kit from SMOK.

- a. 1.4 Ohm Resistor Testing:
 - i. Power: 15 Watts
 - ii. 5 Second Puff Time
 - iii. Collection of 50 mL/Puff
 - iv. Total Collection: 1000 mL = 1 L = 20 puffs each containing 50 mL of vape product
- b. 0.6 Ohm Resistor Testing:
 - i. Power: 15 Watts
 - ii. 5 Second Puff Time
 - iii. Collection of 50 mL/Puff
 - iv. Total Collection: 1000 mL = 1 L = 20 puffs each containing 50 mL of vape product

2.1 Procedure for collection of e-cigarette aerosols

To begin the sample collection process, 1 mL of e-liquid was added to the 3 mL reservoir located on the electronic cigarette ‘MOD’ device [20]. The device used in this study was the Stick N18 Kit from SMOK. Next, the power was set for 15W for both the 1.4 Ω heating coil and the 0.6 Ω heating coils using the dial located at the base of the device. Air flow was set at full air (all four air holes were open) at the neck of the device to allow for consistent measurements across all conducted tests. Before turning on the device, 2-3 dry puffs were taken to prime the coil using a 50 mL syringe. The device was then turned on and left to heat for 1-2 minutes before collecting the first puff (50 mL into syringe). Puffs were collected and injected into a 1L Tedlar bag. Once the sample was collected, the Tedlar bag was placed in an oven set at 45°C and left to sit for 10-15 minutes to evaporate some of the water content produced by the device.

The Tedlar bags were then removed from the oven and attached to a pump, where silica chips were used to collect the aerosols. A microdevice (Figure 1) was fabricated from single-side polished 4-inch diameter silicon wafers in the Micro/Nano Technology Center at the University of Louisville.

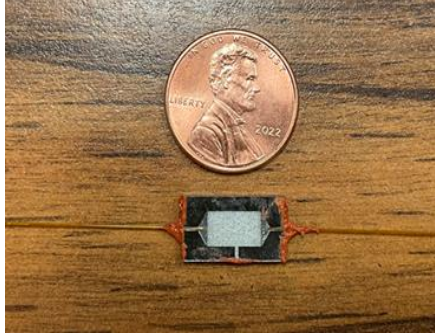


Figure 1. Silica particle packed microfabricated preconcentrate used for all experimentation.

2.2 Microfabrication of the preconcentrates

The microfabricated preconcentrate has dimensions of 14 mm x 8.5 mm x 1 mm. To fabricate the preconcentrates, the cleaned wafer was placed in a furnace to grow around 400 nm thick SiO_2 . Next, a positive photoresist was coated and exposed to UV light using a dark field photomask. The wafer was then developed in Microposit MF319 solution. The thermal oxide in the patterned area was etched by buffered oxide etchant (BOE) to open the wafer for the addition of deep reactive ion etching (DRIE). After BOE, DRIE was performed to create a flow channel, a central cavity with a set of micropillars were also created near the inlet and outlet sections of the device. The depth of channel was measured using the Dektak profilometer and was found to measure 400 μm . The wafers were then placed in an N-Methyl-2-pyrrolidone (NMP) bath, followed by oxygen plasma cleaning. The sacrificial SiO_2 layers were entirely removed by placing the wafer in BOE solution. Later, the wafer was sealed using anodic bonding with a glass wafer. Finally, the wafer was diced into multiple sections to obtain individual

microdevices. The fluidic channels were connected using deactivated fused silica tubes (355µm O.D., 255 µm I.D., Polymicro Technologies) and secured with a silicone adhesive (Duraseal® 1531, Cotronics, NY USA).

Before each sample was collected, the silicon chips being used in the tests were pre-loaded silica gel particles with a size range of 75 to 200 µm and then 15 µL 6.24 mg/mL of PFBHA and left to sit for 24 hours before drying over a hotplate set at 80°C. PFBHA and deuterated propanal, 2-butanone, butanal, 2-pentanone, and hexanal in methanol solutions were prepared. A predetermined amount of these five compounds was mixed to prepare a 1mM concentration mixture along with a 1 mM concentration of each deuterated carbonyl standard. The samples were then reacted with PFBHA solution (PFBHA to carbonyl molar ratio 1.2:1) to form PFBHA-carbonyl adducts. These standard solutions were stored at 4°C in a fridge.

After loading the silicon chips, drying the chips, and collecting the samples produced by the e-cigarette, the pump was set at a flow rate of 40 sccm and bags were left to drain contents into the silica chips for approximately 30-40 minutes. The chips were then eluted with 50 µL of DCM and run through GC-MS for analysis.

2.3 GC-MS Standards

The GC-MS was set to the following standards for all electronic cigarette tests.

- System Type: Agilent Technologies, 7820A, GC System and Agilent Technologies, 5975, Series MSD

- ALS (Front Injector):
- Syringe Size: 10 μ L
- Injection Volume: 2 μ L
- Multiple Injection Delay: 0 sec
- Solvent A Wash: PreInject – 0, PostInject – 0
- Solvent B Wash: PreInject – 1, PostInject – 2
- Sample Wash: 1
- Sample Pumps: 6
- Inlets:
- Split-Splitness Inlet
- Heater: 250 $^{\circ}$ C
- Pressure: 8.2317 psi
- Mode: Split
- Split Ratio: 10:1 at 10 mL/min
- Gas Saver: 20 mL/min after 2 min
- Columns: HP-1
- 450 $^{\circ}$ C: 30 m x 250 μ m x 0.25 μ m
- In: Front SSZ Inlet He

- Out: Vacuum
- Flow: 1 mL/min
- Pressure: 8.2317 psi
- Average Velocity: 36.623 cm/s
- Holdup Time: 1.3653 min

- Oven:
- Equilibrium Time: 0.5 min
- Maximum Oven Temperature: 425°C
- Initial: 60 °C, hold time = 1 min, run time = 1 min
- Ramp 1: Rate = 10 °C/min, 90 °C, hold time = 5 min, run time = 9 min
- Ramp 2: Rate = 5 °C/min, 180 °C, hold time = 1 min, run time = 28 min
- Ramp 3: Rate = 20 °C/min, 250 °C, hold time = 1 min, run time = 32.5 min
- Post Run: 100 °C for 0 min

- MS Instrument:
- Sample Inlet: GC
- Solvent Delay: 3 min

- EMV: Gain Factor
- Gain Factor: 1.00 = 1906 V
- Acq. Mode: Scan and Sim
- Real-Time Plot:
- Time Window: 15 min
- MS Window 1:
- Plot Type: Total
- Y-Scale: 0 to 62592
- MS Window 2:
- Plot Type: None
- Y-Scale: 0 to 100000

All sample information was compiled into tables where the amount of each aldehyde produced by the e-cigarette was calculated by taking the peak areas for the individual aerosol amounts from the GC-MS and dividing them by the amounts collected from the calibration curves. The following results summarize the collected and analyzed data retrieved from GC-MS testing.

CHAPTER 3. RESULTS AND DISCUSSION

3.1 Evaporation rate (weight loss rate) of e-liquids

All e-liquid sample weights were measured to standardize a collection and the number of puffs that would be used for all rounds of testing. To determine the puff related e-liquid weight loss, tests were conducted using 5 puffs, 10 puffs, 15 puffs, and 20 puffs of e-liquid with both 1.4 Ω and 0.6 Ω resistors. The results can be viewed in Table I and Figure 2.

Table I. The total loss of e-liquid vs. puff number using 1.4 Ω and 0.6 Ω resistors at power output of 15W on the SMOK Stick N18 Kit device; (a) pure and mixed PG/VG. (b) flavored e-liquid compounds of 50/50 PG/VG and 5% (V/V) strawberry, mango, and menthol (c) puff number tested for Juice Head manufactured and lab formulated tobacco free nicotine e-liquid samples containing 30/70 PG/VG and 3 mg/ml nicotine.

(a)

5 Puffs (1.4 ohm)							5 Puffs (0.6 ohm)						
Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70	Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70
Total Loss (grams)	0.0363	0.0255	0.0288	0.0307	0.0321	0.0275	Total Loss (grams)	0.0462	0.0309	0.0314	0.0322	0.0335	0.0295
Total Loss (grams)/puff	0.00726	0.0051	0.00576	0.00614	0.00642	0.0055	Total Loss (grams)/puff	0.00924	0.00618	0.00628	0.00644	0.0067	0.0059
10 Puffs (1.4 ohm)							10 Puffs (0.6 ohm)						
Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70	Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70
Total Loss (grams)	0.0625	0.0422	0.0506	0.0562	0.0603	0.0466	Total Loss (grams)	0.0637	0.0595	0.0617	0.0641	0.0654	0.0526
Total Loss (grams)/puff	0.00625	0.00422	0.00506	0.00562	0.00603	0.00466	Total Loss (grams)/puff	0.00637	0.00595	0.00617	0.00641	0.00654	0.00526
15 Puffs (1.4 ohm)							15 Puffs (0.6 ohm)						
Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70	Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70
Total Loss (grams)	0.0916	0.0616	0.0747	0.0806	0.0861	0.0691	Total Loss (grams)	0.1008	0.0889	0.0947	0.0984	0.1002	0.0882
Total Loss (grams)/puff	0.00611	0.00411	0.00498	0.00537	0.00574	0.00461	Total Loss (grams)/puff	0.00672	0.00593	0.00631	0.00656	0.00668	0.00588
20 Puffs (1.4 ohm)							20 Puffs (0.6 ohm)						
Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70	Sample Name	Pure PG	Pure VG	50/50	60/40	70/30	30/70
Total Loss (grams)	0.11911	0.0811	0.099	0.1034	0.1064	0.0913	Total Loss (grams)	0.1394	0.1206	0.1065	0.1065	0.1092	0.0946
Total Loss (grams)/puff	0.0059555	0.004055	0.00495	0.00517	0.00532	0.004565	Total Loss (grams)/puff	0.00697	0.00603	0.005325	0.005325	0.00546	0.00473

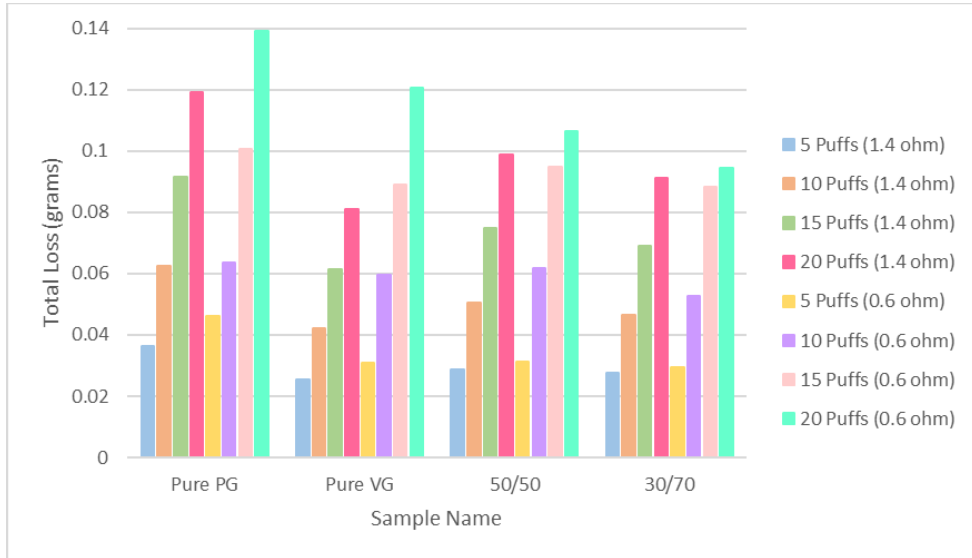
(b)

Strawberry			Mango			Menthol		
5 Puffs (Strawberry)			5 Puffs (Mango)			5 Puffs (Menthol)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0273	0.0522	Total Loss (grams)	0.0232	0.0376	Total Loss (grams)	0.0113	0.03
Total Loss (grams)/puff	0.00546	0.01044	Total Loss (grams)/puff	0.00464	0.00752	Total Loss (grams)/puff	0.00226	0.006
10 Puffs (Strawberry)			10 Puffs (Mango)			10 Puffs (Menthol)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0623	0.0722	Total Loss (grams)	0.0465	0.0573	Total Loss (grams)	0.0318	0.0416
Total Loss (grams)/puff	0.00623	0.00722	Total Loss (grams)/puff	0.00465	0.00573	Total Loss (grams)/puff	0.00318	0.00416
15 Puffs (Strawberry)			15 Puffs (Mango)			15 Puffs (Menthol)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0713	0.0973	Total Loss (grams)	0.0693	0.0798	Total Loss (grams)	0.0488	0.0585
Total Loss (grams)/puff	0.00475	0.00649	Total Loss (grams)/puff	0.00462	0.00532	Total Loss (grams)/puff	0.00325	0.00390
20 Puffs (Strawberry)			20 Puffs (Mango)			20 Puffs (Menthol)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0974	0.1273	Total Loss (grams)	0.0889	0.094	Total Loss (grams)	0.0672	0.0763
Total Loss (grams)/puff	0.00487	0.006365	Total Loss (grams)/puff	0.004445	0.0047	Total Loss (grams)/puff	0.00336	0.003815

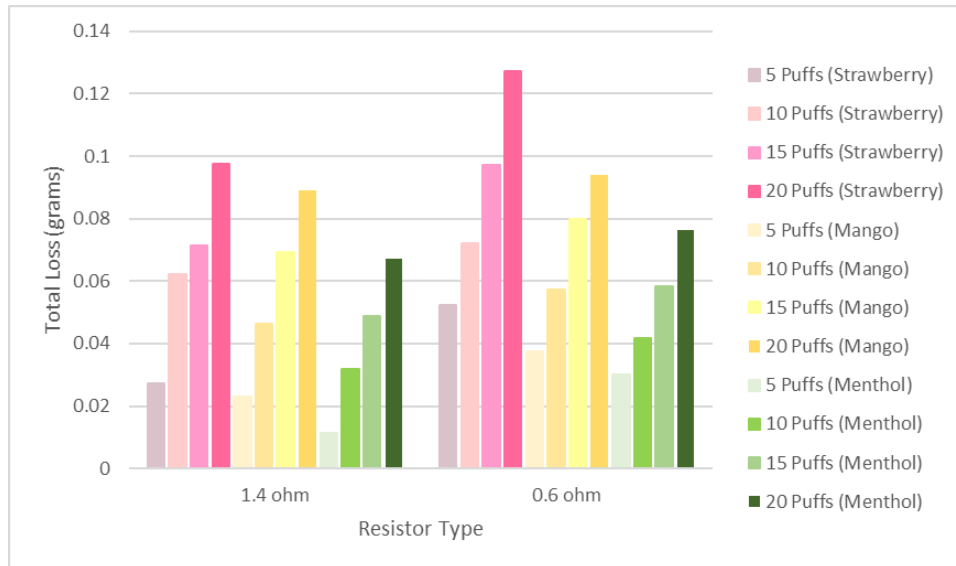
(c)

