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Article

Effects of Manufacturing Variation in Electronic Cigarette Coil Resistance and E-liquid Characteristics on Coil Lifetime and Aerosol Generation

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Abstract: This work investigated the effects of manufacturing variations including coil resistance, initial pod mass, and e-liquid color on coil lifetime and aerosol generation of Vuse ALTO pods. Random samples of pods were used until failure (where e-liquid was consumed, and coil resistance increased to high value indicating a coil break). Initial coil resistance, initial pod mass, and e-liquid net mass ranged between 0.89 to 1.14 [Ω], 6.48 to 6.61 [g], and 1.88 to 2.00 [g] respectively. Coil lifetime with light color e-liquid was μ (mean) = 149, σ (standard deviation) = 10.7 puffs while pods with dark color e-liquid was μ = 185, σ = 22.7 puffs with a difference of ~36 puffs ($p < 0.001$). Total mass of e-liquid consumed until coil failure was μ = 1.93, σ = 0.035 [g]. TPM yield per puff of all test pods for the first session (brand new pods) was μ = 0.0123, σ = 0.0003 [g]. During usage, TPM yield per puff of pods with light color e-liquid was relatively steady while it was continuously decreasing for pods with dark e-liquid. Coil lifetime and TPM yield per puff were not correlated with either variation in initial coil resistance or variation in initial pod mass. The absence of e-liquid in the pod is an important factor in causing coil failure. Small bits of the degraded coil could be potentially introduced to the aerosol. There is a potential correlation of e-liquid color with both coil lifetime and TPM yield per puff. Change of e-liquid color might have been a result of oxidation which changed some nicotine into nicotyrine.

Keywords: aerosol generation, e-cigarette, coil resistance, e-liquid, manufacturing variation.

1. Introduction

The aerosol generated by an Electronic Nicotine Delivery System (ENDS) or electronic cigarette depends on the electrical characteristics of the heating coil [1-5], the characteristics of e-liquid [6-9], the ENDS Power Control Unit (PCU) and battery [10-13], and user behavior such as puff flowrate and puff duration [10,14-16]. It has been widely reported that altering one or more of these factors could change the aerosol emissions from the ENDS such as the total particulate matter (TPM) yield, hazardous and potentially hazardous constituents (HPHC) of the aerosol, and consequently the health effects. In particular, changing coil resistance, which results in altered power consumption, has been associated with changes in some carbonyls and reactive oxygen species (ROS) [1], changes in concentration of selected aldehydes [1-3], changes in nicotine delivery, and changes in puff topography and e-liquid consumption [4,5]. Other studies showed that changing e-liquid characteristics such as nicotine concentration, propylene glycol to glycerin PG/GL ratio, and flavor could lead to changes in the constituents of the aerosol [8,9,17], puffing topography [16], nicotine yield [13,18], and e-liquid consumption [19]. These characteristics could inadvertently change due to factors such as manufacturing variation, environment, user modification, and storage conditions. Such variations may be found across different units of the same product.

Studies have reported manufacturing variations in key device parameters. Davis et. al. [20] studied nicotine concentration in 54 different e-liquid products in the US. Their tests showed the actual nicotine concentration varied between samples of the same product and was not consistent with the labels on the product. They also observed differences in colors of e-liquid which are supposed to be duplicates of the same constituents and characteristics from the same manufacturer. Kim et. al. [21] reported that nicotine concentrations of some e-liquid samples from South Korea varied from the label information provided by the manufacturer. Goniewicz et. al. [22] found that nicotine content in UK brands of e-liquid varied between batches of the same product of up to 12% relative standard deviation (RSD). They also reported that this difference increased up to 31% when comparing samples of different batches. In a study conducted by Pagano et. al. [23], results showed that the actual nicotine content and number of delivered puffs were different from the values claimed on product packaging of some disposable ENDS products. In a recent study, Kosmider et. al. [24] also reported variation in the nicotine content in some e-liquid, and suggested that this variation could be related to degradation of e-liquid product over time.

The variation in coil resistance has the potential to change the power consumed in the coil and thus the amount of heat generated. Consequently, it could change the performance of the ENDS which in turn leads to changes in aerosol emission and presence of the HPHCs. Less attention has been given to the manufacturing variability of coil resistance and the relative impact on variations in aerosol yield. Our previous work [25] introduced a robust method to measure coil resistance of e-cigarettes and documented manufacturing variation in coil resistance of two popular pod-style ENDS: Vuse ALTO and JUUL. Pod units included in the test showed variation in coil resistance of ~30% and ~7.4% for ALTO and JUUL, respectively. In preliminary work for this study, we observed manufacturing variation in the gross mass of Vuse ALTO pods, thought to be dominated by the amount of e-liquid in the pod, rather than differences in the container mass.

