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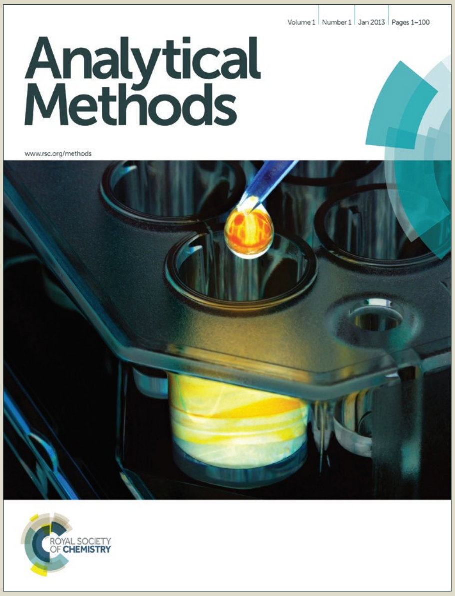

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Mass change tracking approach as collection guidelines for aerosol and vapor samples released during e-cigarette smoking

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Abstract

As the popularity of electronic cigarettes (EC) rise, there are growing concerns not only about the lack of methodological basis for the quantification of various pollutants but also the absence of proper regulations or guidelines for its production and distribution. Considering the urgent need for such guidelines, this study was carried out to explore the potent role of key sampling variables in the quantitation of EC-produced aerosols/vapors: the sampling flow rate (or puff velocity), battery charge condition, and solution composition. All of these variables were evaluated in relation to the consumption rate of EC solution. Accordingly, the effects of all these variables were explained very effectively when bound with the information regarding the consumption rate of EC solution, namely mass change tracking (MCT) approach. As such, MCT is regarded as the vital component to reduce considerable uncertainties associated with the use of a simple criterion like pollutant mass per puff.

Keywords: Electronic cigarette; aerosol; carbonyl compounds; hazards; consumption rate

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1. Introduction

The electronic nicotine delivery system (ENDS), commonly called electronic cigarettes (EC) is a battery-powered device that generates vapor and/or smoke to facilitate the delivery of nicotine or other chemicals to its consumers.[1-6] The marketing of EC has grown rapidly as a healthier and/or more reliable alternative to conventional cigarettes (CC), but suspicion of such claims has also been rising among the public.[7,8] Many researchers began to focus more intensively on adverse effects of EC (not only of CC) emissions on public health after legal ban of smoking CC in public areas of many European countries.[7,8] As the potent role of EC as an alternative to CC is being questioned, it is important to accurately quantify the various pollutants released during the use of EC.[9-11]

Because of growing concern over the health hazards of environmental tobacco smoke (ETS), official protocols have been established and systematically validated to facilitate the quantitation of pollutants released via smoking of CC (e.g., ref. 12). This current situation for CC contrasts sharply with that of EC. It was demonstrated that the generation of vapor from EC was not possible in the standard laboratory conditions assumed for conventional cigarette testing (ISO3808);[13] accordingly, these authors observed that inhalation of a volume of 35 ml anticipated in a conventional cigarette standard was not applicable to the activation of most EC products.

Despite a strong need for proper assessment of the health risk posed by the use of EC, researchers currently suffer from the absence of reliable or official testing protocols for experimental quantitation of EC-related pollutants. In this respect, the significance of user puff topography (duration and puff velocity) and device characteristics (e.g., power source voltage) on the quantitation of the pollutant yield is gradually being recognized. As such, the effects of such variables have been quantified experimentally and also estimated using mathematical models (e.g., ref 14). Such efforts have been successful enough to demonstrate that the yield rates of EC-related components (like nicotine) are far more variable than a single combustible cigarette. The results of such an informative study confirm the need for establishment of separate official protocols for ECs, as the smoking characteristics of EC are very different from those of CC.

