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A Direct Method for E-Cigarette Aerosol Sample Collection

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Abstract

E-cigarette use is increasing in populations around the world. Recent evidence has shown that the aerosol produced by e-cigarettes can contain a variety of toxicants. Published studies characterizing toxicants in e-cigarette aerosol have relied on filters, impingers or sorbent tubes, which are methods that require diluting or extracting the sample in a solution during collection. We have developed a collection system that directly condenses e-cigarette aerosol samples for chemical and toxicological analyses. The collection system consists of several cut pipette tips connected with short pieces of tubing. The pipette tip-based collection system can be connected to a peristaltic pump, a vacuum pump, or directly to an e-cigarette user for the e-cigarette aerosol to flow through the system. The pipette tip-based system condenses the aerosol produced by the e-cigarette and collects a liquid sample that is ready for analysis without the need of intermediate extraction solutions. We tested a total of 20 e-cigarettes from 5 different brands commercially available in Maryland. The pipette tip-based collection system condensed between 0.23 and 0.53 mL of post-vaped e-liquid after 150 puffs. The proposed method is highly adaptable, can be used during field work and in experimental settings, and allows collecting aerosol samples from a wide variety of e-cigarette devices, yielding a condensate of the likely exact substance that is being delivered to the lungs.

Keywords

E-cigarettes; aerosol; sampling; method

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1. Introduction

Over the last few years, the use of e-cigarettes has been growing fast in many countries around the world (McMillen et al., 2015). These nicotine delivering devices have emerged as an attractive alternative to traditional cigarettes, since they are perceived as a less hazardous product and can be used as an aid to quit smoking (Amrock et al., 2015; McRobbie et al., 2014), although their efficacy as a smoking cessation tool is still unclear (Drummond and Upson, 2014). Concerns, however, have been raised regarding the safety and chronic toxicity of these products. E-cigarette refill liquids are generally made of a mixture of glycerol, propylene glycol, nicotine, and other ingredients in different proportions, depending on the brand (Allen et al., 2015). Hazardous chemicals such as toxic carbonyl compounds (acetaldehyde, formaldehyde and acrolein) have been detected in e-cigarette emissions (Geiss et al., 2015; Kosmider et al., 2014). Other toxicants of concern include metals, which could be transferred from the heating coil to the e-cigarette liquid (Hess et al., 2016) and the vapor (Saffari et al., 2014).

The collection of aerosol samples, either in vapor form or condensed back to liquid form, can be challenging. Published studies, summarized in Table 1, have collected a sample of the aerosol generated by vaping using a variety of sampling devices including filters, impingers, sorbent tubes or flasks. These sampling systems require the aerosol to be directly diluted or later extracted into solution (Figure 1), adding a step to the sample preparation before analysis, a process which could alter the originally collected sample.

Our objective was to develop a method for collecting e-cigarette aerosol that can directly condense the aerosol sample without requiring dilution/extraction. Through trial and error and after testing different methods, including passing the e-cigarette aerosol through a refrigerated condenser or a piece of tubing in dry ice (methods which were not successful as the dry ice was too cold, directly freezing the aerosol and blocking the tubing, and other milder refrigerators could not condense sufficient aerosol, even when narrow 1.5 mm internal diameter tubing was used), we have developed a novel method for collection of e-cigarette aerosol that is fast, inexpensive, and can be easily adapted with a wide variety of e-cigarette devices. The present manuscript describes the method, which directly condenses the aerosol produced by the e-cigarette and collects a liquid sample that is ready for chemical analysis.

