Direct Single Puff GC-MS Analysis of Electronic Cigarettes and the Effect of Inter-puff Interval on Relative Nicotine Delivery

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Abstract

As electronic cigarettes (e-cigarettes) have become more popular, research labs have begun to propose standardized vaping protocols to produce machine-generated aerosols from e-cigarette products. Investigations have largely focused on the effects of puff volume and duration on total particulate matter (TPM) deliveries, but little has been done to investigate the effects of the inter-puff interval when testing these products. In this study, disposable, rechargeable, and tank systems were evaluated on a puff-by-puff basis using direct gas chromatography-mass spectrometry (GC-MS) analysis of each puff. A 55-mL puff volume and 3-second puff duration was used for each puff, which was captured in individual sample loops for direct GC-MS analysis. The relative abundance of nicotine was evaluated for each puff at increasing inter-puff intervals (30 sec to 5 min) to determine the relative nicotine emissions, as well as to evaluate e-cigarette performance over the vaping session. The e-cigarette aerosol was also evaluated using traditional methods in which TPM is collected on a Cambridge filter pad, and nicotine extracted and measured using GC-flame ionization detection (FID). For all products tested, the largest nicotine delivery was observed in the first few puffs, with a rapid decrease as the number of puffs increased. When inter-puff intervals were extended beyond 30 seconds, the relative nicotine delivery increased with increasing time interval until a plateau was observed after four minutes. These results suggest that the inter-puff interval may significantly influence the delivery of nicotine for e-cigarette products. E-cigarettes may require a "recovery period" in order to produce more consistent puff-to-puff nicotine delivery during machine testing.

Experimental

Single Puff Experiments: Relative nicotine delivery was evaluated on a puff-by-puff basis using direct injection of each puff onto a GC-MS system (Wasson ECE, Fort Collins, CO, Figure 1) equipped with an EI source (230 ˚C) and run in scan mode with a 5:1 inlet split ratio. The inlet was kept at 225 ˚C. Chromatographic separation was achieved on a Zebron Inferno (Phenomenex, Torrance, CA) ZB-5HT column (30 m x 25 mm x 0.25 µm) with a helium flow of 1.8 mL/min. The GC oven ramp conditions were as follows: 40 ˚C hold 1 min, 8 ˚C/min to 185 ˚C, 15 ˚C/min to 325 ˚C, hold 2 min; total run time: 30.5 min. The MS transfer line was held at 255 ˚C, sample loops were held at 230 ˚C, and the interface bridge was held at 250 ˚C. Inter-puff intervals were 30 sec, 60 sec, 2 min, 3 min, 4 min, and 5 min. Puffs were taken using a new or fully charged device for the first 10 puffs. The subsequent 10 puffs used the same product without alteration. Comparison of relative nicotine delivery was achieved by overlaying total ion chromatograms to assess the relative nicotine level in each puff. Blank puffs were run after analysis to ensure the loops did not contain residual e-liquid solvents.

Vaping Experiments: Five replicates (N=5) of the same products were evaluated on a Borgwaldt LX5.1 e-cigarette smoking machine. Nicotine levels in the products were evaluated *via* extraction from Cambridge filter pads for each 25-puff segment collected under the puff regimes outlined above using an Agilent (Dover, DE) GC-FID as described previously with minor modifications.[1] Total particulate matter (TPM) was determined by subtracting the mass difference between the unused filter pad and the used filter pad.

Samples: One disposable "cigalike", one rechargeable "cigalike," and one tank system (3.2V) was evaluated (N=1). The e-liquid used was menthol flavored and contained 1.8% nicotine (labeled concentration) as well as water and propylene glycol.

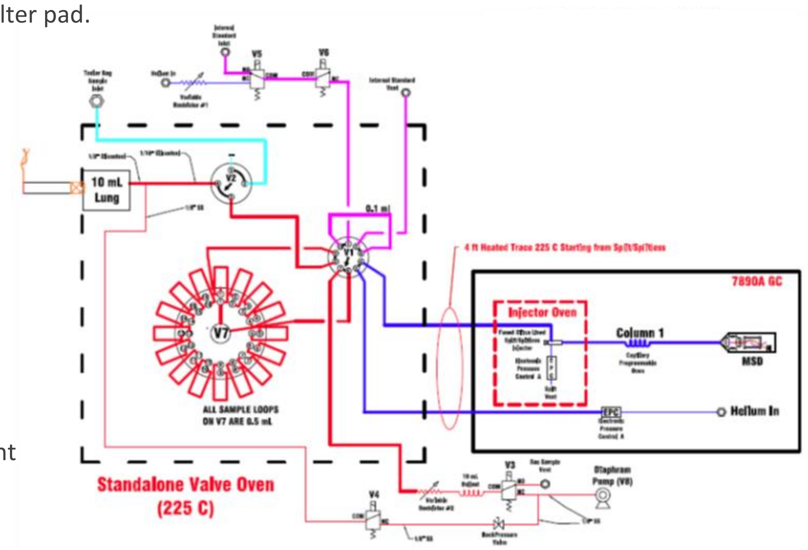


Figure 1. Schematic diagram of the instrument used for direct single puff GC-MS analysis.