More study is needed to better understand manufacturing variation and the impact of this variation on device performance. This study leads to better understanding of the impact of manufacturing variation on coil lifetime and aerosol emissions. Therefore, this study begins to lay the groundwork for regulations requiring manufacturer's to report variations in tobacco product components as part of the premarket approval process.

1.1. Study Objectives

This study focused on investigating the effects of manufacturing variations on e-cigarette performance; specifically: (1) the effects of the variation in initial coil resistance on coil lifetime and TPM yield per puff, and (2) the effects of the variation in initial pod mass on coil lifetime and TPM yield per puff. As we were analyzing the results of this experiment, we determined a posteriori objective which was (3) to investigate the effects of e-liquid color on coil lifetime and TPM yield per puff. To the best of our knowledge, this is the first work which systematically investigates effects of manufacturing coil and e-liquid variations on the lifetime and emissions characteristics of ENDS.

2. Materials and Methods

In order to investigate the effects of initial coil resistance and initial pod mass on device performance, this study measured coil lifetime and total particulate matter (TPM) yield per puff. Coil lifetime was measured as the number of repeated puffs delivered from a brand-new pod until the coil broke, without refilling the pod, as indicated by a sharp increase in coil resistance. Coil breakage could be a result of coil aging, excessive usage, or a result of energizing the coil in the absence of e-liquid. The excessive heat generated by the coil could lead to melting and breaking the coil. All of these failure mechanisms would be reflected in such a sharp increase in coil resistance. Therefore, coil resistance was deemed to be a good indicator of coil breakage.

2.1. Test Specimens

The experiments were conducted on a commercially available pod style ENDS, Vuse ALTO [26] pods N = 15 which is one of the most popular e-cigarettes among teenagers [27,28]. The manufacturer reported that these pods are filled with 1.8 [mL] of e-liquid. The pods used in this study filled with nicotine flavor e-liquid manufacturer labeled 5% nicotine concentration. They were purchased from local retail shops and national online vendors.

2.2. Aerosol Generation and Collection

The previously validated Programmable Emissions System™ (PES™-1) was used to activate and run the ENDS under test to generate and collect aerosols as described in [29]. The system can be configured to perform puffing profiles based on a wide range of puff flowrates, puff durations, and inter-puff intervals. It uses a vacuum tank (5.0 [L] with pressure as low as -60[kPA]), a proportional valve (KPIH-VP-20-156-25, Kelly Pneumatic Inc. with 10 [m Sec] response time) and a gas flow meter (M-50SLPM-D-30PSIA/5M, Alicat Scientific, Inc.) connected in series to generate the required flowrates. The flow meter and proportioning valve are digitally monitored and controlled to implement the desired puffing profile. Several particulate phase collection modules can be used with this system. The current study used Cambridge style single stage filter pads.

The TPM collected during each trial was measured by differencing the mass of the filter pad before and after the trial. A Mettler AE240 Analytical Balance gravimeter was used. The Mettler balance provided a protected weighing space with accuracy of +/- 0.0002 [g] (0.2 [mg]) [30]. The same gravimeter was also used to measure pod mass before and after each trial.

Figure 1 shows the entrance region of the experimental setup, which includes the inlet of the PES-1, filter pad holder, short connecting tube, and the ENDS under test. The PES-1 was set up with an angle of 30° to mimic the declination angle of the ENDS while being puffed, determined in a previous Master's thesis which analyzed data from YouTube videos of e-cigarette users while vaping their personal ENDS in their natural environment [31]. The mouthpiece of the Vuse ALTO ENDS was connected to the inlet of the filter pad holder through a short connecting tube. The tube was fixed to the mouthpiece with Bemis™ Parafilm™ M Laboratory Wrapping Film (not illustrated in the picture for visibility).