Juice Head (Commercaill) 3 mg/ml			Lab Formulated 50/50 3 mg/ml		
5 Puffs (Commercial)			5 Puffs (Formulated)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0056	0.015	Total Loss (grams)	0.0175	0.0307
Total Loss (grams)/puff	0.00112	0.003	Total Loss (grams)/puff	0.0035	0.00614
10 Puffs (Commercial)			10 Puffs (Formulated)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0137	0.0327	Total Loss (grams)	0.0262	0.0551
Total Loss (grams)/puff	0.00137	0.00327	Total Loss (grams)/puff	0.00262	0.00551
15 Puffs (Commercial)			15 Puffs (Formulated)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0199	0.0581	Total Loss (grams)	0.0366	0.0787
Total Loss (grams)/puff	0.00133	0.00387	Total Loss (grams)/puff	0.00244	0.00525
20 Puffs (Commercial)			20 Puffs (Formulated)		
Sample Name	1.4 ohm	0.6 ohm	Sample Name	1.4 ohm	0.6 ohm
Total Loss (grams)	0.0339	0.0731	Total Loss (grams)	0.0504	0.0958
Total Loss (grams)/puff	0.001695	0.003655	Total Loss (grams)/puff	0.00252	0.00479

(a)



(b)



(c)

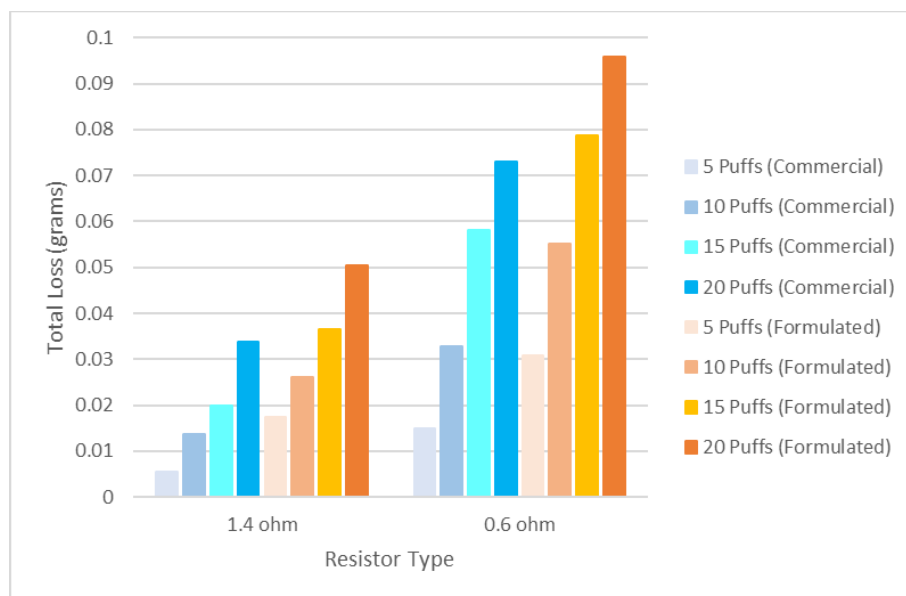


Figure 2. The total loss of e-liquid vs Puff numbers using 1.4 Ω and 0.6 Ω coil at power output of 15W on the SMOK Stick N18 Kit device (a) pure PG, pure VG, 50/50 PG/VG, and 30/70 PG/VG mixture (b) flavored e-liquid 50/50 PG/VG samples (5% v/v strawberry, mango, and menthol) (c) Juice Head brand manufactured, and lab formulated e-liquid containing 30/70 PG/VG and 3 mg/ml of tobacco free nicotine run.

Both Table I and Figure 2 indicate that as the number of puff increases, the total loss of e-liquids increases proportionally. Pure propylene glycol has the highest total loss and total loss/puff among all e-liquids. Lower coil resistance leads to higher total loss/puff.

Therefore, the generated aldehydes in aerosols of e-cigarettes are related the total loss of e-liquids which is affected by the e-liquid composition, number of puffs, e-cigarette power output. These results are important for estimating inhaled total volatile organic compounds for the same number of puffs that e-cigarette users vape in a day. Comparing

all rounds of puff variation data, the conclusion was drawn that the samples with the most consistent data were those taken using 20 puffs of e-liquid from the device on both resistor coils. Selecting the 20-puff variation also allowed for a more standardized collection size of 1000 mL or 1 L sample sizes.

By selecting 20 puffs for all sample sizes, evaporation rate measurements were conducted on each raw material found in the standard e-liquid solution. E-liquid solution is composed of a base of propylene glycol (PG) and vegetable glycerin (VG), a flavor component of 5% (volumetric) added because this mixture is the most common e-liquid on the American market and nicotine. The three flavor components used in this study were strawberry, mango, and menthol. A standard amount of 3 mg/ml of nicotine was used for both the commercial e-liquid and the formulated e-liquid. The physical properties of the base components are summarized in Table II.

Table II. Physical properties of base components (PG/VG).

Physical Properties		
	Propylene Glycol	Vegetable Glycerin
Appearance	Liquid	Liquid
Color	Colorless and transparent liquid	Colorless and transparent liquid
Odor	Little to none	Little to none
Boiling Point	187.4°C	290°C
Flash Point	99°C	160°C
Saturation Pressure	0.089 mg Hg (25°C)	0.0002 mg Hg (25°C)
Relative Density	1.038g/cm ³ (20/20°C)	1.26g/cm ³ (20/20°C)
Chemical Formula	C ₃ H ₆ O ₂	C ₃ H ₈ O ₃

The following results were summarized in Table III and Figure 3 for the evaporation rates of base components, assorted base component amounts of each, flavor components, and added nicotine amounts.

Table III. Assorted evaporation rates of 1.4 Ω and 0.6 Ω resistors using a 15W power output on the SMOK Stick N18 Kit device. For flavored e-liquids, 5% (v/v) flavor concentration was added to 50/50 PG/VG. A standard amount of 3 mg/ml of nicotine was added to 30/70 PG/VG).

1.4 Ohm Resistor											
20 Puffs											
Sample Name	Pure PG	Pure VG	50/50 PG/VG	60/40 PG/VG	70/30 PG/VG	30/70 (PG/VG)	Strawberry (50/50 PG/VG)	Mango (50/50 PG/VG)	Menthol (50/50 PG/VG)	Nicotine (Commercial- 30/70 PG/VG)	Nicotine (Formulated- 30/70 PG/VG)
Total Loss (grams)	0.11911	0.0811	0.099	0.1034	0.1064	0.0913	0.0974	0.0889	0.0672	0.0769	0.0804
Total Loss (grams)/puff	0.0059555	0.00406	0.00495	0.00517	0.00532	0.004565	0.00487	0.004445	0.00336	0.003845	0.00402

0.6 Ohm Resistor											
20 Puffs											
Sample Name	Pure PG	Pure VG	50/50 PG/VG	60/40 PG/VG	70/30 PG/VG	30/70 (PG/VG)	Strawberry (50/50 PG/VG)	Mango (50/50 PG/VG)	Menthol (50/50 PG/VG)	Nicotine (Commercial- 30/70 PG/VG)	Nicotine (Formulated- 30/70 PG/VG)
Total Loss (grams)	0.1394	0.1206	0.1065	0.1065	0.1092	0.0946	0.1273	0.094	0.0763	0.0816	0.0958
Total Loss (grams)/puff	0.00697	0.00603	0.005325	0.005325	0.00546	0.00473	0.006365	0.0047	0.003815	0.00408	0.00479

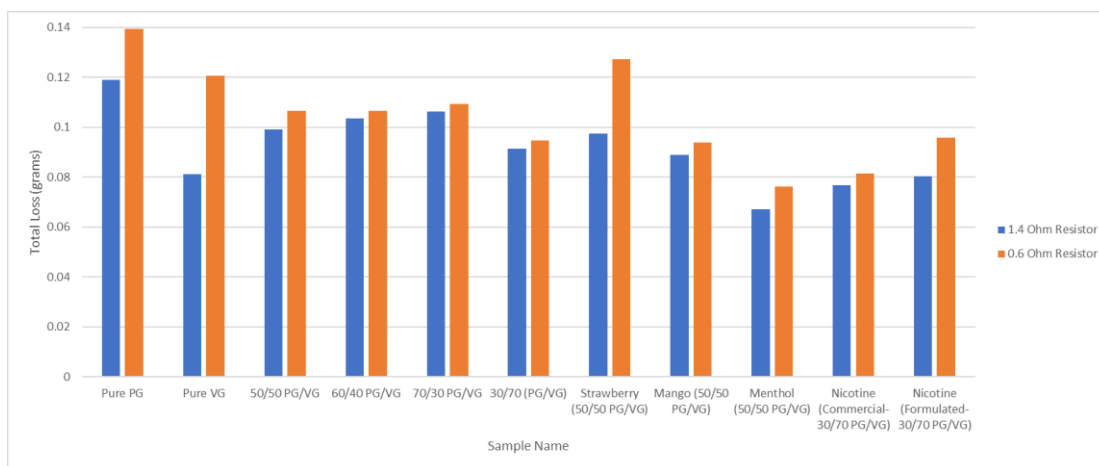


Figure 3. Total loss of e-liquid contents between 1.4 Ω and 0.6 Ω resistors per 20-puff samples, 15W power output on the SMOK Stick N18 Kit device.

Evaporation rates of the lower resistor (0.6 Ω) are notably higher than those of the higher resistor (1.4 Ω). The lower resistor will release the greater number of compounds because it will generate a higher battery power. The greater the battery power, the more compounds the device will produce.

The two resistors were used to test evaporation rates (total loss (grams)/puff) of all e-liquids used in the study and a comparison was conducted to determine the percentage increase of e-liquid weight loss per puff between the two coils. The percent difference between the two resistors averaged a 60% increase between coil strengths.

Examining evaporation rates of various e-liquids brought up the question of, if the power output on the device is changed, will this change the evaporation rates of the samples?

To test this, commercial Juice Head brand tobacco-free 3 mg/ml strawberry-mango flavored nicotine e-liquid with 30/70 PG/VG was used to test the variation in evaporation rates from 9W-30W power outputs from the e-cigarette device. Both 1.4 Ω and 0.6 Ω resistors were used in this study to determine if coil strength paired with power variation would increase evaporation rates. Results are presented below in Table IV.

Table IV. Evaporation rates using variation in power output and coil strength, tested using Juice Head brand tobacco-free 3 mg/ml strawberry-mango flavored nicotine e-liquid with 30/70 PG/VG solution on the SMOK Stick N18 Kit device.

	1.4 Ohm Resistor					0.6 Ohm Resistor				
Power Output	9 W					Power Output	9 W			
Sample Name	5 Puff	10 Puff	15 Puff	20 Puff		Sample Name	5 Puff	10 Puff	15 Puff	20 Puff
Total Loss (grams)	0.0034	0.0121	0.0159	0.0306		Total Loss (grams)	0.0062	0.023	0.0491	0.0647
Total Loss (grams)/puff	0.00068	0.00121	0.00106	0.00153		Total Loss (grams)/puff	0.00124	0.0023	0.00327	0.00324
Power Output	15 W					Power Output	15 W			
Sample Name	5 Puff	10 Puff	15 Puff	20 Puff		Sample Name	5 Puff	10 Puff	15 Puff	20 Puff
Total Loss (grams)	0.0056	0.0137	0.0199	0.0339		Total Loss (grams)	0.015	0.0327	0.0581	0.0731
Total Loss (grams)/puff	0.00112	0.00137	0.00133	0.0017		Total Loss (grams)/puff	0.003	0.00327	0.00387	0.00366
Power Output	20 W					Power Output	20 W			
Sample Name	5 Puff	10 Puff	15 Puff	20 Puff		Sample Name	5 Puff	10 Puff	15 Puff	20 Puff
Total Loss (grams)	0.0092	0.0151	0.0222	0.0439		Total Loss (grams)	0.0175	0.0413	0.0589	0.0744
Total Loss (grams)/puff	0.00184	0.00151	0.00148	0.0022		Total Loss (grams)/puff	0.0035	0.00413	0.00393	0.00372
Power Output	25 W					Power Output	25 W			
Sample Name	5 Puff	10 Puff	15 Puff	20 Puff		Sample Name	5 Puff	10 Puff	15 Puff	20 Puff
Total Loss (grams)	0.0097	0.0158	0.0281	0.0469		Total Loss (grams)	0.0191	0.0465	0.0644	0.0847
Total Loss (grams)/puff	0.00194	0.00158	0.00187	0.00235		Total Loss (grams)/puff	0.00382	0.00465	0.00429	0.00424
Power Output	30 W					Power Output	30 W			
Sample Name	5 Puff	10 Puff	15 Puff	20 Puff		Sample Name	5 Puff	10 Puff	15 Puff	20 Puff
Total Loss (grams)	0.0158	0.022	0.039	0.0549		Total Loss (grams)	0.0238	0.0557	0.0652	0.0867
Total Loss (grams)/puff	0.00316	0.0022	0.0026	0.00275		Total Loss (grams)/puff	0.00476	0.00557	0.00435	0.00434

As power output increased, the evaporation rate of e-liquid solution increased. Between the resistor types, the 0.6 Ω resistor evaporated double the amount of e-liquid that the 1.4 Ω produced.

To ensure repeatability of the results, the device was set at a power output of 15W and both the 1.4 Ω and 0.6 Ω coils were used to run four tests each. The following data can be reviewed in Table V.

Table V. Experimental repeatability using Juice Head brand tobacco-free 3 mg/ml strawberry-mango flavored nicotine e-liquid with 30/70 PG/VG solution, using 20-puff sample collection methods, 15W power output, and 1.4 Ω and 0.6 Ω resistor coils on the SMOK Stick N18 Kit device.

1.4 Ohm Resistor				
Power Output	15W			
Sample Name	Test 1	Test 2	Test 3	Test 4
Total Loss (grams)	0.0336	0.0342	0.0332	0.0339
Total Loss (grams)/puff	0.0017	0.0017	0.00166	0.0017
0.6 Ohm Resistor				
Power Output	15W			
Sample Name	Test 1	Test 2	Test 3	Test 4
Total Loss (grams)	0.0742	0.0739	0.0729	0.0731
Total Loss (grams)/puff	0.0037	0.0037	0.00365	0.00366

Based on the results in Table V, the testing is repeatable. The four tests run using the 15W power output display low error. All tests will be run as a continuation of these data results, running all samples tests using 1.4 Ω and 0.6 Ω resistor coils, full air flow, a power output of 15W, and using 20-puff samples.