2. Material and methods

2.1. Sampling method description

We have developed an e-cigarette vapor sample collection system with common laboratory consumables. This sampling system (Figures 2 and 3) consists of four 250 μ L pipette tips (Supersilk, Labcon. Petaluma, CA, USA), three of which are cut so they are 2 cm long (conserving only the narrow tip part); and four sections of Tygon tubing (0.0600" (1.5mm) internal diameter, S3 E-3603, Saint-Gobain Corporation, France), three of them cut to 2 cm each and one cut to 5 cm, and a 1.5 mL centrifuge tube (Labcon North America, Petaluma, CA, USA). The e-cigarette aerosol can be generated using a peristaltic pump (drive no. 07522-20 and head no. 77200-62, Cole-Parmer, Vernon Hills, IL, USA), which drives the

aerosol from the e-cigarette using a 16 cm tubing piece (4.8 mm internal diameter, Masterflex L/S 15, Vernon Hills, IL, USA) which connects the e-cigarette to the uncut pipette tip.

The uncut pipette tip serves as a funnel to drive the vapor through a sequence of three 2 cm tubing sections assembled manually and connected to each other with the cut pipette tips, with the tip exit in the direction of the vapor flow. The last tubing section (5 cm) serves to drive the condensed aerosol (liquid sample) to the sample storage recipient, which can be a 1.5 mL micro-centrifuge tube or any type of recipient. This sample recipient can be connected to the tubing using Parafilm M (American National Can, Chicago, IL, USA) or any other material. Regardless of the material used to connect the last tubing section to the sample storage recipient, a venting groove is needed to let air escape from the tube.

The pipette tip-based e-cigarette aerosol collection system can also be modified to be used with an air vacuum pump (Figure 4). The vacuum pump is placed after the collecting vessel and pulls the liquid from the e-cigarette through the system using a “Y” or a “T” connector. A longer final tubing section (8 cm instead of 5 cm) is needed to direct the collected liquid to the bottom of the collection vessel through the “Y” connector.

The number, material, diameter, and length of the pipette tips and tubing sections as well as the puffing topography (the flow rate, and the puff and inter-puff times) can be changed to optimize the volume of sample collected, making this method highly adaptable.

2.2. Aerosol collection

The puffed e-cigarette aerosol condenses as it flows through the system; this liquid is pushed to the sample storage recipient by the aerosol flow of each puff. We speculate that the pipette tip-tube iterations produce a funnel effect that together with a reduction in the speed of the aerosol contribute to the merging of the droplets, facilitating their condensation. Therefore, this system allows for the collection of a liquid sample directly from the e-cigarette aerosol (vapor). The peristaltic pump was programmed to operate at a flow rate of 1.0 L/min and a puff topography of four second puffs, with 30 second inter-puff time, for 150 puffs, which is similar to the topography of an average experienced e-cigarette slow user as described by Talih et al. (2015a). Due to the viscosity of the liquid, some droplets remained in the 16 cm tubing section after the 150 puffs. In order to collect this liquid, the 16 cm tubing was held upright, and manually “flicked” until the droplets could be seen in the 2 cm tubing. The system was then placed back into the pump without the e-cigarette attached, and set for an additional five puffs, with the same topography as described above to recover these droplets.

2.3. Method yield estimation

To estimate method yield and compare yield between e-cigarette brands, we tested five different brands of cig-a-like type e-cigarettes commercially available in Maryland (Blu, Logic, Vuse, Fin and Mistic). To obtain the total weight and volume of e-liquid available before vaping for the five brands tested, three cartridges (cartomizers) of each brand were disassembled using standard pliers. The pad inside, free of the heating coil, was pulled using forceps. This pad, which is assumed to contain 100% of the e-cigarette liquid contained in each cartridge, was centrifuged at 4000 rpm during 10 minutes to extract the e-cigarette

liquid in them. The total sample collected after 150 puffs was also weighed and its volume measured. According to our observations, 150 puffs were enough to completely consume the e-cigarette liquid as the devices tested were not able to generate more aerosol. The volumes of pre-vaped juice and final condensed vapor were difficult to measure with precision due to their high viscosity and hence volumes reported should be taken as approximations. We could not extract the liquid of the Blu cartridge pads due to the characteristics of the Blu e-cigarette cartomizers. The total weight and volume of e-liquid before vaping is thus not available for Blu e-cigarettes.