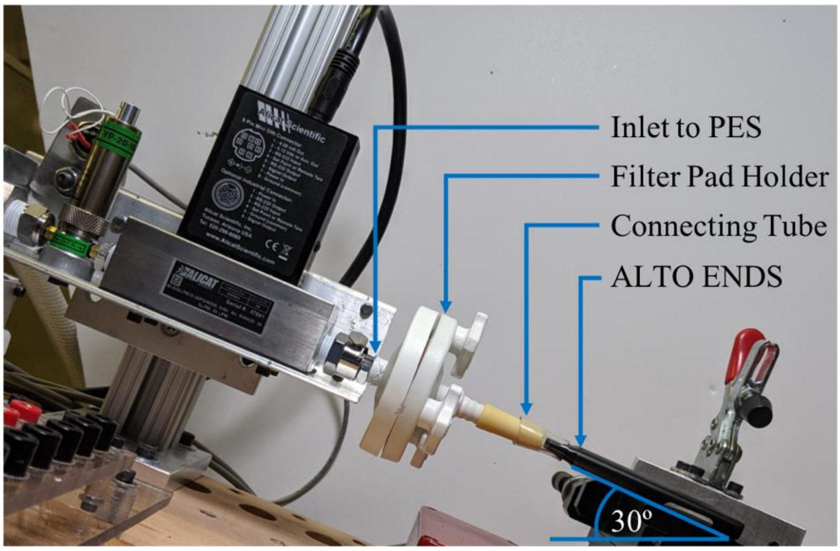

Figure 1: PES-1 setup with ALTO connected to it at 30° angle through a filter pad holder.

2.3. Coil Resistance Testing Apparatus

The test fixture presented in [25,32] was used to measure coil resistance, built by repurposing the housing of the PCU (power control unit) of the targeted ENDS, mimicking the geometrical and electrical conditions of the original ENDS. The test fixture provides measurement of the effective coil resistance which accurately represents the resistance seen by the PCU during operation. The effective coil resistance is the summation of the resistances of the connectors from the PCU to the pod, the internal connection pins in the pod, and the heating coil. The fixture utilizes a four-wire resistance measurement configuration with customized test leads. A detailed step-by-step protocol for building this fixture has been published on protocols.io [32]. The fixture was used with 34465A KEYSIGHT™ Digital Multimeter [33].

The coil resistance test fixture [25,32] was held vertically using a table top vise, to ensure consistency in the measurement and minimize error resulting from motion. The test fixture was connected to the digital multimeter and communicates with the PES-1 personal computer via USB serial connection. When the pod was inserted in the test fixture and the resistance measurement was ready to be made, a button in the PES-1 software can be clicked to make the coil resistance reading and record the results in the dataset.

2.4. Data Acquisition

Several types of data were collected while conducting the experiment, including the measured puffing profile (flowrate), labeling data about the ENDS and pod under test, filter pad mass, pod mass, and coil resistance. The measured flowrate was automatically collected by the PES-1 controller software which was saved as a comma-separated values file at the end of the session for later usage. The PES-1 controller software also provided means to enter the other types of the data. The labeling data of the ENDS and pod under test were scanned by a barcode scanner before the session, the filter pad mass and pod mass values were manually measured using the gravimeter and manually entered to the PES-1 controller software before and after the session (i.e. after 20, 10 or 5 puffs). Coil resistance was read by the PES-1 controller software when the pod was inserted in the test fixture before and after each session. The step by step testing procedure employed in this study has been published [34] to foster reproducibility of this work.

2.5. Puffing Profile

The lifetime testing puffing profile used here included emissions testing sessions with uniform rectangular shape puffs whose puff flowrate was 18.33 [mL/Sec], puff duration was 5.5 [Sec], and puff interval was 11 [Sec]. The number of puffs per session was 20 puffs for the earlier portion of coil lifetime and was reduced to 10 or 5 puffs per session as each coil lifetime test progressed. This puff profile was designed to accelerate lifetime testing by providing long puff duration and short puff interval in order to shorten the time required to fully consume the pod and achieve coil failure. Such technique (accelerated lifetime testing) has been used in quality assurance testing standards of many common products [35,36]. The profile was also carefully designed to consider the parameter margins suggested by the manufacturer, in order to avoid interfering with the results of the experiment while trying to comply with some aspects of the Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA) standard for e-cigarette aerosol generation and collection [37]. The flowrate was chosen based on the results of a preliminary experiment done in our lab which showed that Vuse ALTO ENDS was consistently activated at flowrate of ≥ 15 [mL/Sec]. While the puff flowrate complies with CORESTA standard, it was also intentionally selected to be low in order increase the aerosol generation efficiency. The puff duration of 5.5 [Sec] was chosen to fully exercise the five seconds specified by the manufacturer before the ENDS automatically stops puffing [38], while the CORESTA standard specifies a puff duration of 3 ± 0.1 [Sec]. The puff interval (11 [Sec]) was shorter than the CORESTA interval (27 [Sec]). Complying with the CORESTA flow rate was chosen to make it easier for other researchers to compare our results. We are, however, not suggesting either the CORESTA profile or the accelerated lifetime puffing profile used herein accurately represents human user behavior. This profile might not be suitable for other experiments which focus on different research objectives or test different devices.