To learn more about the experimental protocols needed for analysis of the key components in EC smoke, we have been involved in the development of analytical methods for the important EC-related pollutants.[9,10] As part of an extension to such efforts, we here report the relationship between the key variables for accurate collection of EC smoke to help assess the basic mechanism of its smoke generation. To build up the basic protocols involved in the collection of smoke samples produced via EC smoking, we considered a list of key variables (puff rate, EC solution composition, and battery charge rate) that can affect the accuracy and/or reproducibility of such sampling conditions. To this end, all these variables were measured in association with the consumption rate of an EC refill solution. The results of this study will thus provide valuable insights into the establishment of protocols required for accurate quantitation of EC smoke samples while facilitating estimation of their emission factors.

2. Methods

2.1 Definition of EC samples: vapor vs. aerosol

Here we classified EC samples into two independent forms as ‘aerosol (particle)’ or ‘vapor’, as depicted in Fig. 1.[11] Upon puffing, EC solutions are heated up electronically and converted into an aerosol phase. Consequently, puffing triggers the transfer of many pollutants in the form of aerosol particles via two different pathways, i.e., liberation of certain species originally contained in the solution and of some others newly formed due to heating-induced chemical conversion of the EC solution. Hence, for a better understanding of the behavior of EC-aerosols, an understanding of the dynamic relationship of various pollutants between before (in the solution phase) and after puffing is required.

It is also important to know that the phase of EC smoke samples generated from cartridges can be distinguished at different stages of sampling. Upon puffing, aerosol particle fraction is generated through heating of the EC solution. The dominant fraction of the EC samples are thus transferred and captured in the form of aerosol particle in the puffing stage.[9,10] However, as sampling (or inhaling smoke) can be continued between or after puffs (e.g., interpuff intervals), a certain fraction of vapor (but not aerosol particle) containing very volatile species (e.g., formaldehyde) can also be collected without puffing. As such, one can technically define that both aerosol and vapor (A/V) samples are collected during a puff, while only vapor can be collected during interpuff conditions (Fig. 1). It should, however, be noted that samples collected during such a break (or interpuff) period are very different from those of the puffing period in that there is virtually no recognizable or actual consumption of the refill solution when the vapor alone is generated and collected during the former.

2.2 Preparation of EC product and test refill solution

Two EC products (Shark, Seven Korea and Gear Pro, Aida) were used in this work for comparative analysis of the sampling conditions for Exps. 1 and 2, respectively. All EC products used for this comparative analysis have a plug at one end of the cartridge. Upon detaching this plug, one can insert a heating element into the cartridge or fill the EC solution into the cartridge. At a constant interpuff interval (10 seconds), a puff of EC smoke was drawn for 2 seconds through the electronic cigarette. For the purpose of this study, all sampling was conducted with the same experimental setup. As shown in Fig. 2, a scrubber made of quartz wool was placed in front of a vacuum pump for protection against overflow of EC smoke in the course of the EC sample collection via puffing.

2.3 EC puff sampling procedures

In this work, we formulate a sampling procedure for EC smoke by considering a list of key variables affecting the rate of smoke generation. To develop a strategy for the collection of EC-related pollutants, we first explored the general variables to be considered for such purposes by focusing on a list of controllable variables like total sample volume, puff duration, interpuff interval, number of puffs, sampling flow rate, refill solution composition, and battery charge conditions.

Assuming that the consumed quantity of an EC solution can vary considerably due to the combined effects of all the relevant variables, we focused on the consumption rate of an EC solution to track all the changes associated with the use of EC. In light of such an assumption, we adopted a mass change tracking (MCT) approach in which the consumed amount of the EC solution is tracked down in relation to each variable used for our testing. Indeed, it was found that the use of this MCT concept should allow an experimentalist to systematically confirm the effects of diverse variables. Hence, the use of this approach should be considered a prerequisite for establishment of the simple and general guidelines needed for an EC sampling protocol.

As summarized in Tables 1 and 2, we analyzed the combined effects of the three sampling variables as the key to assess the major variability involved in EC smoke sampling. First, the sampling flow rate of EC smoke was considered at the very beginning stage of our experiment (Exp. 1: Table 1). Then, the combined effect of both the EC solution composition and the battery charge conditions was evaluated (Exp. 2: Table 2).