2.4. Safety considerations

The pipette tip-based e-cigarette collection system has been set up inside a fume hood to avoid the exposure to the generated aerosol and the contamination of the laboratory.

3. Results

The sampling system described in this article was successfully used to collect between 0.25 and 0.53 mL (mean 0.32 mL, standard deviation 0.12 mL) of the condensed aerosol produced by five e-cigarette brands after 150 puffs (Table 2). We vaped 4 to 5 cartridges (cartomizers) of Blu, Logic, Vuse, Fin, without any issues. For Mystic, a high proportion of the cartridges generated a very reduced amount of vapor and were stopped before reaching 150 puffs. After using 10 cartridges, we were able to complete the experiment for two cartridges. Thus, we sampled a total of 20 e-cigarettes cartridges of 5 different brands.

The overall mean percentage of the aerosol weight recovered after 150 puffs was 40.8% of the originally available liquid, ranging from 30.6% to 51.9% (Table 2). This number could not be estimated for Blu e-cigarettes as the e-liquid pre-vaping could not be extracted. In all the brands tested, we obtained sample weights ranging on average from 0.255 g to 0.561 g (0.23 mL–0.53 mL) of condensed e-cigarette aerosol (0.348 ± 0.123 g, 0.32 ± 0.12 mL). This condensed aerosol sample can be later prepared according to the analytical technique to be employed, for instance with a known dilution factor using an appropriate solution.

In addition to cig-a-like e-cigarette cartridges, the proposed aerosol collection method has been successfully employed with refillable (tank-style) e-cigarette devices with the only limitation that the button-activated devices have to be activated right before every puff which is not necessary for cig-a-like devices since they are activated directly by the puffing action. Thus, an operator needs to be present for the collection of tank-like devices, making the procedure slightly more cumbersome. On the other hand, 0.25 mL were obtained after only 40 puffs (at 4 s puffs, 10 s intervals and 1.0 L/min flow rate) with the tank-like device tested. The proposed method can also be employed during fieldwork to condense the e-cigarette aerosol puffed by a participant by blowing the puffed aerosol directly through the collection system. Therefore, this simple, inexpensive and effective e-cigarette aerosol collection system can be easily adapted to any kind of e-cigarette devices and puffing systems (peristaltic pump, air vacuum pump or human puffing).

4. Discussion

Our pipette tip-based aerosol collection system directly condenses aerosol samples and provides an alternative to currently used methods for e-cigarette aerosol sample collection. The intermediate solution used to collect/extract the aerosol in previous methods dilutes and can potentially modify the chemicals in the original aerosol sample. By not mixing it with other chemicals, the direct method collects a sample that is more likely to reflect what e-cigarette users are inhaling (Figure 1). Second, the collection/extraction solution used in indirect methods is generally specific for the type of analyte that is intended to be measured. The direct method, on the other hand, provides a sample that can be used for measuring many types of analytes. This may be an efficient advantage compared to methods using analyte-specific collection/extraction solutions.

The direct method described in this manuscript does not collect the total amount of aerosol generated by the e-cigarette. However, we could easily estimate the sampling yield (amount of the aerosol emitted by the e-cigarette that was collected). This is an advantage compared to published studies using indirect methods, which generally report no information on yield. Information on yield is important to evaluate the performance of the method and to interpret the data obtained and should be required in all studies of e-cigarette analyses of aerosol samples. Part of the aerosol emitted that is not recovered by the direct method could be particularly important for chemicals in the gas phase such as formaldehyde and other volatile organic compounds. This could also be a limitation in other indirect methods unless they specifically attempt to trap chemicals in the gas phase. Another limitation of published studies using indirect methods is that most of them provide the concentrations as mass of chemical per number of puffs (see Table 1). Other units used are mass of analyte per e-cigarette or per filter. Comparison across studies is thus difficult as the type of puff is not standardized and the analyte concentration per puff can markedly vary by the vaping topography used to generate the puffs. The reporting of per puff, per e-cigarette or per filter units should be replaced by real concentration units such as mass of analyte per volume of air (gas) or volume of condensed aerosol (liquid). The direct method reported here directly provides results in concentration units (μg of analyte/L of condensate). These liquid concentration units allow the comparison of the results between studies and additionally enable to compare the e-cigarette liquid before (liquid in the cartridge) and after vaping (condensed generated aerosol), (pre and post-vaped).