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3. Results

3.1. Illustration of coil lifetime

Coil lifetime was defined as a sharp increase in coil resistance wherein the coil melts or disconnects. Verification of a sharp increase in coil resistance as a measure of coil lifetime was demonstrated by dissecting and inspecting three Vuse ALTO pods with different levels of usage. Figure 2 shows pictures of Vuse ALTO coils with three different conditions: New coil, Pre-Failed, and Failed. The ALTO coils are 'S' shaped metal strips on a porous ceramic wick substrate. Two metal connectors, evident as the circular area on the left and right side of each image, are mounted to the terminals of the coil, and connect the coil to the power control unit. The new coil had never been used and exhibited no sign of wear. The pre-failed coil had been used until the pod appeared visually empty of e-liquid. It was, however, a working coil with a functional resistance value. The pre-failed coil exhibited signs of erosion and oxidation especially in the lower portion of the 'S'. The failed coil had been used until its resistance value increased to ~400 [kΩ] indicating coil failure. The failed coil exhibited severe signs of wear and a complete physical break in the metal 'S' coil can be easily seen below and to the right side of the left terminal.

Figure 2: Pictures of 'S' shaped Vuse ALTO coils with three different conditions. 'New Coil' is a never used coil, 'Pre-Failed' is a coil which had been used until the pod appeared nearly empty of E-Liquid, and 'Failed' is a coil which had been used until its re resistance value increased to ~ 400KΩ.

3.2. Impact of initial coil resistance on coil lifetime and TPM yield

The first objective was to investigate the effects of variation in initial coil resistance (prior to first puff) on coil lifetime (measured as number of puffs until coil failure) and TPM yield per puff. We generated a scatter plot (not shown) of this data and conducted linear regression analysis to investigate a possible association between initial coil resistance and coil lifetime. The initial coil resistance of the pods ranged between 0.89 [Ω] and 1.14 [Ω] with sample mean (μ) = 1.02 [Ω] and standard deviation (σ) = 0.081 [Ω]. Coil resistance was relatively steady for the first 120 puffs (~ first 6 sessions). After 125 puffs, coil resistance values started to increase as some pods started to exhibit coil failure. Coil lifetime varied between pods from 135 puffs to 215 puffs with μ = 158 puffs and σ = 21.5 puffs. There was insufficient evidence to support an association between coil lifetime and initial coil resistance ($r = -0.07$, $p = 0.79$). Next, we generated a scatter plot (not shown) to investigate a possible association between initial coil resistance and initial TPM yield per puff (first session). The initial TPM yield per puff ranged from 0.0118 [g] to 0.0129 [g] with μ = 0.0123 [g] and σ = 0.0003 [g]. We found no evidence to support this relation ($r = -0.26$, $p = 0.35$).

3.3. Impact of initial pod mass on coil lifetime and TPM yield

The second objective was to investigate the effects of variation in initial pod mass on coil lifetime and TPM yield per puff. We generated scatter plots of this data and conducted linear regression analysis to investigate a possible association between initial pod mass and coil lifetime. The gross mass of the brand new pods (initial pod mass) ranged from 6.48 [g] to 6.61 [g] with μ = 6.54 [g] and σ = 0.0469 [g] while the tare mass of the pods after failure (end pod mass) ranged from 4.56 [g] to 4.67 [g] with μ = 4.61 [g] and σ = 0.0342 [g]. During the full exhaustive test, the net mass of the e-liquid consumed out of each pod ranged from 1.88 [g] to 2.00 [g] with μ = 1.93 [g] and σ = 0.035 [g]. We found insufficient evidence to correlate coil lifetime with either initial pod mass (r = 0.03, p = 0.9) or the net mass of e-liquid consumed (r = -0.2, p = 0.45). Next, we generated scatter plots (not shown) to investigate a possible association between initial pod mass and initial TPM yield per puff. We found insufficient evidence to correlate TPM yield per puff with initial gross pod mass (r = -0.23p = 0.41) or with net mass of e-liquid consumed (r = 0.08, p = 0.76).

3.4. Quantifying variation in e-liquid color

We observed trends in the results which could not be associated with either coil resistance or initial pod mass, which led us to re-examine the data from many different perspectives. The original study factors of initial coil resistance and initial pod mass focused on manufacturing variation associated with the metal coil and filling of the e-liquid reservoir. Neither factor accounted for variation in the composition of the e-liquid itself. Because the pods used for this study were not refillable, and contained nominally the same e-liquid based on product labeling, we ultimately focused on investigating the role of e-liquid color as a surrogate for investigating e-liquid composition as an 'after the fact' variable, and formed the basis for *a posteriori* objective (3) to investigate the effects of e-liquid color on coil lifetime and TPM yield per puff. This investigation proved fruitful, and results describing each of the associations are presented in sequence, laying the foundation for the implications of the results presented in the discussion.