3.2 Measurements of aldehydes in aerosols of e-cigarettes

Following the weight and evaporation rate collection results, calibration curves were created using varying strengths of PFBHA solution containing a total of 16 known compounds added to 50 μL of methanol. The calibration curve was developed for carbonyl analysis by injecting 1-15 nmol of PFBHA into 50 μL solution, followed by an injection of 0.5 nmol of heptane- d_{16} to act as an internal reference (IR). The solution was then added to a silica-loaded microdevice, flushed with 50 μL of methanol, and run on GC-MS. The four samples were run through GC-MS and the following results were collected (listed in Table VI).

Table VI. Calibration curve data collection using GC-MS.

Type	5 nmol					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	2376113	1435628	1549946	440308	822059	1679339
Type	10 nmol					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3541508	2769465	3746768	1050180	1210582	4824088
Type	15 nmol					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	7843582	4520855	5686975	1640588	2043974	7778380
	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
5 nmol	2376113	1435628	1549946	440308	822059	1679339
10 nmol	3541508	2769465	3746768	1050180	1210582	4824088
15 nmol	7843582	4520855	5686975	1640588	2043974	7778380

The results were summarized in tables and the values were graphed, as shown in Figure

4.

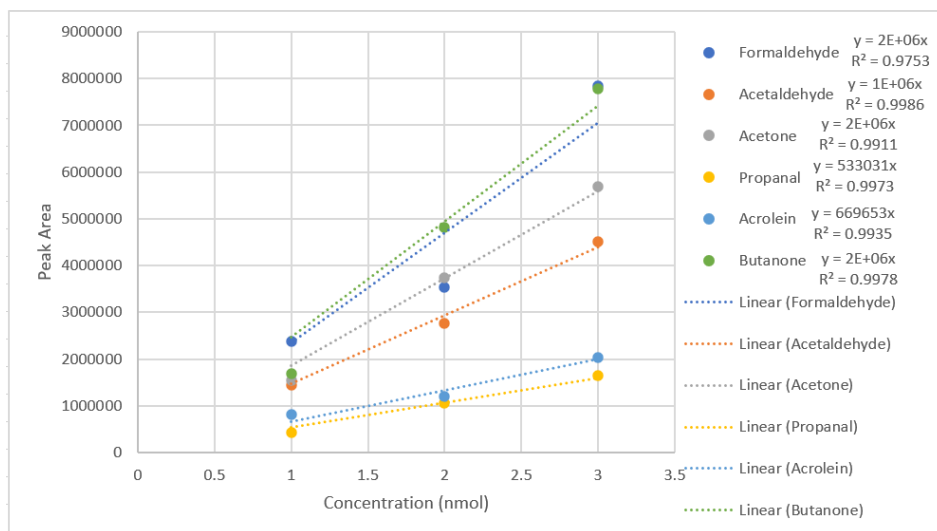


Figure 4. Calibration curves of formaldehyde, acetaldehyde, acetone, propanal, acrolein and butanone to be used to calculate amounts of carbonyl compounds in e-cigarettes.

All linear curves were fitted to the listed data and linear equations were used to calculate the amounts of each carbonyl compound produced by the device for all test runs in the study.

Further calibration curves were created to detect direct amounts of Glyoxal (GO) and methylglyoxal (MGO) compounds. The calibration curves were created by running 10 nmol (with added 200 μ L of methanol) of PFBHA-glyoxal, and PFBHA-methylglyoxal compounds on GC-MS (Figure 5).

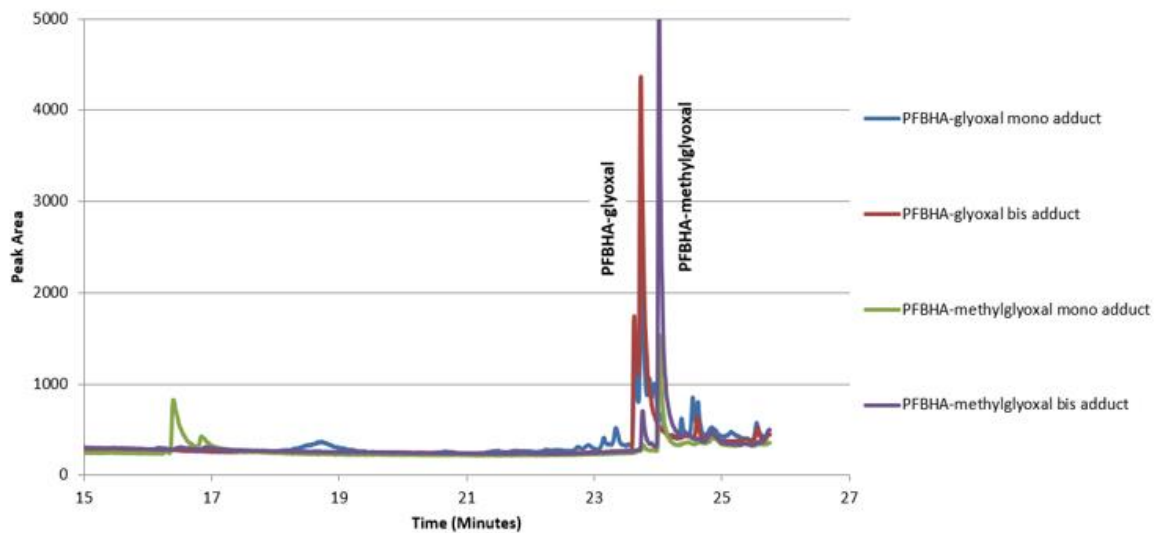


Figure 5. GC-MS plot collected for 10 nmol (with added 200 μ L of methanol) PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) compounds.

The following data was collected, and the linear curves were fitted to the listed data (Figure 6). The linear equations were then used to calculate the amounts of glyoxal and methylglyoxal for all remaining samples tested throughout the study.

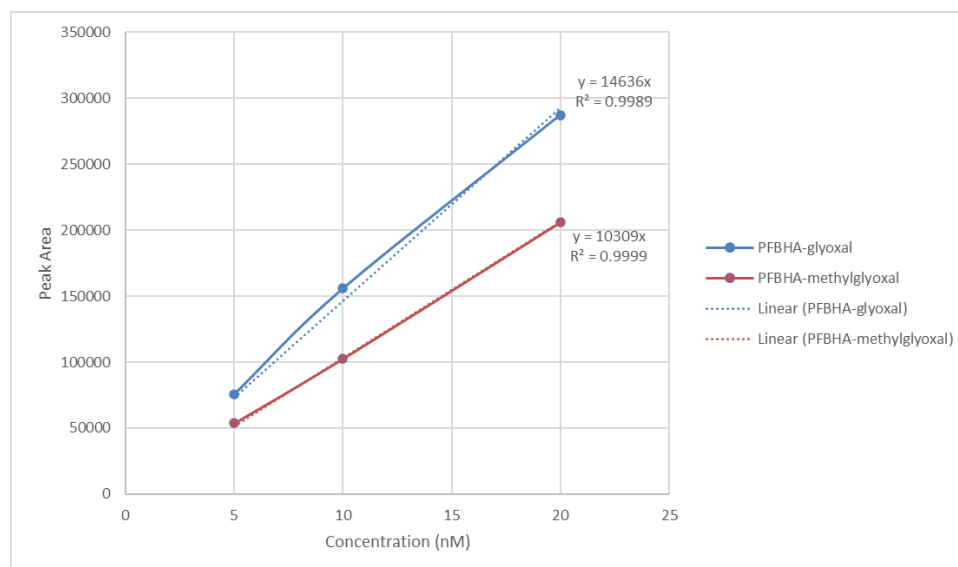


Figure 6. Calibration curve for detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) for mono and bis compounds in e-cigarettes.

The values collected from the plot were used to calculate the total amounts of compounds found in the various e-liquids tested throughout this study by dividing the peak areas acquired from GC-MS with the slope pulled from linear lines of the calibration curve of GO and MGO compounds.

To better understand the impact puff amount had on generation of aerosols, preliminary tests were conducted using a 0.6 Ω resistor and strawberry flavored 50/50 PG/VG e-liquid (5% v/v). The power on the electronic device was changed from 9W through 30W to aloud for better observation of aerosol production. The results are listed in Table VII and Figure 7.

Table VII. Variation of power output using 20-puff collection methods on 0.6 Ω coil resistor for strawberry (5% v/v) flavored 50/50 PG/VG e-liquid on the SMOK Stick N18 Kit device.

Power Output		9W					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	
Area	1573989	5696676	666542	292271	124992	199236	
nmol/20 puffs	3.3398	19.4169	1.7874	2.7416	0.9333	0.4023	
nmol/puff	0.1670	0.9708	0.0894	0.1371	0.0467	0.0201	
Power Output		15W					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	
Area	2407612	13645197	881996	1986907	787334	333028	
nmol/20 puffs	5.1086	46.5092	2.3651	18.6379	5.8787	0.6725	
nmol/puff	0.2554	2.3255	0.1183	0.9319	0.2939	0.0336	
Power Output		20W					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	
Area	2550153	21404834	1807208	2510587	1129555	437407	
nmol/20 puffs	5.4111	72.9577	4.8461	23.5501	8.4339	0.8833	
nmol/puff	0.2706	3.6479	0.2423	1.1775	0.4217	0.0442	
Power Output		25W					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	
Area	3362671	23324835	1951264	3393991	1458490	468311	
nmol/20 puffs	7.1351	79.5019	5.2324	31.8368	10.8899	0.9457	
nmol/puff	0.3568	3.9751	0.2616	1.5918	0.5445	0.0473	
Power Output		30W					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	
Area	6332543	24155350	3585336	3674171	1499767	708632	
nmol/20 puffs	13.4368	82.3327	9.6142	34.4650	11.1981	1.4311	
nmol/puff	0.6718	4.1166	0.4807	1.7232	0.5599	0.0716	

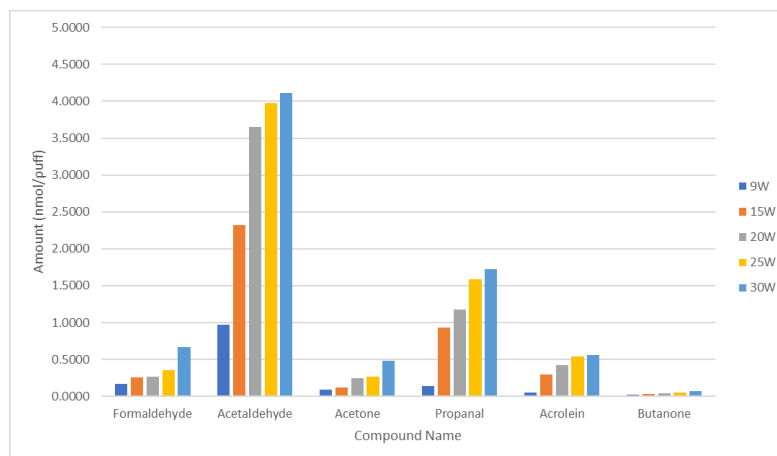


Figure 7. Variation of power output using 20-puff collection methods, 15W power output, and a 0.6 Ω coil resistor to observe aerosol generation with 5% v/v strawberry 50/50 PG/VG e-liquid on the SMOK Stick N18 Kit device.

Analyzing the data and looking at the variation between power outputs, the conclusions that can be made with this set of data are as follows: (1) As the power on the device increased, the amounts of carbonyl compounds produced increased; (2) The lowest power output setting of 9W produced three times less carbonyl compounds than the power output setting at 30W; (3) The most consistent data was that produced by a power output of 15W and 25W.

Following the standard carbonyl compound tests, the device was tested at various power outputs to examine the affect power generation had on production of GO and MGO compounds. The results can be seen in Table VIII and Figure 8.

Table VIII. Detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) using 20-puff collection methods on 0.6 Ω coil resistor and strawberry 50/50 PG/VG e-liquid at various power output ranges on the SMOK Stick N18 Kit device.

Type	9W	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	327216	203252
nmol/20 puffs	22.36	19.72
nmol/puff	1.12	0.99
Type	15W	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	369813	235437
nmol/20 puffs	25.27	22.84
nmol/puff	1.26	1.14
Type	20W	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	427107	266662
nmol/20 puffs	29.18	25.87
nmol/puff	1.46	1.29
Type	25W	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	458795	287381
nmol/20 puffs	31.35	27.88
nmol/puff	1.57	1.39
Type	30W	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	484787	298594
nmol/20 puffs	33.12	28.96
nmol/puff	1.66	1.45

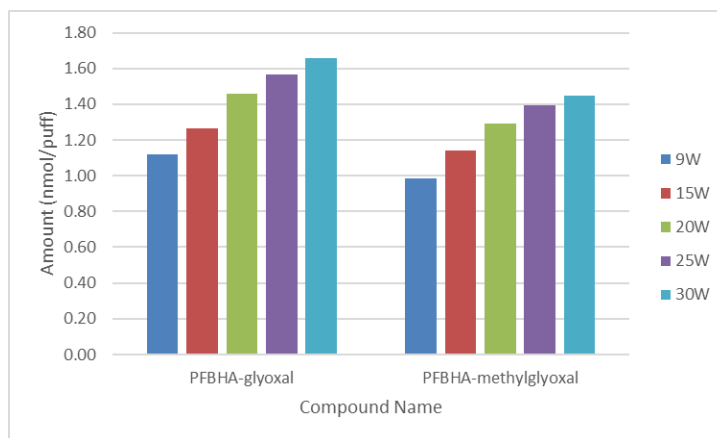
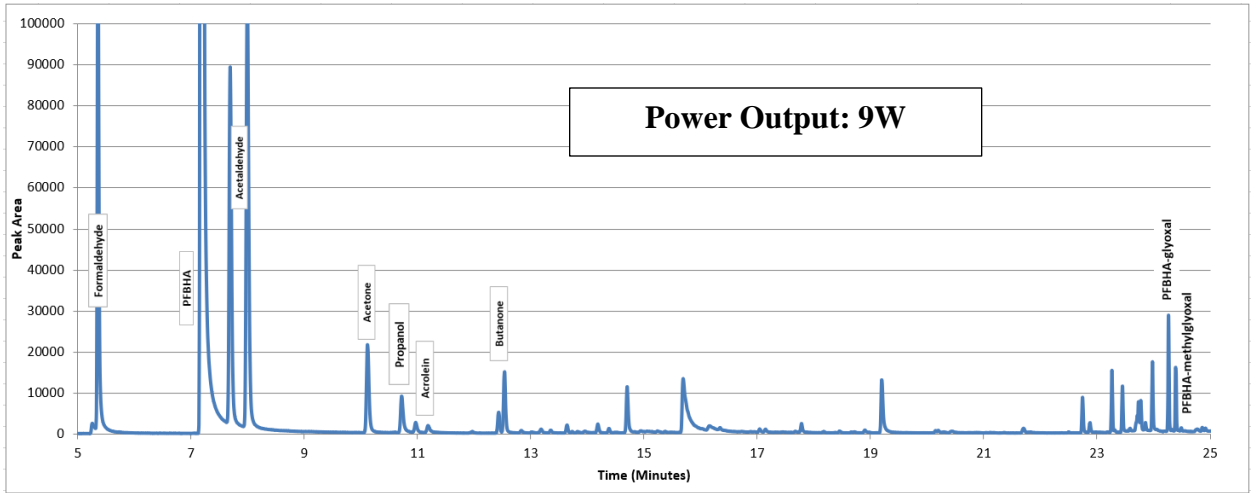


Figure 8. Comparison of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) amounts using 20-puff collection methods on 0.6 Ω coil resistor and 5% v/v strawberry 50/50 PG/VG e-liquid at various power output ranges on the SMOK Stick N18 Kit device.