A major advantage of the direct method presented in this manuscript is its low cost. The sampling device is made with common laboratory consumables: pipette tips, tubing pieces, parafilm and a micro-centrifuge tube. The estimated cost of a sampling device is 6 dollars per unit. The time needed to assemble each unit is less than 5 minutes. Sample collection is also achieved in only 15 to 20 minutes (depending on the volume desired and the vaping device). Both direct and indirect methods require an air pump to collect the sample, and our direct method has the additional advantage of allowing to collect directly by an e-cigarette user and to be used during fieldwork. Although sorbent tubes and filters also cost a few dollars each, and the cost of the desorbing chemicals is low, our method is the most cost-effective in the collection and extraction time, since the only time required is the time to operate the pump to condense the aerosol, whereas indirect methods involve the time to

collect the sample plus the time spent extracting/desorbing from the collection substrate. In Table 3, our direct method is compared with indirect methods in terms of cost and time.

5. Conclusions

We have developed a method that directly condenses e-cigarette aerosol samples using common laboratory consumables including pipette tips and tubing sections. This pipette tip-based collection system yields a condensate that likely reflects the vaped sample that is delivered to the lungs. The e-cigarette vaped sample is collected in its original form, as it is inhaled by the user, without an additional extracting step. The number, material, diameter and length of the pipette tips and the tubing sections as well as the puffing topography (flow rate and the puff and inter-puff times) can be changed to optimize the volume of sample collected, contributing to its high adaptability to many kinds of e-cigarette devices. Although future research is needed to compare the performance across methods, by allowing the direct collection of e-cigarette aerosol samples without the use of collection/extraction solutions, this method constitutes an efficient and low-cost alternative to currently existing e-cigarette aerosol sampling systems.

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Highlights

- E-cigarette toxicant evaluation is limited by difficulty to directly collect aerosol.
- Methods used so far require using an extraction solution which could modify sample.
- Our novel method for e-cigarette directly condenses the e-cigarette aerosol sample.
- Our method is fast, inexpensive and highly adaptable to multiple e-cigarette devices.

A. Indirect Method Based on Collection/Extraction Solution (e.g. filters, impingers, sorbent tubes and bottles):

- Intermediate solution needed to collect/extract the aerosol.
- Solution is generally specific to the intended type of analyte.
- Results are mainly reported by number of puffs.
- Time-consuming.
- Requires highly trained personnel to collect the sample.

B. Direct Method (Connected pipette tips that condense the aerosol):

- Sample is directly condensed from the generated vapor (no intermediate solution).
- Sample can be used to measure any type of analytes.
- Amount of collected and uncollected aerosol can be quantified easily.
- Cheap and fast.
- Little training is needed to collect the sample.

Figure 1.

Comparison of indirect methods to evaluate chemicals in e-cigarette aerosol, based on using a solution to collect or extract the aerosol sample, to our proposed method, which directly condenses the aerosol sample without an intermediate solution.

Figure 2.
Drawing of the e-cigarette vapor sampling system using a peristaltic pump.

Figure 3. Pictures of the e-cigarette vapor sampling system using a peristaltic pump (Cole-Parmer). 2.a. Front view of the whole e-cigarette sampling system. 2.b. Detailed view of the vapor condensation and the sample collection.