Visual inspection of pods which were just taken out of the consumer over-packing (not exposed to light or air since packaged by the manufacturer) revealed variations in e-liquid color among pods in different blister-packs and between pods in the same blister-pack as shown in Figure 3. The pods were primitively classified as "light" or "dark" in color (prior to running experiments) by visually comparing the pods with each other. Luckily, this manual classification was performed prior to conducting any emissions trials, even though e-liquid color was not an original study objective. Out of 15 ALTO pods, 11 pods were classified as "light" while 4 were classified as "dark".

Figure 3: Nicotine flavor ALTO pods with 5.0% nicotine concentration. The two pods in same pack have e-liquids with two different colors. The left pod was classified as light color while the right pod was classified as dark color.

3.5. Impact of e-liquid color on coil lifetime

Figure 4 shows coil resistance values for each session as a function of cumulative puff count. The data points are presented as a scatter plot of coil resistance vs cumulative puff count, overlaid by a boxplot of the same data. The scatter plot employs the symbol * for light-colored e-liquid specimens and the symbol o for dark-colored specimens. The first six emissions sessions for all pod specimens consisted of 20 puffs per session. Thereafter, the operator reduced the count from 20 to 10 to 5 puffs per session as the e-liquid remaining in each pod decreased. As coils began to fail, the number of scatter points decreases as a function of cumulative puff count. As of 160 puffs, only 3 coils remained operable, while after 190 puffs only one coil remained in operation.

The scatter plot was overlaid with a boxplot in an effort to understand changes in variation over the course of coil life. The sample mean and standard deviation for the initial coil resistance of the pods in this figure was μ = 1.02 [Ω], σ = 0.081 [Ω]. Median and Mean coil resistance and variability remained steady for the first 120 puffs (~ first 6 sessions). After 125 puffs, coil resistance values started to increase as some pods exhibited coil failure, with concurrent increasing variation in coil resistance as evidenced by the broadening inter-quartile range of the boxplots. Coil lifetime of the entire sample of pods varied between pods from 135 puffs to 215 puffs. It was noticed, however, that coil lifetime of pods with light color e-liquid appeared to be consistently shorter than that of dark color e-liquid. The light color group was found to have coil lifetime of μ = 149, σ = 10.7 puffs while the dark color group had coil lifetime of μ = 185, σ = 22.7 puffs. A t-test between the two groups showed a difference of 36 puffs ($p < 0.001$), confirming an association between coil lifetime and e-liquid color.

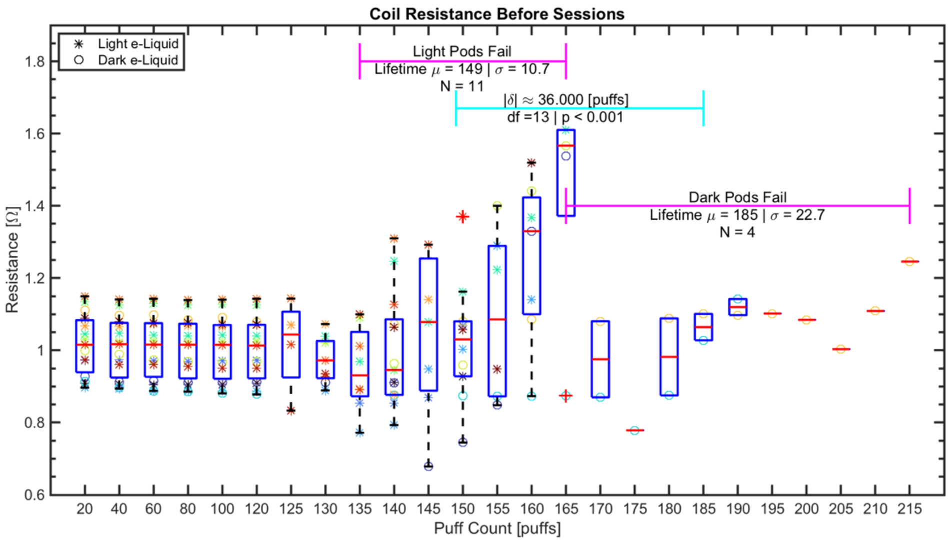

Figure 4: Coil resistance over time (no. puffs) starting with brand new full pod until failure for the N= 15 pods (N=11 light color "*", N=4 dark color "o") tested in this study. Data is represented as scatter plot where each pod is represented by a different marker color. Data is also represented with a box plot where the horizontal red marker indicates the group mean and a red plus marker indicates the corresponding data point is an outlier.

3.6. Variation in coil resistance as a function of e-liquid remaining in the pod

Figure 5 shows coil resistance values vs pod mass at each session starting from a brand-new full pod to coil failure point. The connecting lines between scatter points are used as a visual aid to show that coil resistance remains relatively steady for most of the session series, while e-liquid remained in the pods. However, coil resistance values initially decreased as the e-liquid level approached the wick and then sharply escalated, indicating coil failure, at which point the pods visually appeared completely empty. We observed the range of initial variation in pod mass (the first data point in each series) closely matched the variation in final pod mass (the last data point in each series). The cohort mean net mass of e-liquid consumed until coil failure was μ = 1.93, σ = 0.035 [g].