In Exp. 1, the consumption amount of EC solution was tested using an EC solution with a fixed solution composition of propylene glycol (PG) and vegetable glycerol (VG) (7:3) with a sampling flow rate or puff velocity set at four values of 0.05, 0.5, 1, and 1.5 L min^{-1}. Hence, at each flow of the test, the smoke samples were taken at total puffing counts of [1] 5, [2] 10, [3] 30, and [4] 50 times under a fixed setup for the puff duration (~ 2 seconds) and interpuff interval (10 seconds). In the case of the very first five puffing counts, the A/V samples were taken five times from each puff lasting 2 seconds (a total of 10 seconds for five puffs) plus four interpuff intervals for a total duration of 40 seconds (=4 x 10 s). As such, it took a minimum of 50, 110, 350, or 590 seconds to complete one cycle of sampling at each puff velocity as explained above. As shown in Table 1, the amount of EC solution consumed for each experiment was recorded along with the corresponding total sample volume for the aerosol (and A/V) samples.

In Exp. 2, the effect of the solution composition was investigated as a priori target. Hence, we prepared a mixture of propylene glycol (PG) and vegetable glycerol (VG) at five different compositional conditions from 2:8 to 8:2 (Table 2). In addition, the capturing performance of the smoke was also evaluated in relation to the battery charge rate from low (25 to 35%) to high conditions (91 to 99%). To conduct a series of Exp. 2 tests, the puff time and interpuff interval were set at 2 and 10 s, respectively. However, unlike the case of Exp. 1, the flow rates for each individual test in Exp. 2 were fixed at a constant value of 1 L min^{-1}.

Nonetheless, the volumes of the EC sample were also similarly compared in relation to the consumed amount of the EC solution at total puff numbers of 5, 10, 30, and 50, as tested in Exp. 1.

As a part of Exp. 2, the smoke collection efficiency was also compared in relation to the low and high battery charge conditions. The electronic cigarette device contains a battery charge rate sensor that is actuated by a puff of air. As the LED panel at the tip of the cigarette shows the charge rate, such information is also used as a valuable variable to test the sampling efficiency. Because of such diversities, one needs to measure and compare the changes in their composition before (the total amount of EC solution prior to use) and after smoking (the EC solution left after smoking).

3. Results and discussion

3.1 Effect of the puff velocity on the EC sampling

To investigate the factors controlling the collection efficiency of the EC smoke, we first investigated the effect of the puff velocity. To this end, the collection of EC smoke was made at four vacuum sampling flow rates of 0.05, 0.5, 1.0, and 1.5 L min^{-1}. As shown in Table 1, for each of these four flow rates, we collected four samples by applying 5, 10, 30, and 50 consecutive puffs with a puffing interval of ~ 2 seconds. As summarized in Table 1, the actual consumption rates of the EC solution, expressed as ΔEC, were also derived as part of MCT approach and compared with the total sample volume computed in terms of aerosol only and aerosol plus vapor (A/V) samples.

Analysis of the ΔEC data confirmed that there was virtually zero consumption of the EC solution when the vapor samples were collected only during the interpuff period. The results of this analysis are quite comparable to those seen in our recent studies of VOCs or carbonyls.[9,10] In other words, the actual consumption of the EC solution took place only under puffing conditions. Nonetheless, evaluation of the collection efficiency was basically made by comparing the total sample volume (A/V) instead of aerosol particle only to consider the total time spent for completion of each sampling, as shown in Fig. 3.