Figure 4.
Drawing of the e-cigarette vapor sampling system using an air vacuum pump and a “Y” connector.

Table 1

Methods used for e-cigarette vapor sample collection in some recent articles, and units used to report results.

Method	Substance/s for Sample Collection / Extraction	Analyte/s	Result Units	Reference/s
Filters	Ethyl acetate	Nicotine	mg/15 puffs	Talih et al. (2015a)
	Methanol	Nicotine	mg/e-cig	Pagano et al. (2015)
	i-propanol	Nicotine, propylene glycol and glycerol.	mg/filter	Geiss et al. (2015)
	Nitric and hydrochloric acids	Metals	mg/99 puffs	Tayyarah and Long (2014)
Impingers	DNPH and acetonitrile	Carbonyl compounds	µg/10 puffs	Farsalinos et al. (2015)
	DNPH	Carbonyl compounds	mg/99 puffs	Tayyarah et al. (2014)
Sorbent tubes or silica cartridges	DNPH and acetonitrile	Carbonyl compounds	µg/15 puffs	Talih et al. (2015b)
			ng/puff	Geiss et al. (2015)
			ng/15 puffs	Kosmider et al. (2014)
	Hydroquinone and DNPH; Acetonitrile in phosphoric acid.	Carbonyl compounds	mg/m ³	Uchiyama et al. (2013)
Bottles or flasks	PFBHA in a ethyl alcohol and water solution	Diacetyl, 2,3-Pentanedione, and Acetoin.	µg/e-cig	Allen et al., 2015
	DNPH and acetonitrile	Carbonyl compounds	µg/puff	Hutzler et al. (2014)
	Methanol with quinoline	Nicotine	mg/150 or 300 puffs	Goniewicz et al. (2014a)
	Nitric acid, hydrochloric acid and deionized water	Elements	µg/10 puffs	Williams et al. (2013)
	Methanol	Metals and	µg/150 puffs	Goniewicz et al. (2014b)

DNPH: 2,4-dinitrophenylhydrazine

PFBHA: O-(2, 3, 4, 5, 6-pentafluorobenzyl) hydroxylamine hydrochloride

Table 2

Means and ranges of total e-cigarette liquid available before vaping, sample collected after 150 puffs, and percentage recovered for each e-cigarette brand tested after 150 puffs.

E-cigarette brand	Mean (range) total e-cigarette liquid available before vaping*	Mean (range) sample collected after 150 puffs**	Mean percentage recovered after 150 puffs
	g	g	% (weight)
Blu	-	0.289 (0.236-0.314)	0.25 (0.23-0.26)
Logic	1.301 (1.260-1.353)	0.561 (0.481-0.617)	0.53 (0.49-0.58)
Vuse	0.492 (0.484-0.506)	0.255 (0.226-0.281)	0.23 (0.20-0.25)
Fin	0.915 (0.890-0.937)	0.344 (0.183-0.416)	0.35 (0.20-0.40)
Mistic	0.955 (0.927-0.976)	0.292 (0.278-0.306)	0.25 (0.25-0.25)
All (Mean±SD)	0.916±0.331	0.348±0.123	0.32±0.12
			40.8±9.0

SD: Standard Deviation.

* For each of the brands tested n=3, except for Blu which e-cigarette liquid available before vaping could not be obtained as the pad could not be extracted from the cartridge.

** Blu n=5, Logic n=5, Vuse n=4, Fin n=4 and Mistic n=2.

Table 3

Time and cost comparison between our direct method and the indirect methods commonly found in the literature.

	Our direct method	Indirect methods		
		Filters	Impingers / bottles	Sorbent tubes
Cost of materials	\$6	\$1 - \$5	\$100 - 150	\$1 - \$10
Time to assemble sampling train	5 min		10 - 15 min	
Time to collect sample	20 min		Minimum 20 min *	
Time to extract sample	none		15 – 120 minutes	

*
Depends on concentration and sampling method.