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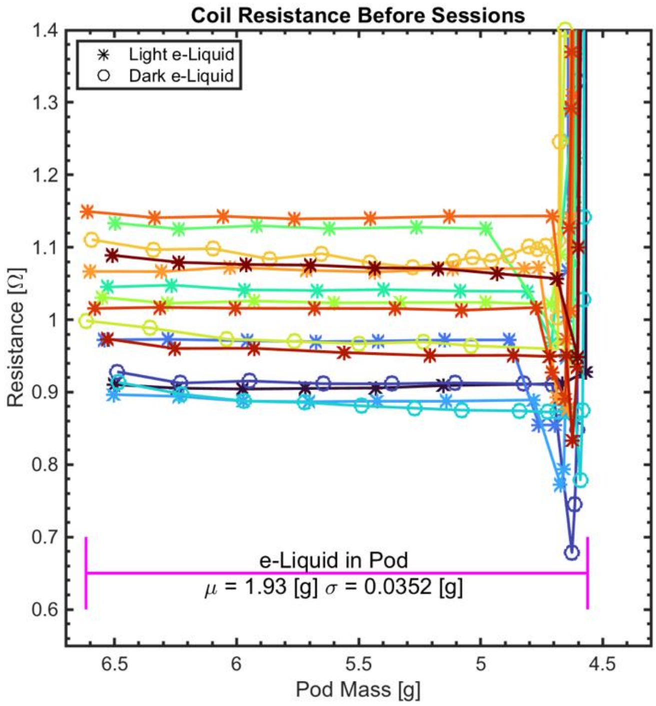

Figure 5: Coil resistance value vs pod mass before each session starting from brand new full pod until failure where the pod visually looks empty for N= 15 pods (N=11 light color, N=4 dark color) tested in this study. Each pod is represented by a different marker color.

3.7. Impact of e-liquid color on TPM yield

Figure 6 shows the TPM yield per puff vs pod mass at each session starting from a brand-new full pod to coil failure point. As expected, the TPM yield per puff approaches 0 when the e-liquid in the pod is fully or almost fully consumed. However, the pods in the light e-liquid group and the dark e-liquid group behave differently while the e-liquid is being consumed. The TPM yield per puff of the light e-liquid group is relatively steady for most of the sessions and sharply decreases when the e-liquid is fully consumed or almost consumed. On the other hand, for the dark e-liquid pods, the TPM yield per puff gradually (linearly) decreases while more e-liquid is being consumed until it sharply decreases just before the e-liquid is fully consumed.

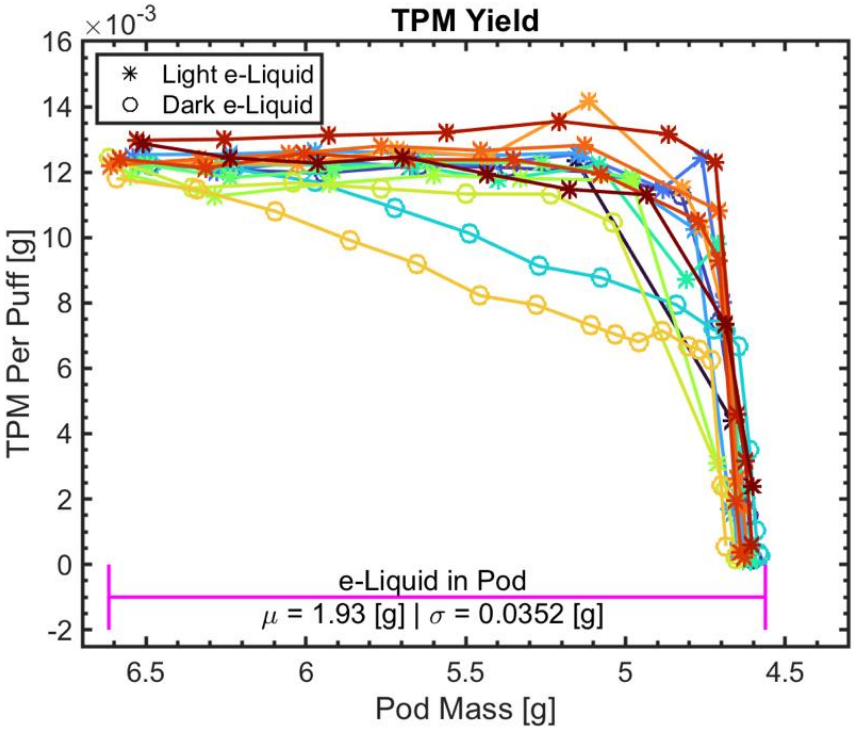

Figure 6: TPM yield per puff vs pod mass for each session starting from brand new full pods until coil failure where the pod visually looks empty for N= 15 pods (N=11 light color, N=4 dark color) tested in this study. Each pod is represented by a different marker color.