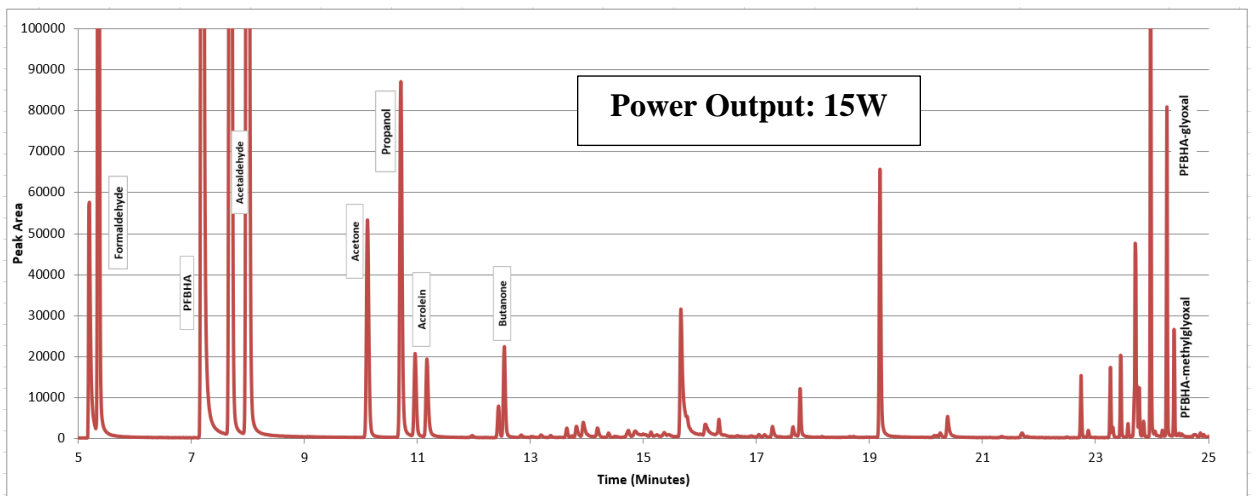
By comparing how power output relates to the generation of GO and MGO products, one can note that the 15W and 25W power outputs generated less amounts of products than 30 W power output. As the power put increases, there is a consistent increase in carbonyl compounds. Through the collection of these results, the conclusion that can be drawn is that the power outputs recommended by the e-cigarette companies (15W and 25W) for greatest performance of the device, from a vaper's standpoint (greatest amount of vapor, best flavor profile, and longest coil life), are the two power settings that generate the greatest amounts of carbonyl compounds.

Under further investigation of how the better performance settings listed by the device manufacture compare to the total generated output of compounds, the following graphs were pulled from GC-MS for direct comparison (Figure 9).

(a)



(b)



(c)

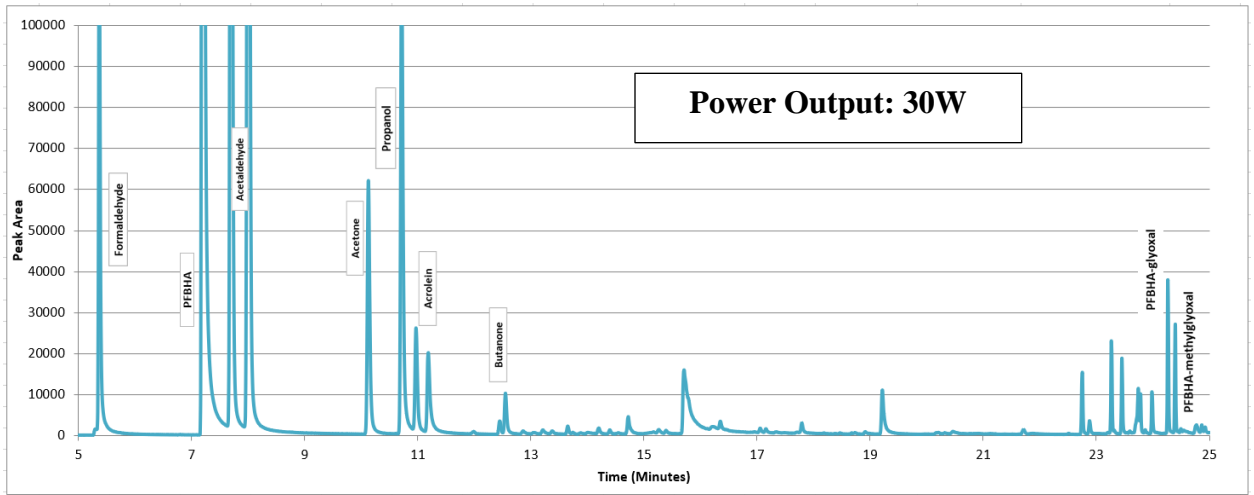


Figure 9. GC-MS data for variation of power output using 20-puff collection methods on 0.6 Ω coil resistor for 5% v/v strawberry 50/50 PG/VG e-liquid on Stick N18 Kit from SMOK device for (a) 9W power output (b) 15W power output (c) 30W power output.

3.3 Generation of aldehydes for Pure and mixed PG/VG at constant battery power output

Analyzing the data, use of a 0.6 Ω coil resistor at various power outputs will generate an increased amount of carbonyl compounds under the recommended power outputs of 15W and 25W deemed safe by the e-cigarette manufacturer of the SMOK device. For this study, 15W was selected to run all tests with because this was the recommended power output setting on the device manual for the Stick N18 Kit from SMOK device used for all testing.

The initial tests that were run were on pure substances of propylene glycol and vegetable glycerin. The two main components that make up the e-liquid solution are PG and VG. To gain an initial base that would help to map out the rest of the experimental procedure, the pure components of PG and VG were tested using the 1.4 Ω and 0.6 Ω resistors at a constant power output of 15 W. The following results were collected, and the calibration curve equations were used to calculate the amounts of individual components that were expressed by the device. The results for pure PG and VG solutions are recorded in Table IX.

Table IX. Pure PG and VG samples at 15 W power output testing to determine baseline for remaining data collection.

Type	1.4 ohm PG					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	2395661	2971690	1756371	872088	468246	1517129
nmol/20 puffs	5.08	10.13	4.71	8.18	3.50	3.063786777
nmol/puff	0.25	0.51	0.24	0.41	0.17	0.15
Type	1.4 ohm VG					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	2927039	3204983	2139766	927855	500495	1791234
nmol/20 puffs	6.21	10.92	5.74	8.70	3.74	3.617331844
nmol/puff	0.31	0.55	0.29	0.44	0.19	0.18
Type	0.6 ohm PG					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3786036	4120115	3208522	1247239	642882	2584089
nmol/20 puffs	8.03	14.04	8.60	11.70	4.80	5.22
nmol/puff	0.40	0.70	0.43	0.58	0.24	0.26
Type	0.6 ohm VG					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	4942725	4947240	4348408	1570596	773433	2922366
nmol/20 puffs	10.49	16.86	11.66	14.73	5.77	5.901611734
nmol/puff	0.52	0.84	0.58	0.74	0.29	0.30

The total amount per puff of each compound can be viewed in the yellow highlighted section of Table IX. The amount of each compound was calculated by dividing the area collected from the GC-MS reading by the calibrated data and then dividing that value by 20 to get the amount per puff of collected aldehydes from the e-cigarette. The individual compounds were then graphed to gain a visual and side-by-side comparison on collection rates between resistor strengths and pure substances (Figure 10).

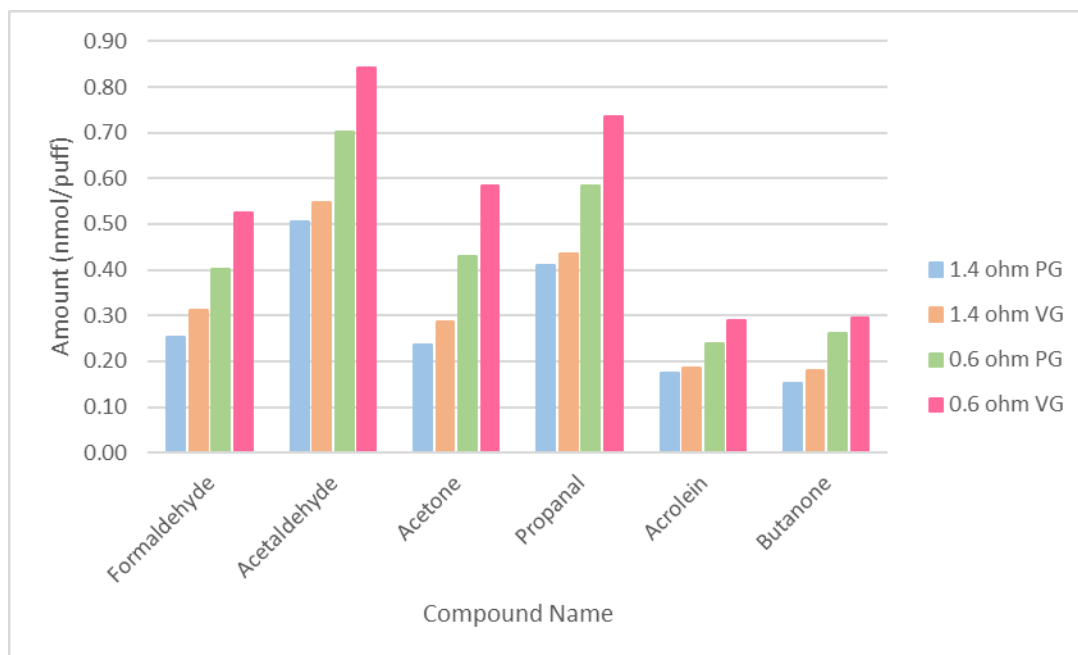


Figure 10. Pure PG/VG aerosol detection amounts per puff of on the SMOK Stick N18 Kit device using a 15W power output.

Following the testing of pure PG/VG components, mixtures of PG/VG were tested. The mixtures that were created included 50/50 PG/VG, 60/40 PG/VG, 70/30 PG/VG, and 30/70 PG/VG samples. The mixtures were tested on both resistor types and were then sent through GC-MS. The results are listed below in Table X.

Table X. Mixed PG/VG samples and amounts of individual components detected per puff of Stick N18 Kit from SMOK device for 1.4 Ω and 0.6 Ω resistors using 15W power output.

1.4 ohm Resistor							0.6 ohm Resistor						
Type	50/50						Type	50/50					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	2913754	2339708	2353939	1696495	435103	1011545	Area	3896877	3372062	3168742	2326464	906168	1958842
nmol/20 puffs	6.18	7.97	6.31	15.91	3.25	2.04	nmol/20 puffs	8.27	11.49	8.50	21.82	6.77	3.96
nmol/puff	0.31	0.40	0.32	0.80	0.16	0.10	nmol/puff	0.41	0.57	0.42	1.09	0.34	0.20
Type	60/40						Type	60/40					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3318133	2827868	2636272	2168932	635490	1455325	Area	4636541	3997670	4079106	2802714	1142120	2382501
nmol/20 puffs	7.04	9.64	7.07	20.35	4.74	2.94	nmol/20 puffs	9.84	13.63	10.94	26.29	8.53	4.81
nmol/puff	0.35	0.48	0.35	1.02	0.24	0.15	nmol/puff	0.49	0.68	0.55	1.31	0.43	0.24
Type	70/30						Type	70/30					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3676917	3306943	3092821	2595452	842142	2059752	Area	5519839	4270078	4573517	3108364	1401920	2871371
nmol/20 puffs	7.80	11.27	8.29	24.35	6.29	4.16	nmol/20 puffs	11.71	14.55	12.26	29.16	10.47	5.80
nmol/puff	0.39	0.56	0.41	1.22	0.31	0.21	nmol/puff	0.59	0.73	0.61	1.46	0.52	0.29
Type	30/70						Type	30/70					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	2852952	1966247	2236850	1618343	430933	961871	Area	3669329	2453142	2936632	2062484	783396	1832680
nmol/20 puffs	6.05	6.70	6.00	15.18	3.22	1.94	nmol/20 puffs	7.79	8.36	7.87	19.35	5.85	3.70
nmol/puff	0.30	0.34	0.30	0.76	0.16	0.10	nmol/puff	0.39	0.42	0.39	0.97	0.29	0.19

The results were then graphed to allow for a comparison between resistor types to be conducted (Figure 11).

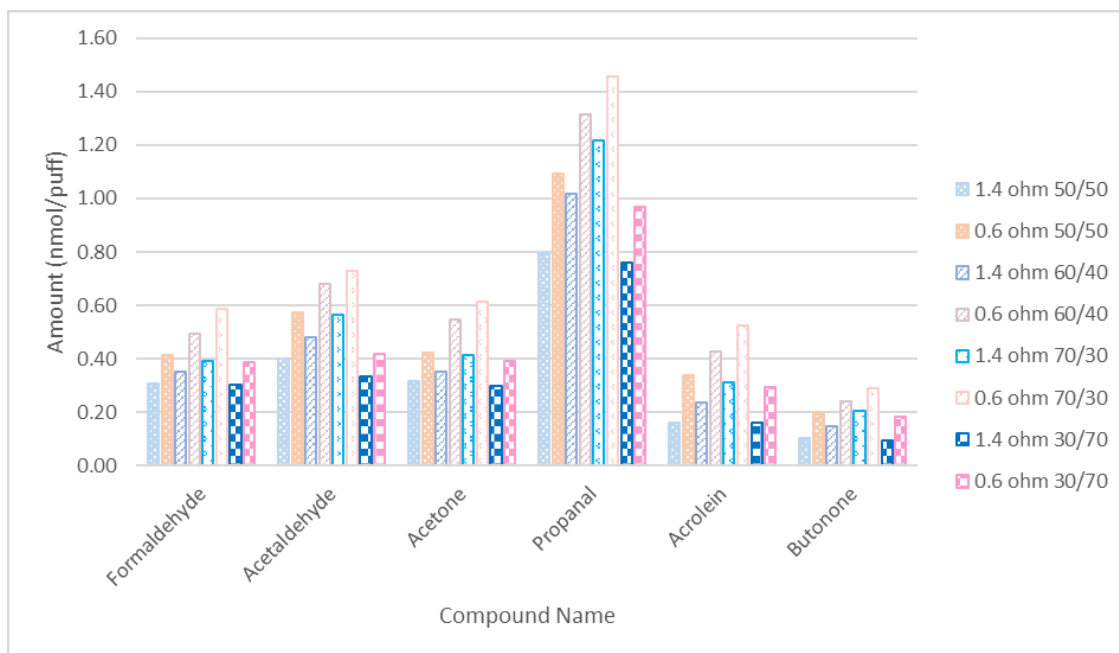


Figure 11. Resistor strength comparison between PG/VG mixtures on the SMOK Stick N18 Kit device using 15W power output.