Results

Single Puff Experiments

Figure 2. An example of an overlaid Total Ion Chromatograms (TIC) of puffs 1-10 of the nicotine peak at 55 mL/3 sec/120 sec smoking regime for each product type.

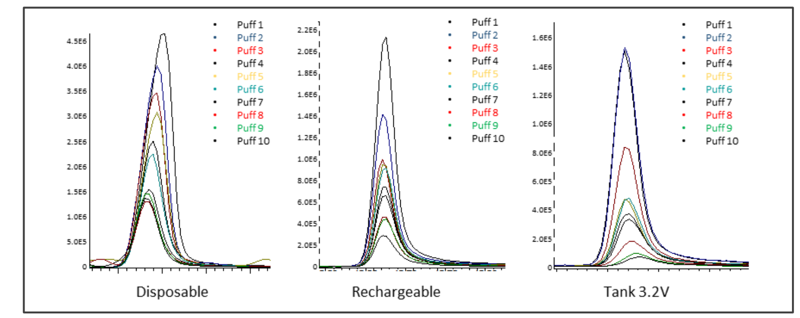
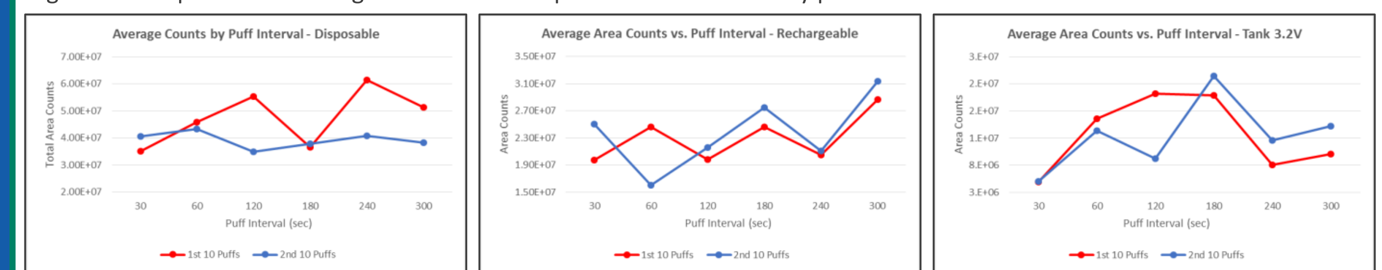


Figure 3. Comparison of average area counts for puffs 1-10 and 11-20 by puff interval.



Vaping Experiments

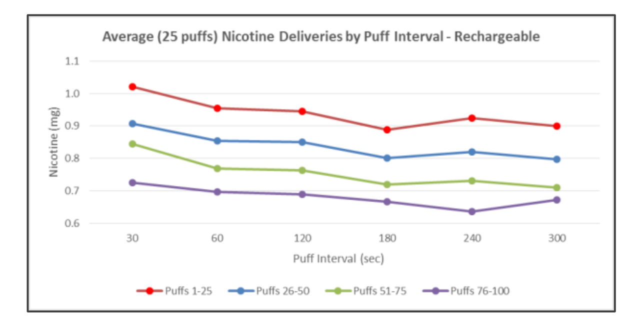


Figure 4. The plot above illustrates the average nicotine deliveries (N=5) at four 25-puff segments (100 total puffs per e-cigarette) at each puff interval (represented here using the rechargeable product type; the disposable product behaved similarly, while the tank product data was highly variable). Errors bars have been excluded for clarity. A relatively comparable decrease in average nicotine deliveries was observed with each subsequent 25-puff segment, regardless of the puff interval.

Conclusions and Limitations

- Initial puffs from an e-cigarette contain the highest levels of nicotine. Nicotine delivery decreases over time during an individual puffing session (Figure 2).
- As shown in Figure 3, the relative nicotine delivery increases as puff interval increases with time for the 1st 10 puffs of the disposable product. The relative nicotine delivery was much more consistent in puffs 11-20 for the disposable product.
- The rechargeable product tested showed consistent nicotine delivery at all puff intervals. Comparison of the 1st 10 puffs and the 2nd 10 puffs showed approximately the same relative nicotine delivery (Figure 3).
- The tank system (3.2V) produced increasing relative nicotine deliveries as puff intervals increased to a plateau at 3 minutes (Figure 3).
- Puff interval did not have an effect on nicotine concentration after analysis of the Cambridge filter pad after 25 puff segments (Figure 4). Further investigation of smaller puff segments (i.e. 5 puffs, 10 puffs) are currently underway.
- The data shown is for only one representative product and one replicate for each category for single puff experiments. Additional products need to be tested in order to examine the generalizability of the trends seen in the products above.
- Product performance variability needs to be monitored and minimized in order to strengthen the impact of the observations seen above.

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References: 1. Calafat *et. al*. Determination of tar, nicotine, and carbon monoxide yields in the mainstream smoke of selected international cigarettes. Tobacco Control, **2004**; 13: 45:51.

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