4. Discussion

4.1 Oxidation of nicotine may explain the results observed.

Prior studies point to oxidation as a cause for color changes observed in e-liquid. Kim et. al. [39] suggested the darkness in the e-liquid could be caused by oxidation of nicotine. Their custom-made e-liquids exhibited a change in color from clear to yellow in 7 days after manufacturing. Kim et. al. reported the change in the color continued to reach 2 and 16 folds darker compared to the original clear color after 1 and 6 months, respectively. Older studies by Wada et. al. [40] and Linnell [41] provided experimental results which suggest a connection between changing nicotine color and nicotine oxidation. Linnell showed that the nicotine turned from water-clear color to yellow a few hours after nicotine is exposed to air. Wada et. al [40] showed that 20% of the nicotine was oxidized and turned to dark brown after 4 weeks of exposure to air. Wada concluded that the oxidation process of nicotine produced a considerable amount of nicotyrine among other materials. Also, online e-cigarette communities have presented an aging process of e-liquid called “steeping” [42]; suggesting it as a method to smooth the flavor taste of e-liquid. This process is associated with nicotine oxidation. Interestingly, these articles suggest monitoring e-liquid color as an indicator of progress of the “steeping” process. Turning e-liquid color from light (or transparent) to dark was presented as a sign of successful “steeping”. A relatively modern study by Martinez et. al. [43] also connected nicotine aging and production of nicotyrine. Their results showed a measurable increase in the nicotyrine to nicotine ratio (NNR) two weeks after opening the e-liquid container. NNR continued to increase for the full time of the experiment of 65 days. Etter et. al. [44] mentioned that nicotyrine in addition to other materials are products of oxidative degradation of nicotine. In addition to liquid phase, nicotyrine could be a product of a vapor phase oxidation of nicotine as mentioned by Clayton et. al. [45] and Woodward et. al [46].

Other byproducts have been reported to be formed during the oxidation of nicotine such as nicotinic acid, oxynicotine, cotinine, and mysomine [40]. In addition to nicotine, aging and oxidation could affect other compounds in the e-liquid such as PG, GL, flavoring, and other additives. Oxidation or degradation of all of these components could form a variety of other byproducts which change the pharmacological and physiochemical properties of e-liquid [39]. The next paragraph presents an example of the effects of oxidation byproducts on the aerosol generation and TPM.

Based on this literature, it is reasonable to assume dark color e-liquid pods may have had a higher NNR compared to the light color e-liquid pods. Further, this might provide a mechanistic explanation of both the observed variation in TPM yield per puff (*Figure 6*) and the between groups difference in coil lifetime (Figure 4). The saturation temperature of nicotine is 247.0 °C [47] while the saturation temperature of nicotyrine is 281.0 °C [48]. Not only does this mean nicotyrine vaporizes at higher temperatures, it also indicates that e-liquids with higher NNR would have lower TPM yield compared to those with lower NNR, particularly if the coil temperature is above 247 C and below 281 C. Just as the ratio of propylene glycol to glycerin changes the effective saturation temperature of the e-liquid solvent, so too will an increase in NNR increase the effective saturation temperature. And, as the effective saturation temperature increases, the yield per puff will decrease and the number of puffs required to empty the pod, expose the coil, and induce failure will increase. For this reason, and if all other factors are held constant, we should expect pods with dark color e-liquid to exhibit lower TPM and longer coil lifetime than pods with light color e-liquid, consistent with the results reported in our study.

4.2. Oxidation of nicotine may have important public health and cessation implications.

A limited amount of prior work [49] has suggested that nicotyrine may be metabolized in humans more slowly than nicotine, and could potentially mitigate symptoms of withdrawal and craving. If future research bears this out, then managing the NNR of e-liquid may be one important factor in formulating e-liquids for use as a cessation tool. A clinical study which investigates the joint effect of increased NNR in conjunction with reduction in nicotine concentration on subjective effects of craving and cumulative TPM exposure could provide insight into both cessation studies and prevention of unintended consequences (such as increasing cumulative TPM exposure) in response to regulatory actions (such as decreasing permissible e-liquid nicotine concentration).