From the charts and graphs, the results show that as the resistance decreases the amount of generated carbonyl compounds will increase. Also, by comparing amounts of generated compounds, it can be concluded that the base compounds of PG and VG aerosolize the greatest amounts of acetaldehyde and propanal compounds.

Further tests were conducted on base components to determine how change in resistance and variation of base component amounts influenced the production of GO and MGO compounds. The results were summarized in Table XI and Figure 12 displayed below.

Table XI. Detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) using 20-puff collection methods on 0.6 Ω coil resistor and assorted PG/VG e-liquid mixtures at 15W power output on the SMOK Stick N18 Kit device.

Type	Pure PG	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	164491	66173
nmol/20 puffs	11.24	6.42
nmol/puff	0.56	0.32
Type	Pure VG	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	509049	162067
nmol/20 puffs	34.78	15.72
nmol/puff	1.74	0.79
Type	50/50	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	183466	21166
nmol/20 puffs	12.54	2.05
nmol/puff	0.63	0.10
Type	60/40	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	167491	3389
nmol/20 puffs	11.44	0.33
nmol/puff	0.57	0.02
Type	70/30	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	140781	5104
nmol/20 puffs	9.62	0.50
nmol/puff	0.48	0.02
Type	30/70	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	189503	12326
nmol/20 puffs	12.95	1.20
nmol/puff	0.65	0.06

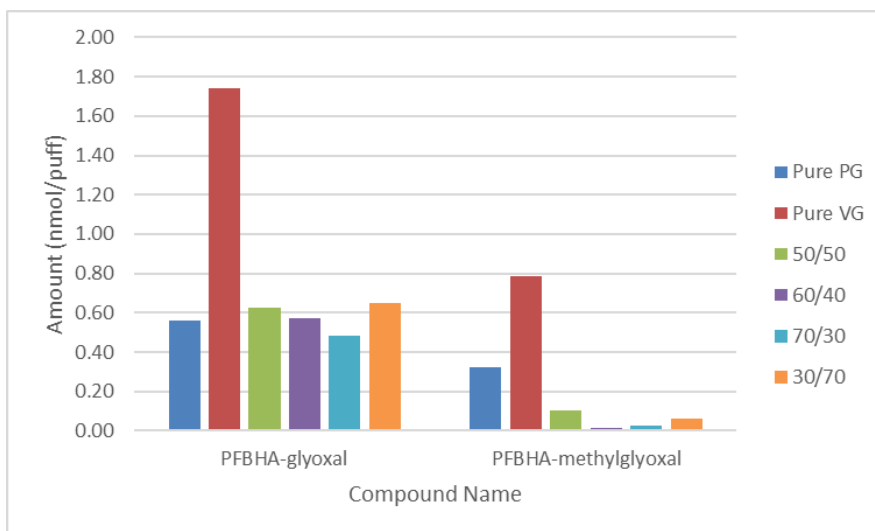
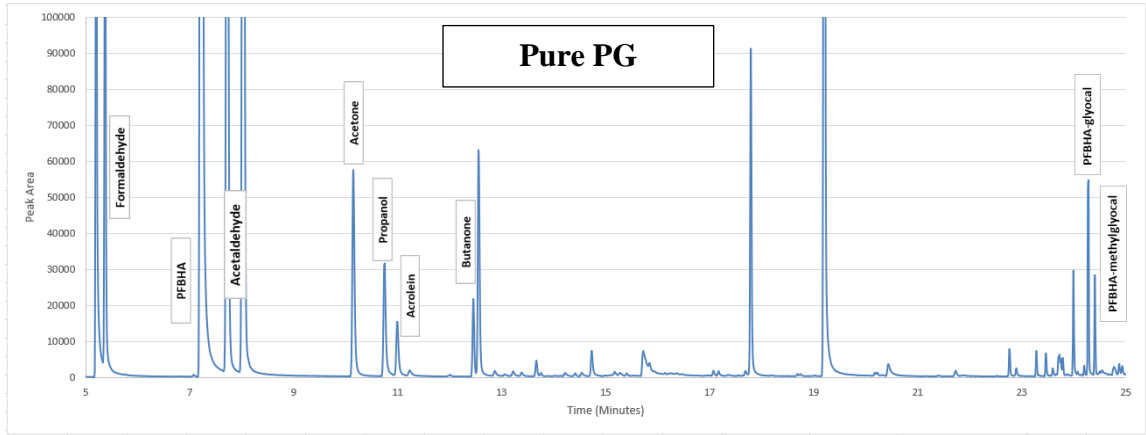


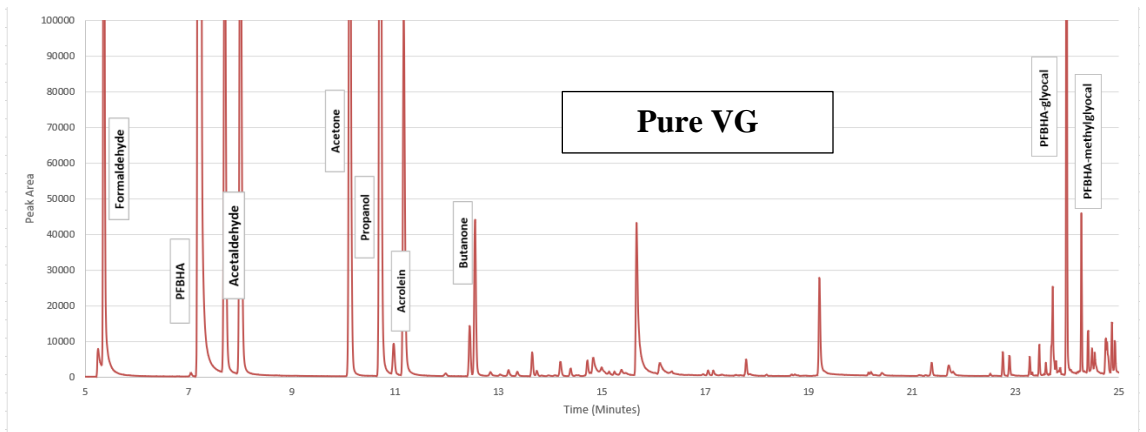
Figure 12. Comparison between PG/VG mixtures for detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) using 20-puff collection methods on 0.6 Ω coil resistor and 15W power output on the SMOK Stick N18 Kit device.

The results were pulled from GC-MS and a direct comparison between the individual base components of PG and VG, along with the most common mixtures found on the market (50/50 PG/VG and 30/70 PG/VG) was conducted. The following graphs display the acquired data (Figure 13).

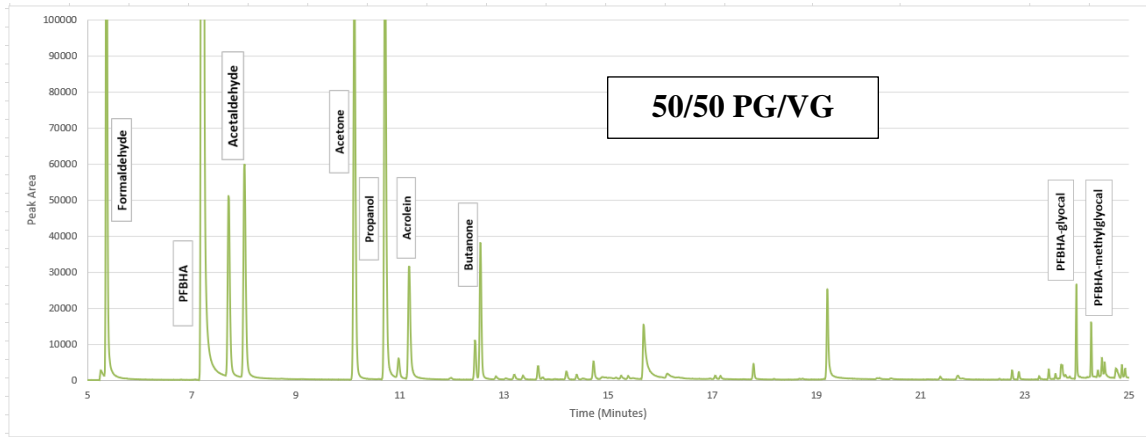
(a)



(b)



(c)



(d)

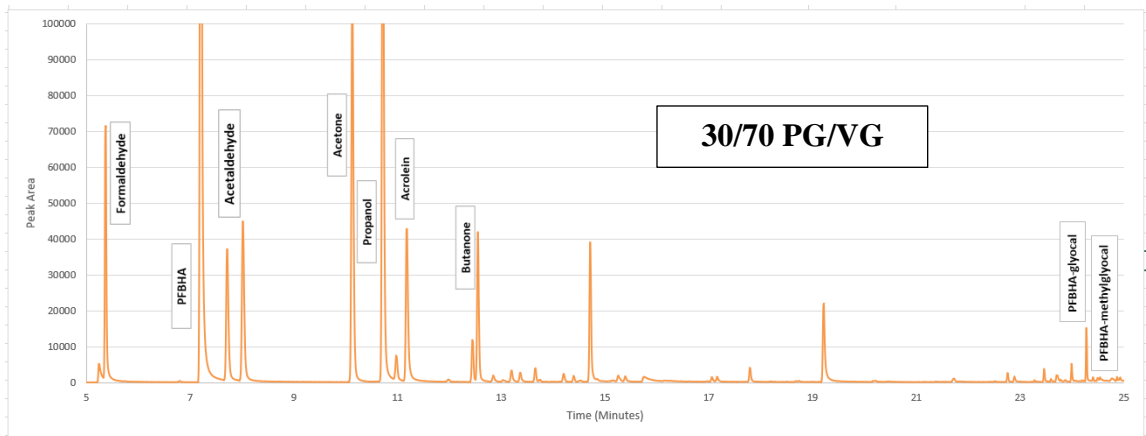


Figure 13. GC-MS data for detection of carbonyl compounds and PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) on mixed PG/VG e-liquids using 20-puff collection methods on 0.6 Ω coil resistor using the Stick N18 Kit from SMOK device for (a) Pure PG (b) Pure VG (c) 50/50 PG/VG (d) 30/70 PG/VG.

To conduct further analysis, percentages of each aerosol were mapped from 100%-30% of each PG/VG component. The individual aerosol components were placed into a table (Table XII) and were graphed (Figure 14) for each coil strength.

Table XII. The number of aldehydes (nmol/puff) for different percentages of PG in PG/VG mixtures for both (a) 1.4 Ω and (b) 0.6 Ω resistors on the SMOK Stick N18 Kit device using a 15W power output and 20 puffs.

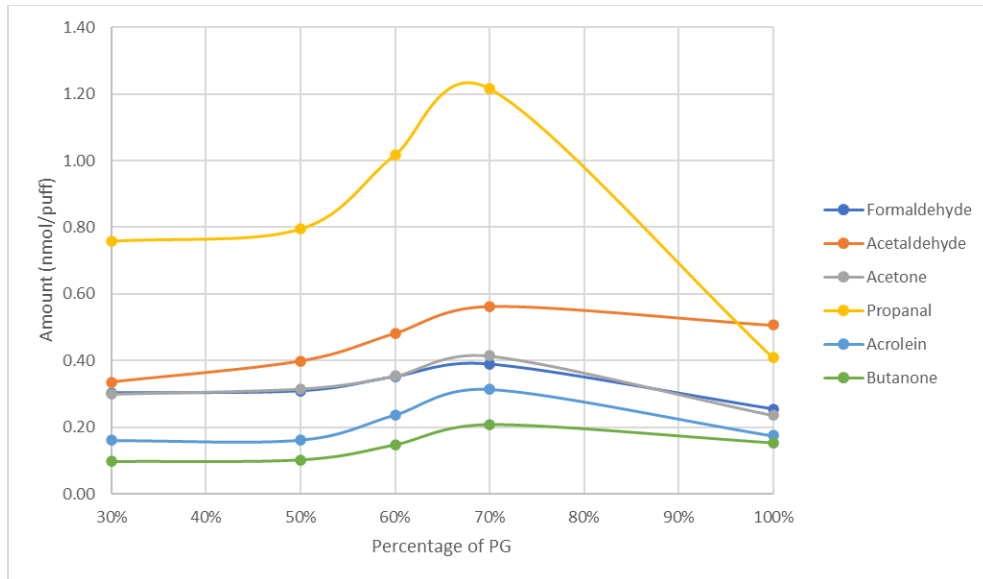
(a)

	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
100%	0.25	0.51	0.24	0.41	0.17	0.15
70%	0.39	0.56	0.41	1.22	0.31	0.21
60%	0.35	0.48	0.35	1.02	0.24	0.15
50%	0.31	0.40	0.32	0.80	0.16	0.10
30%	0.30	0.34	0.30	0.76	0.16	0.10

(b)

	Formaldehyde (PG)	Acetaldehyde (PG)	Acetone (PG)	Propanal (PG)	Acrolein (PG)	Butanone (PG)
100%	0.40	0.70	0.43	0.58	0.24	0.26
70%	0.59	0.73	0.61	1.46	0.52	0.29
60%	0.49	0.68	0.55	1.31	0.43	0.24
50%	0.41	0.57	0.42	1.09	0.34	0.20
30%	0.39	0.42	0.39	0.97	0.29	0.19

(a)



(b)

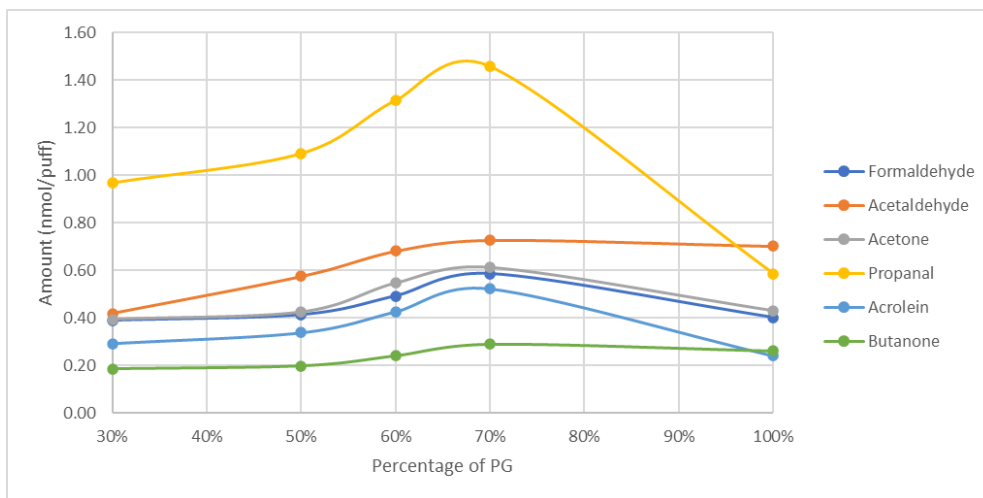


Figure 14. The amount of carbonyls in 20 puffs for different percentages of PG in PG/VG mixtures fusing 1.4 Ω and 0.6 Ω resistors using a 15W power output on the SMOK Stick N18 Kit device.

Using Figure 14 to analyze the variations of aerosol production in relation to the amounts of PG and VG added to the samples, the following results can be gathered. First, 70/30 PG/VG produced the greatest amount of carbonyl compounds because PG is a more volatile component and will evaporate at a faster rate than the VG component. With the extended evaporation rate of the 70/30 PG/VG mix, the aerosols have time to produce and be released within the vapor content gathered during testing. The highest amounts of aldehyde that were produced by all strengths were propanal followed by acetaldehyde. Both propanal and acetaldehyde have low boiling points and will be produced in high amounts in e-cigarette vapor. Under a 1.4 Ω resistor, higher levels of VG will produce larger amounts of acetaldehyde. Under a 0.6 Ω resistor, higher levels of VG will produce larger amounts of acrolein. The higher risk for human health will be found in the lower resistors, the newer generations of e-cigarettes, because as battery power/battery life increases, so will the power sent to the resistors.

3.4 The effect of flavor concentrates and nicotine in e-liquids on generation of aldehydes in aerosols

Following the mixed sample testing, flavor profile testing was run to determine how the three e-liquid flavors of strawberry, mango, and menthol affected the production rate of aerosols. All flavor profile tests were run using 50/50 PG/VG base solution with 5% flavor concentrate (volumetric) added because this mixture is the most common e-liquid on the American market. To make the flavored components, containing no nicotine, the following recipe was used to develop a 50/50 PG/VG mixture (most common strength of non-nicotine containing solution):

- 4.75 mL PG
- 4.75 mL VG
- 0.5 mL Concentrated Flavor (strawberry, mango, menthol)

The three flavored solutions were run using a power output of 15W and using the 1.4 Ω and 0.6 Ω resistors for a total of 20 puffs. The results are summarized in Table XIII.

Table XIII. Flavor profile samples using 50/50 PG/VG with 5% (v/v) strawberry, mango, and menthol e-liquid using 15W power output on the SMOK Stick N18 Kit device.

1.4 Ohm Resistor							0.6 Ohm Resistor						
Type	Strawberry						Type	Strawberry					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3625833	18468848	1955245	652637	314238	1336390	Area	8844581	24598672	2585837	1107265	1215823	1887518
nmol/20 puffs	7.69	62.95	5.24	6.12	2.35	2.70	nmol/20 puffs	18.77	83.84	6.93	10.39	9.08	3.81
nmol/puff	0.38	3.15	0.26	0.31	0.12	0.13	nmol/puff	0.94	4.19	0.35	0.52	0.45	0.19
Type	Mango						Type	Mango					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3153463	13531912	1497289	160343	265944	860816	Area	8343294	19864787	1984940	513200	716704	1430997
nmol/20 puffs	6.69	46.12	4.02	1.50	1.99	1.74	nmol/20 puffs	17.70	67.71	5.32	4.81	5.35	2.89
nmol/puff	0.33	2.31	0.20	0.08	0.10	0.09	nmol/puff	0.89	3.39	0.27	0.24	0.27	0.14
Type	Menthol						Type	Menthol					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3950835	21451397	2255640	823268	345273	1609229	Area	9070758	25787780	2856073	1532047	1571454	2320900
nmol/20 puffs	8.38	73.12	6.05	7.72	2.58	3.25	nmol/20 puffs	19.25	87.90	7.66	14.37	11.73	4.69
nmol/puff	0.42	3.66	0.30	0.39	0.13	0.16	nmol/puff	0.96	4.39	0.38	0.72	0.59	0.23

The results from the yellow section of Table XIII were summarized and compiled into Figure 15. In comparison with PG/VG 50/50 mixtures without any flavors in Table XII and Figure 14, the flavored e-liquids with strawberry, mango and menthol all generated much higher acetaldehydes in aerosols. Therefore, there was also thermal degradation of these flavorings.

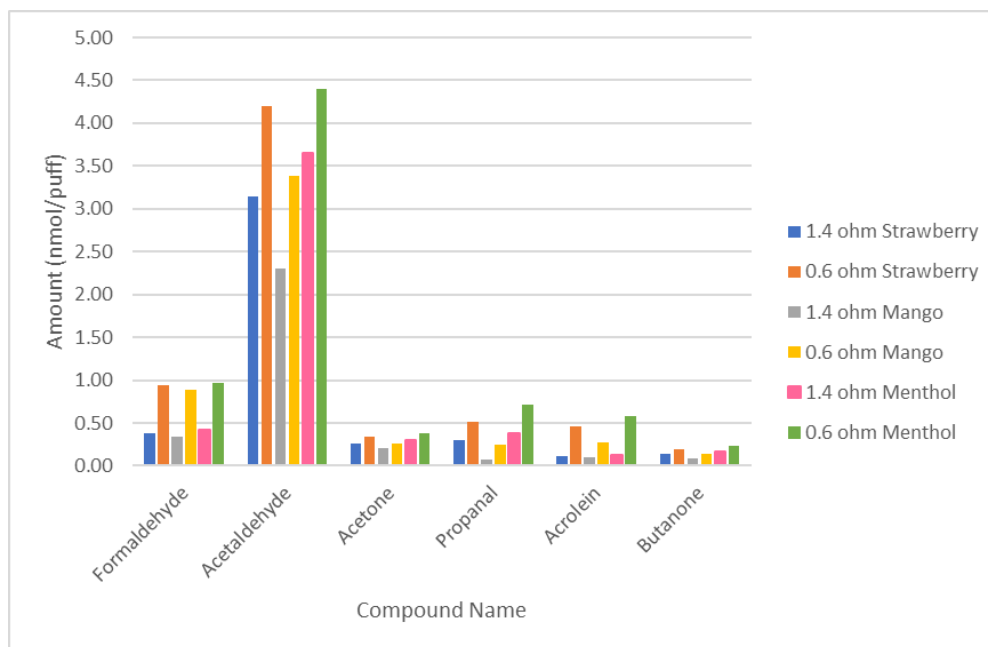


Figure 15. Production of carbonyl compounds by 50/50 PG/VG with 5% (v/v) flavor profile e-liquids using 15W power output on the SMOK Stick N18 Kit device.

To determine how flavor concentrations reacted with the 50/50 PG/VG base mixture to form GO and MGO, the three flavored e-liquids were tested using GC-MS. The results were as follows (Table XIV and Figures 16 and 17). In comparison with Table XI and Figure 12 for pure PG and VG, flavor concentrations also significantly contributed to the increase of GO in aerosols. The highest GO amount was detected from the strawberry flavored e-liquids.

Table XIV. Detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) using 20-puff collection methods on 0.6 Ω coil resistor and 50/50 PG/VG with flavored e-liquid mixtures of 5% (v/v) strawberry, mango, and menthol at 15W power output on the SMOK Stick N18 Kit device.

Type	Strawberry	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	949813	325437
nmol/20 puffs	64.90	31.57
nmol/puff	3.24	1.58
Type	Mango	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	394534	218295
nmol/20 puffs	26.96	21.18
nmol/puff	1.35	1.06
Type	Menthol	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	240627	151463
nmol/20 puffs	16.44	14.69
nmol/puff	0.82	0.73

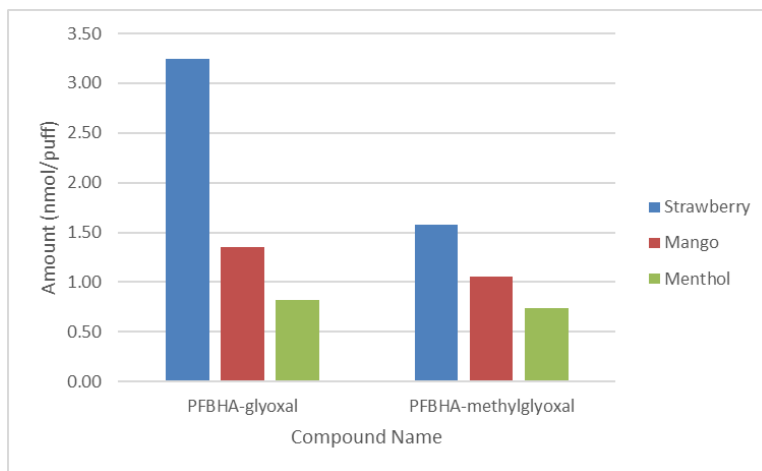
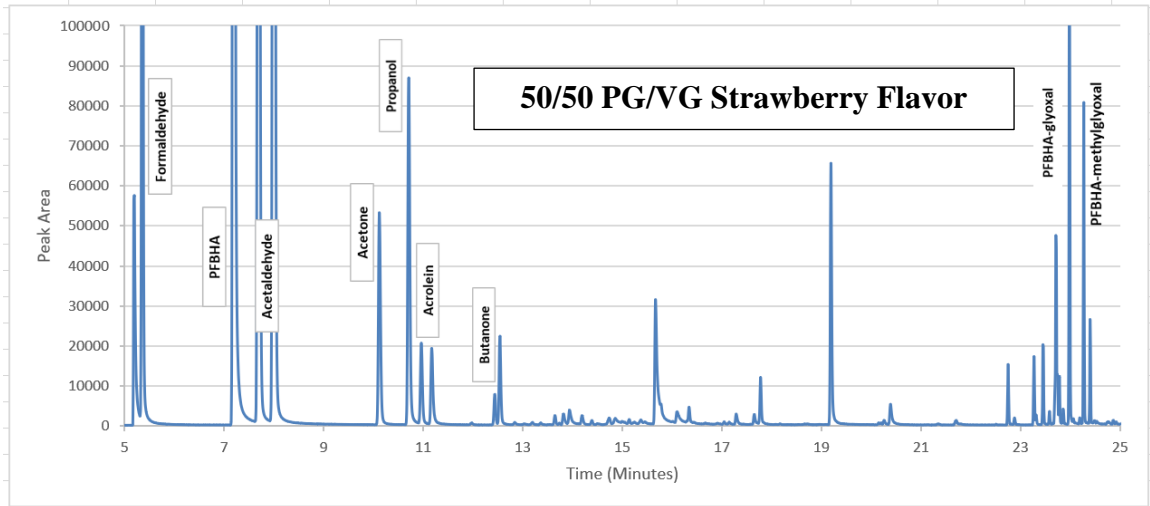
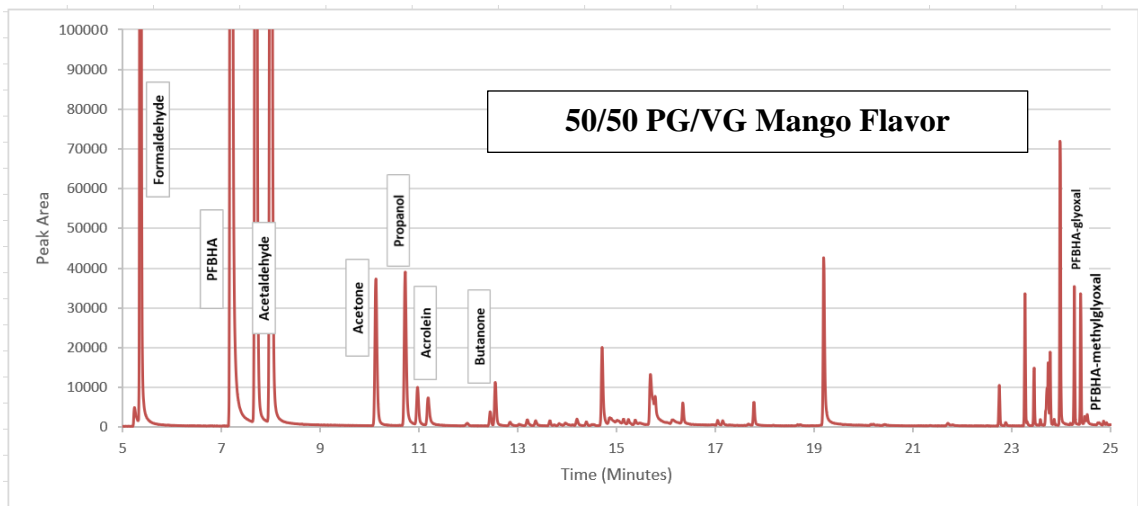


Figure 16. Comparison between flavored e-liquids for detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) using 20-puff collection methods on 0.6 Ω coil resistor and 15W power output for 50/50 PG/VG with flavored e-liquids on the SMOK Stick N18 Kit device.

(a)



(b)



(c)

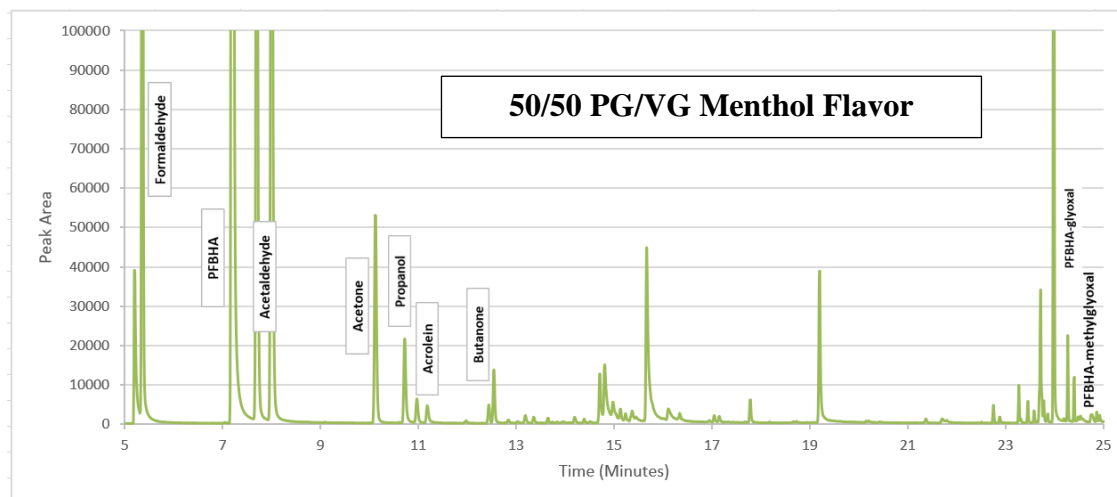


Figure 17. GC-MS data for detection of carbonyl compounds and PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) on flavored e-liquids using 20-puff collection methods on 0.6 Ω coil resistor using the Stick N18 Kit from SMOK device for (a) 50/50 PG/VG strawberry flavor (5% v/v) (b) 50/50 PG/VG mango flavor (5% v/v) (c) 50/50 PG/VG menthol flavor (5% v/v).