4.3. Why didn't initial coil resistance affect TPM yield?

The results presented herein demonstrated insufficient evidence to support correlations between initial coil resistance with either coil lifetime or TPM yield per puff. If an ENDS PCU simply applied a voltage across the coil terminals (by short-circuiting the battery across the coil) then the power of a freshly charged lithium battery should vary inversely with the coil resistance in accordance with Ohm's Law. Furthermore, the TPM yield per puff should increase with power dissipated in the coil, based on first principles of heat and mass transfer. We did not observe this correlation between coil resistance and TPM yield. This suggests that some ENDS PCU may employ algorithms to overcome variation in coil resistance or actively control the power (either through current or voltage manipulation). Such algorithms could use a closed-loop control system which dynamically measures coil resistance and adjusts the power supplied to the coil in real time in order to keep the heating energy within a limit [50]. Thus, the temperature of the coil and heating chamber could be controlled in a cycle to keep aerosol emission steady. No articles have been presented in the literature which assess how effective such PCU methods are and whether they can completely eliminate the effects of coil resistance variation on the performance of the ENDS. The results presented herein establish a firm premise for the study of PCU control algorithms. Such PCU algorithms offer potential for both positive and negative health effects, and thus are worthy of detailed investigation and possible regulatory action.

4.4. What are the mechanisms of coil failure?

The results demonstrate that a dramatic increase coil resistance is a sufficient indication of coil failure. At failure point, the coil melts or breaks leaving an open circuit between its terminals. This break is reflected as a sudden increase in measured coil resistance from order of ~1 [Ω] to order of

~400 [KΩ]. While our definition of coil failure suggests coil break, our measured coil resistance was not infinite. The residual resistance measured after the failure point could be related to the resistance of the wick and some drops of e-liquid that might still exist around the wick. The results presented herein may explain the mechanisms underlying coil failure. When the coil is active, it generates heat that is transferred to the e-liquid causing it to vaporize. Concurrently, the e-liquid cools the coil as generated aerosol carries the heat away. This suggests that the presence of e-liquid around the coil contributes to limiting the coil temperature below its melting point, consistent with the observed steadiness in coil resistance values while there is e-liquid left in the pod (Figure 5). When insufficient e-liquid remains to fully submerge the coil and the wick, the heat generated by the coil remains in the coil and the wick causing the coil temperature to increase and thus the coil melts or breaks. A follow-up experiment could be conducted to confirm this explanation by demonstrating the coil lifetime can be extended indefinitely by refilling the e-liquid reservoir, even though the pods studied here are intended by the manufacturer to be disposable. We observed the ENDS continued to operate during this pre-failure condition, when the coil was degraded (Figure 2, middle image) and loss of metal was observed.

4.5. Potential health impact of coil failure and product misuse.

Metal is likely ejected from the pod into the aerosol while the coil fails during the final puffing session and potentially much earlier. This is a potentially critical juncture, particularly in the instance where consumers misuse their product and refill the e-liquid in a pod which was nearly emptied on a prior use and whose coil had experienced degradation. When a compromised coil is subsequently heated, there may be increased risk of metal exposure even when the coil has not fully failed. Follow-up experiments to confirm the extension of coil lifetime are recommended, and, if successful, the presence of metal contaminants in the resulting aerosol may be investigated.

4.6. Understanding a potential mechanism for why e-liquid color changes

The nicotine could oxidize under different circumstances. For example, the nicotine in the e-liquid could oxidize during the manufacturing of the original ingredient or during production of the e-liquid which results in some unstable formula [44]. The oxidation could be a result of unexpected interaction between the e-liquid and the packaging material or due to improper handling and storage process [43,51]. Some storage environments or prolong storage could also expedite the oxidation process or the nicotine and hence change e-liquid color[42,43]. Structured investigations or formal studies of the correlation between color change and elapsed time are needed.

4.7. Limitations and Future work

Investigating the color of e-liquid was not intended as an original objective of this study. For this reason, there was an unbalanced number of pods between the light e-liquid color group and dark e-liquid color group. The pods could have been divided into more than two categories based on e-liquid color. Also, a spectrophotometer could have been used to classify e-liquids. A thread of conclusions from previous works has been yarned to explain the variation in e-liquid color and the consequence chemical constituent differences. In future studies a GC-MS analysis is suggested for the analysis of e-liquid and measurement of NNR and other e-liquid constituents. A formal experiment to address these issues is under way now and will be reported in a future article.

5. Conclusions

The amount of e-liquid remaining in the pod appears to be the single most important factor in determining coil failure. Oxidation of nicotine into nicotyrine and its role in e-cigarette emissions and public health should be further investigated. A dramatic sharp increase in observed coil resistance is a robust method for quantifying coil lifetime. Further investigation is needed to assess the potential adverse health impacts of coil degradation during the final stage of coil lifetime.

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