Flavor concentration adds to the number of carbonyl compounds aerosolized by the electronic cigarette. The higher the resistance, the higher the compound production. Overall, strawberry flavor produced the greatest amounts of all detected carbonyl compounds, followed by mango, and menthol. Most flavor components on today's market, those containing no amounts of nicotine, will be made with equal parts PG and VG (50% - 50%). Equal parts PG/VG will add to the higher amounts of carbonyl compounds inhaled by the majority of the youth population consuming the product. Strawberry e-liquid is the most popular flavor in the vaping community followed by

mango, a close second. When using a higher resistor, for our case a 1.4 Ω , one is increasing the levels of toxic compounds accumulating within the vapor produced by the device.

The final set of tests were used to examine the relation between carbonyl compound generation and nicotine with e-liquids manufactured by Juice Heads and samples formulated within the lab. All samples for this round of testing used a combination of 30/70 PG/VG, 5% v/v strawberry and mango concentrate, and 3 mg/ml of tobacco-free nicotine. Two rounds of testing were run using 1.4 Ω and 0.6 Ω resistors and a power output of 15W for both commercial and lab formulated samples. The results of this study can be found in Table XV.

Table XV. Comparison between Juice Head brand (commercial), and lab formulated e-liquids composed of 30/70 PG/VG base, 5% strawberry-mango flavor concentrate, and 3 mg/ml of tobacco-free nicotine using 1.4 Ω and 0.6 Ω resistors and a 15W power output on the SMOK Stick N18 Kit device.

Type	1.4 ohm (Commercial)					
Time	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	1456190	18251842	3128505	1074530	384350	1039980
nmol/20 puffs	3.09	62.21	8.39	10.08	2.87	2.10
nmol/puff	0.15	3.11	0.42	0.50	0.14	0.11
Type	0.6 ohm (Commercial)					
Time	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acetone	Butanone
Area	10603624	26442741	7020877	2054613	1378948	2565863
nmol/20 puffs	22.50	90.13	18.83	19.27	10.30	5.18
nmol/puff	1.12	4.51	0.94	0.96	0.51	0.26
Type	1.4 ohm (Formulated)					
Time	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	1123022	9754514	725310	346585	156844	763785
nmol/20 puffs	2.38	33.25	1.94	3.25	1.17	1.54
nmol/puff	0.12	1.66	0.10	0.16	0.06	0.08
Type	0.6 ohm (Formulated)					
Time	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acetone	Butanone
Area	1969808	15376062	1767396	466189	255431	1764439
nmol/20 puffs	4.18	52.41	4.74	4.37	1.91	3.56
nmol/puff	0.21	2.62	0.24	0.22	0.10	0.18

To gain a visual on all data gathered in this round of testing, the results from Table XV are summarized in Figure 18.

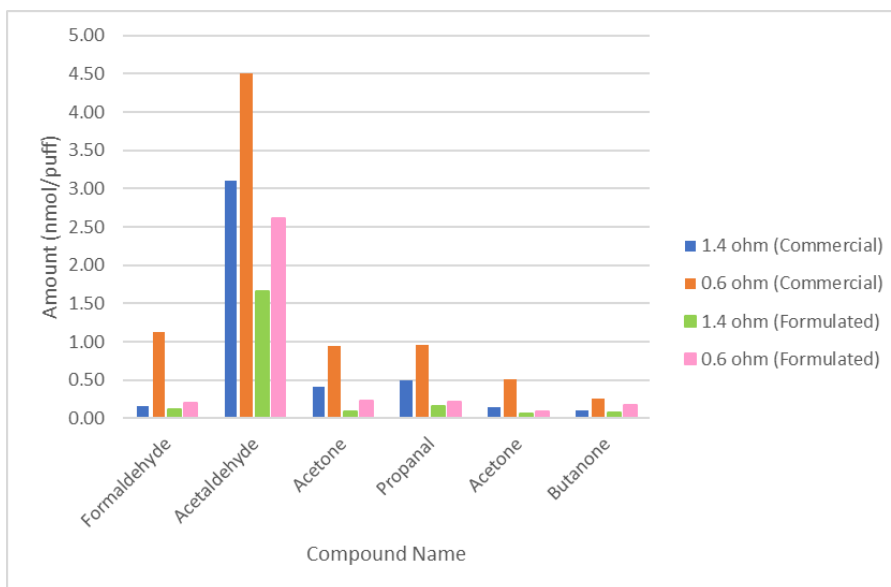


Figure 18. Generation of carbonyl compounds in tobacco-free nicotine, both industry and lab formulated e-liquids, samples using 1.4 Ω and 0.6 Ω resistors and 15W power output on the SMOK Stick N18 Kit device.

Further analysis was conducted on GO and MGO compound amounts within e-liquids that contain nicotine. These tests were conducted to determine if the addition of nicotine to these flavored e-liquids produced more compounds. The results are summarized below in Table XVI and Figures 19 and 20. Figure 19 shows that commercial e-liquid generated more GO and MGO. Figure 20 (a) and (b) further indicate that there were many carbonyl compounds generated from this commercial e-liquid in comparison with the formulated e-liquid.

Table XVI. Detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) using 20-puff collection methods on 0.6 Ω coil resistor and Juice Head brand strawberry-mango 30/70 PG/VG 3 mg/ml tobacco-free nicotine and lab formulated 30/70 PG/VG strawberry-mango 3 mg/ml tobacco-free nicotine e-liquids at 15W power output on the SMOK Stick N18 Kit device.

Type	Commercial	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	1247067	1497445
nmol/20 puffs	85.21	145.26
nmol/puff	4.26	7.26
Type	Lab Formulated	
Name	PFBHA-glyoxal	PFBHA-methylglyoxal
Area	282356	151635
nmol/20 puffs	19.29	14.71
nmol/puff	0.96	0.74

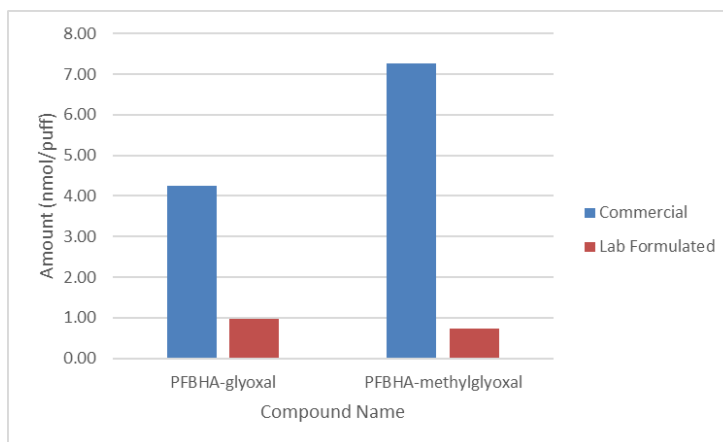
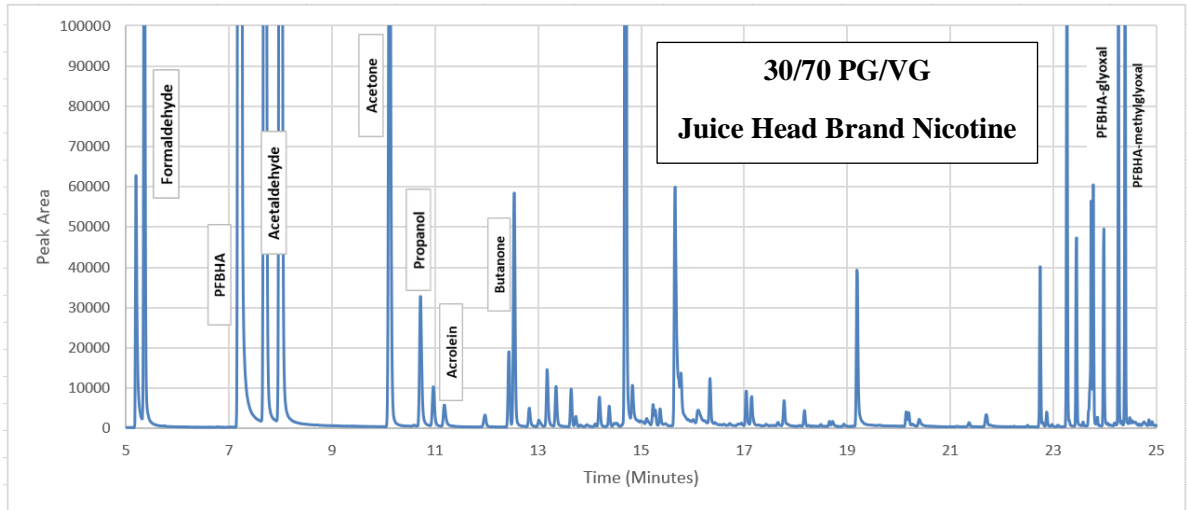


Figure 19. Comparison between Juice Head brand strawberry-mango 30/70 PG/VG 3 mg/ml tobacco-free nicotine and lab formulated 30/70 PG/VG 5% v/v strawberry-mango 3 mg/ml tobacco-free nicotine e-liquids for detection of PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) using 20-puff collection methods on 0.6 Ω coil resistor and 15W power output on the SMOK Stick N18 Kit device.

(a)



(b)

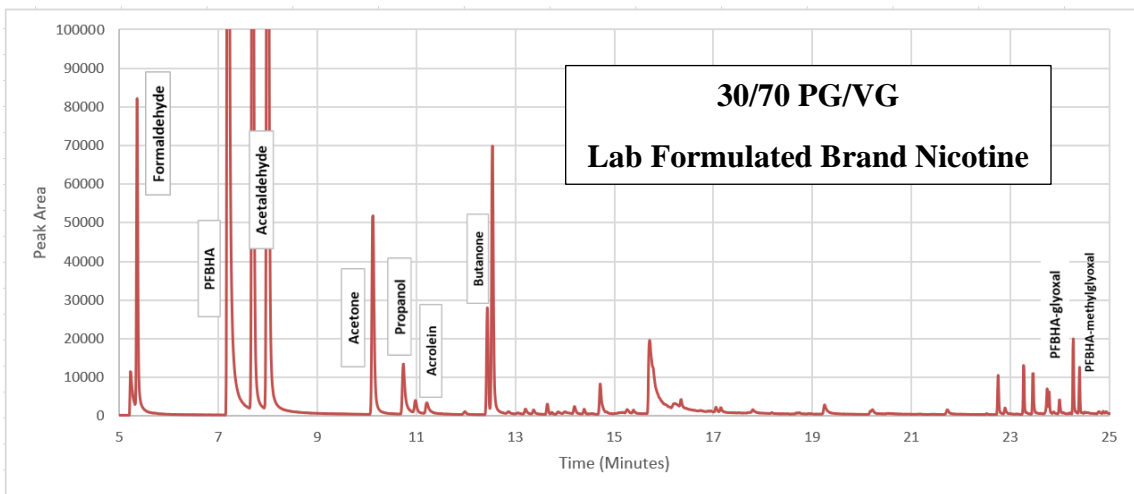


Figure 20. GC-MS data for detection of carbonyl compounds and PFBHA-glyoxal (GO) and PFBHA-methylglyoxal (MGO) on nicotine containing e-liquids using 20-puff collection methods on 0.6 Ω coil resistor using the Stick N18 Kit from SMOK device for (a) Juice Head brand strawberry-mango 30/70 PG/VG 3 mg/ml tobacco-free nicotine (b) lab formulated 30/70 PG/VG strawberry-mango 3 mg/ml tobacco-free nicotine.

Overall, for lower resistance (0.6Ω), the addition of nicotine increases the amount of carbonyl compound output from the electronic cigarette. For higher resistance (1.4Ω), there is much less compound output by the electronic cigarette with the addition of nicotine. The addition of nicotine contributes to a significant increase in acetaldehyde production by the device. Higher amounts of VG will aid in the decrease of carbonyl compounds inhaled by the user rather than just inhaling straight flavored e-liquids. Both resistors contribute similar amounts of aerosolized vapors released by the device, with the higher resistor releasing slightly more compounds than the lower resistor. The lower the resistor, the higher the power and the more flavoring and nicotine strength that will be delivered directly to the consumer.

The final comparison that took place in this study was between all sets of 50/50 PG/VG (mixed samples and flavor profile samples) as well as all sets of 30/70 PG/VG mixture (mixed samples and nicotine samples). A comparison was necessary to determine how the addition of 5% flavor concentrate and nicotine to the 50/50 PG/VG base solution could alter the generation of carbonyl compounds. Table XVII and Figure 21 compare the results for 50/50 PG/VG mixed samples and Table XVIII and Figure 22 compare the results for 30/70 PG/VG mixed samples.

Table XVII. Comparison between 50/50 PG/VG base samples and flavor concentrate samples (5% v/v strawberry, mango, menthol) across 1.4 Ω and 0.6 Ω resistors using 15W power output on the SMOK Stick N18 Kit device.

1.4 ohm Resistor							0.6 ohm Resistor						
Type	50/50						Type	50/50					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	2913754	2339708	2353939	1696495	435103	1011545	Area	3896877	3372062	3168742	2326464	906168	1958842
nmol/20 puffs	6.18	7.97	6.31	15.91	3.25	2.04	nmol/20 puffs	8.27	11.49	8.50	21.82	6.77	3.96
nmol/puff	0.31	0.40	0.32	0.80	0.16	0.10	nmol/puff	0.41	0.57	0.42	1.09	0.34	0.20
1.4 Ohm Resistor							0.6 Ohm Resistor						
Type	Strawberry						Type	Strawberry					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3625833	18468848	1955245	652637	314238	1336390	Area	8844581	24598672	2585837	1107265	1215823	1887518
nmol/20 puffs	7.69	62.95	5.24	6.12	2.35	2.70	nmol/20 puffs	18.77	83.84	6.93	10.39	9.08	3.81
nmol/puff	0.38	3.15	0.26	0.31	0.12	0.13	nmol/puff	0.94	4.19	0.35	0.52	0.45	0.19
Type	Mango						Type	Mango					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3153463	13531912	1497289	160343	265944	860816	Area	8343294	19864787	1984940	513200	716704	1430997
nmol/20 puffs	6.69	46.12	4.02	1.50	1.99	1.74	nmol/20 puffs	17.70	67.71	5.32	4.81	5.35	2.89
nmol/puff	0.33	2.31	0.20	0.08	0.10	0.09	nmol/puff	0.89	3.39	0.27	0.24	0.27	0.14
Type	Menthol						Type	Menthol					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	3950835	21451397	2255640	823268	345273	1609229	Area	9070758	25787780	2856073	1532047	1571454	2320900
nmol/20 puffs	8.38	73.12	6.05	7.72	2.58	3.25	nmol/20 puffs	19.25	87.90	7.66	14.37	11.73	4.69
nmol/puff	0.42	3.66	0.30	0.39	0.13	0.16	nmol/puff	0.96	4.39	0.38	0.72	0.59	0.23

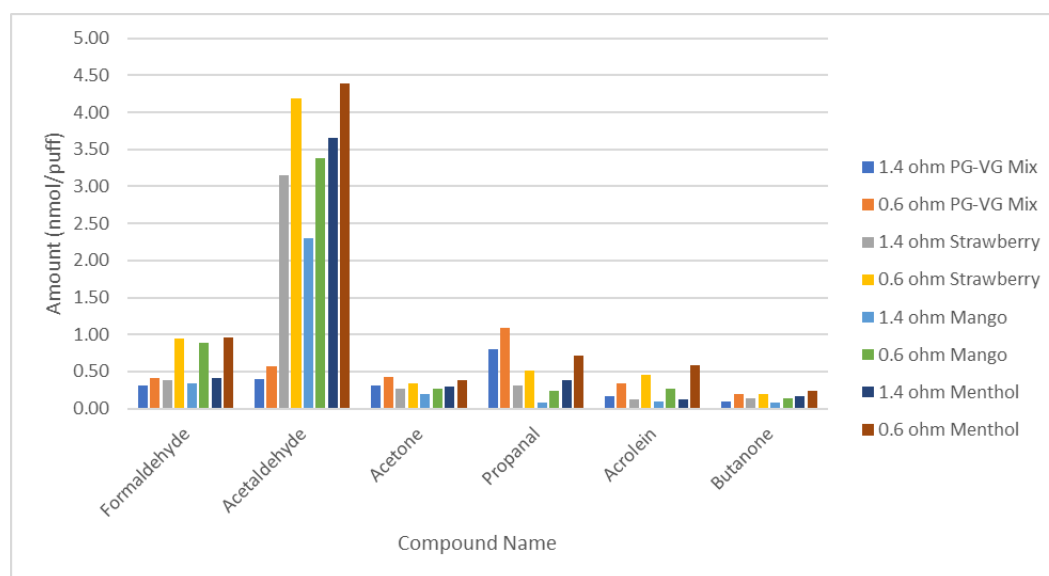


Figure 21. Detection of aerosols in 50/50 PG/VG base samples and flavor concentrate samples (strawberry, mango, menthol) across 1.4 Ω and 0.6 Ω resistors using 15W power output on the SMOK Stick N18 Kit device.

The average was taken between the Juice Heads brand and lab formulated nicotine samples, the following results are summarized below in Table XVIII and Figure 22.

Table XVIII. Comparison between 30/70 PG/VG base samples and tobacco-free 3 mg/ml strawberry-mango manufactured and lab formulated samples across 1.4 Ω and 0.6 Ω resistors using 15W power output on the SMOK Stick N18 Kit device.

1.4 ohm Resistor							0.6 ohm Resistor						
Type	30/70						Type	30/70					
Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Name	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone
Area	2852952	1966247	2236850	1618343	430933	961871	Area	3669329	2453142	2936632	2062484	783396	1832680
nmol/20 puffs	6.05	6.70	6.00	15.18	3.22	1.94	nmol/20 puffs	7.79	8.36	7.87	19.35	5.85	3.70
nmol/puff	0.30	0.34	0.30	0.76	0.16	0.10	nmol/puff	0.39	0.42	0.39	0.97	0.29	0.19
Type	1.4 ohm Nicotine						Type	0.6 ohm Nicotine					
Time	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acrolein	Butanone	Time	Formaldehyde	Acetaldehyde	Acetone	Propanal	Acetone	Butanone
Area	1289606	14003178	1926908	710557.5	270597	901882.5	Area	6286716	20909401.5	4394137	1260401	817189.5	2165151
nmol/20 puffs	2.74	47.73	5.17	6.67	2.02	1.82	nmol/20 puffs	13.34	71.27	11.78	11.82	6.10	4.37
nmol/puff	0.14	2.39	0.26	0.33	0.10	0.09	nmol/puff	0.67	3.56	0.59	0.59	0.31	0.22

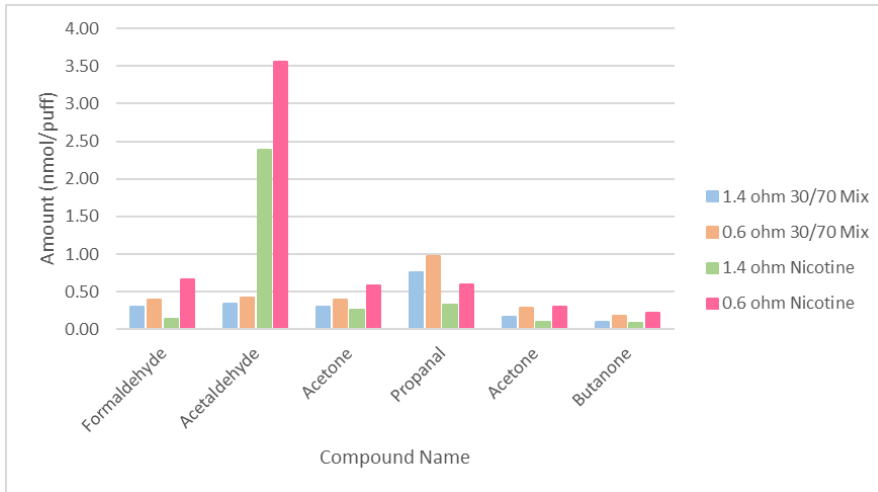


Figure 22. Detection of aerosols in 30/70 PG/VG base samples and Juice Head brand tobacco-free 3 mg/ml strawberry-mango e-liquid and lab formulated samples across 1.4 Ω and 0.6 Ω resistors using 15W power output on the SMOK Stick N18 Kit device.

The results determine that the more volatile substances are the compounds that evaporated first. For the 50/50 PG/VG samples, the samples compared with previous findings in that the lower resistors produce the greatest number of carbonyl compounds and the aerosolized compound that accumulated most in the e-cigarette vapor was acetaldehyde. For most tests conducted in the 50/50 PG/VG comparison, the flavor components produced the greatest number of compounds which directly compares to base compound and flavor profile sample results. For the 30/70 PG/VG tests, the nicotine solution produced the highest numbers of compounds, directly comparing to previous findings. The outlier of both sets of testing was the propanal compound. Propanal for both rounds of testing generated at the greatest amounts for PG/VG base components rather than for flavor profile and nicotine samples. This could be the result of thermal degradation of PG/VG during vaping.

CHAPTER 4. CONCLUSIONS

The study focuses on the how supplied power and atomizer design of e-cigarette devices influence evaporation rates of e-liquids and development and production of aerosols produced by newer generations of e-cigarettes. The studied e-liquids consisted of a quaternary mixture made of PG/VG base, flavor concentrate, and tobacco-free nicotine formulated on a volumetric scale. Two commercial coils were tested, and user behavior was simulated using 50 ml syringes and 1L Tedlar bags, silicon microreactors, and GC-MS analyzation.

Initially, puff numbers were used to determine the ideal sample size to use for the remaining tests, tests were conducted using 5 puffs, 10 puffs, 15 puffs, and 20 puffs of the e-cigarette with both 1.4 Ω and 0.6 Ω resistors. The reproducibility and the repeatability of e-liquid consumption were verified over 15 series of 20 puffs for one of the two tested atomizers. Evaporation rates were collected for all samples that would be used in the study to determine which combinations of e-liquids produced the greatest amounts of carbonyl compounds (formaldehyde, acetaldehyde, acetone, propanal, acrolein, butanone, PFBHA-glyoxal (GO), and PFBHA-methylglyoxal (MGO)). Testing continued using base tests of PG/VG, flavor profiles of strawberry, mango, and menthol, and nicotine samples to compare aerosol production rates between all possible e-liquids available to the American consumer.

The results that can be drawn from the data are that the percent difference of generated aldehydes in e-cigarette aerosols between the two resistors averaged at a 60% increase with the coil resistance decrease to about a half. Because the 0.6 Ω resistor

produces double the amount of carbonyl compounds that the 1.4 Ω produces, the higher risk for human health will be found in the lower resistors, the newer generations of e-cigarettes, because as battery power/battery life increases, so will the power sent to the resistors.

Further work focuses on the influence of e-liquid composition and aerosolized vapor profile on e-liquid consumption, this time using assorted e-liquids that are distributed on the market. Also, nicotine salts were utilized as the testing mediums. A more intense profile of aerosol detection shall be standardized and defined in accordance with the typical user's profile for low resistance atomizers (high quantity of generated vapor) as the ones used in this study.

REFERENCES

1. Ogunwale, M. A., et al. (2017). Aldehyde Detection in Electronic Cigarette Aerosols. *ACS Omega* **2017** 2 (3), 1207-1214. <https://pubs.acs.org/doi/10.1021/acsomega.6b00489>.
2. Chen, M., et al. (2022). Increased Levels of the Acrolein Metabolite 3-Hydroxypropyl Mercapturic Acid in the Urine of e-Cigarette. *Chemical Research in Toxicology Article ASAP*. Users <https://pubs.acs.org/doi/10.1021/acs.chemrestox.2c00145?ref=pdf>.
3. Lechasseur, A., et al. (2019). Variations in coil temperature/power and e-liquid constituents change size and lung deposition of particles emitted by an electronic cigarette. *Physiological Reports*, 7(10): e14093. [Doi.org/10.14814/phy2.14093](https://doi.org/10.14814/phy2.14093).
4. Madej, D. and A. Sobczak. (2018). Methods of Carbonyl Compounds Determination in Aerosol Generated from Electronic Cigarettes. 2018;12(1). DOI: 10.2429/proc.2018.12(1)002.
5. National Academies of Sciences, Engineering, and Medicine. (2018). Public health consequences of e-cigarettes. Washington, DC: The National Academies Press. doi: <https://doi.org/10.17226/24952>.
6. Krüsemann, E. J. Z., et al. (2020). Comprehensive overview of common e-liquid ingredients and how they can be used to predict an e-liquid's flavour category. *Tob Control* 2020;0:1–7. doi:10.1136/tobaccocontrol-2019-055447.
7. Kaur, G., PhD, et al. (2018). Mechanisms of toxicity and biomarkers of flavoring and flavor enhancing chemicals in emerging tobacco and non-tobacco products. *Toxicol Lett*. 2018 May 15; 288: 143–155. doi:10.1016/j.toxlet.2018.02.025.

8. Tierney, P. A., et al. (2015). Flavour chemicals in electronic cigarette fluids. *Tob Control* 2016;25: e10–e15. doi:10.1136/tobaccocontrol-2014-052175.
9. Collin, K. A., PhD., et al. (2019). e-Cigarette Use Among Youth in the United States, 2019. *JAMA*. 2019;322(21):2095-2103. doi:10.1001/jama.2019.18387.
10. Fuchs, P., et al. (2010). Breath gas aldehydes as biomarkers of lung cancer. *Int. J. Cancer*: 126, 2663-2670, 2010, 2009 UICC.
11. El-Hellani, A., et al. (2015). Free-Base and Protonated Nicotine in Electronic Cigarette Liquids and Aerosols. *Chem. Res. Toxicol.* 2015, 28, 1532–1537. doi: 10.1021/acs.chemrestox.5b00107.
12. Lomonaco, T., et al. (2018). Determination of carbonyl compounds in exhaled breath by on-sorbent derivatization coupled with thermal desorption and gas chromatography-tandem mass spectrometry. *30;12(4):046004*. doi: 10.1088/1752-7163/aad202. PMID: 29984708.
13. Zelinkova, Z. and T. Wenzl. (2020). Influence of battery power setting on carbonyl emissions from electronic cigarettes. *Tob. Induc. Dis.* 2020;18(September):77. <https://doi.org/10.18332/tid/126406>.
14. Stashenko, E. E., et al. (2000). Solid-phase microextraction with on-fibre derivatisation applied to the analysis of volatile carbonyl compounds. *Journal of Chromatography A*, 886 (2000) 175–181. <https://www.sciencedirect.com/journal/journal-of-chromatography-a>.
15. Cahill, T. M. (2014). Ambient Acrolein Concentrations in Coastal, Remote, and Urban Regions in California. *Environ. Sci. Technol.* 2014, 48, 8507–8513. [dx.doi.org/10.1021/es5014533](https://doi.org/10.1021/es5014533).

16. U.S. Environmental Protection Agency. (2003). Toxicological Review of Acrolein. CAS No. 107-02-8. EPA/635/R-03/003. <https://www.epa.gov/iris>.
17. Soulet, S., et al. (2018). Influence of Coil Power Ranges on the E-Liquid Consumption in Vaping Devices. *Int. J. Environ. Res. Public Health* 2018, 15(9), 1853. <https://doi.org/10.3390/ijerph15091853>.
18. Kwak S., et al. (2021). Glyoxal and Methylglyoxal as E-cigarette Vapor Ingredients- Induced Pro-Inflammatory Cytokine and Mucins Expression in Human Nasal Epithelial Cells. *Am J Rhinol Allergy*. 2021 Mar;35(2):213-220. doi: 10.1177/1945892420946968. Epub 2020 Aug 3. PMID: 32746708.
19. Azimi P., et al. (2021). An Unrecognized Hazard in E-Cigarette Vapor: Preliminary Quantification of Methylglyoxal Formation from Propylene Glycol in E-Cigarettes. *Int J Environ Res Public Health*. 2021 Jan 6;18(2):385. doi: 10.3390/ijerph18020385. PMID: 33419122; PMCID: PMC7825490.
20. U.S. Department of Health and Human Services Centers for Disease Control and Prevention. E-Cigarette, or Vaping, Products Visual Dictionary. CS 311193-B. https://www.cdc.gov/tobacco/basic_information/e-cigarettes/pdfs/ecigarette-or-vaping-products-visual-dictionary-508.pdf.

VITA

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