The results shown in Fig. 3 clearly indicate that the amount of A/V samples collected by the vacuum sampling system is sensitive to selection of the puff velocity. At the lowest puff velocity of 0.05 L min^{-1} (symbol of F0.05 in Fig. 3), consumption of the EC solution took place most effectively to show a maximum consumption rate of 763 mg L^{-1} (derived as the slope value of Fig. 3A). As the flow rate increased to 0.5, 1.0, and 1.5 L min^{-1}, the consumption rate dropped in an exponential mode to 185, 107, and 53 mg L^{-1}, respectively. The results of this observation contrasted with those of Talih et al.[14] in that the nicotine yield was affected more sensitively by the concentration of nicotine or voltage setup rather than the puff velocity. However, as their study was conducted at two flow rates between 1.02 and 1.98 L min^{-1}, such effects are not likely to be as clear as those observed in a relatively broad range of puff velocity tested in this study. As such, selection of the sampling flow rate is found as a critical variable for accurate collection of A/V samples from EC.

3.2 Effect of the battery charge rate on sampling efficiency

The voltage setup of an EC device is often reported to be a critical variable to affect the aerosol particle production rate.[14] In general, the conversion of EC solution to aerosol particle is facilitated by increasing voltage. In this study, the investigation of such an effect was also made but in a highly simplified manner. To this end, the sampling efficiency was examined at two contrasting battery charge conditions (as part of Exp. 2): a low (24-35%) vs. a high charge rate (> 91%).

To assess the effect of the battery charge conditions on the consumption of refill solution or sample loading, all experiments in Exp. 2 were conducted at a fixed sampling flow rate of 1 L min^{-1}. Except for the fact that the flow rate was fixed at a single constant value, the puffing conditions for the collection of EC samples in Exp. 2 (puff duration, interpuff interval, and number of puffs) were maintained identically to those employed in Exp. 1. Moreover, the results of all the variables tested in Exp. 2 were also evaluated in reference to the consumption rate of the EC solution, as presented in Table 2.

In Fig. 4A, the sampling rate of EC smoke was compared between the low and high charge conditions in relation to either total sampling volume (or puff number). The results indicated that the patterns of EC solution consumption contrasted noticeably between the two data groups. In the case of the high charged conditions, the consumption rate was measured at 85.3 g L^{-1} (r^2=0.9993; P <0.01), while it was 63.3 g L^{-1} at low charged conditions (r^2=0.9873; P <0.01). The results of this analysis thus clearly confirmed that the battery charge conditions should directly affect the consumption rate of an EC solution. The significance of such a voltage effect on a puff has a number of important implications. For instance, the decomposition of VG and PG to low molecular, carcinogenic carbonyl compounds (e.g., formaldehyde and acetaldehyde) is known to be facilitated at high temperatures.[15] These authors found that increasing the voltage from 3.2 to 4.8 V resulted in an increase in formaldehyde, acetaldehyde, and acetone levels by 4 to 200 times. Although the concentrations of pollutants released from EC are generally lower than those of CC, the levels of formaldehyde from a high-voltage EC device were comparable to those commonly reported in CC smoke.

3.3 Effect of EC solution composition

As explained above, the effect of EC solution composition was also investigated simultaneously in our Exp. 2 stage. Note that PG and VG are the most common media used for preparation of various EC solutions. However, their physicochemical properties are clearly distinguished including the boiling point (188.2 vs. 290 ^{o}C, respectively) and density (1.036 vs. 1.261 g mL^{-1}, respectively). Hence, if these two chemicals are mixed together, their compositional ratios are likely to affect the rate of smoke generation or the consumption rate of an EC solution. Hence, to learn more about the effect of such compositional differences, the sampling rate of EC smoke was explored by varying the PG/VG mixing ratios at five different ratios of 2:8, 3:7, 5:5, 7:3, and 8:2 (Table 2). The results of all these compositional ratios between PG and VG were examined in reference to the consumption rate of an EC solution (Table 2).

In Fig. 4B, the combined effects of the two key variables in Exp. 2 (i.e., the battery charge condition and the compositional ratios) were compared at the same time. To simplify the effect of the solution composition, the data groups were divided into two categories of lighter (PG-dominant condition: PG/VG ratios above unity, i.e., 7:3 and 8:2) or heavier ratios (PG-deficient condition: ratios at or below unity, i.e., 2:8, 3:7, and 5:5). In Fig. 4B, the combined results of the four data groups are compared in terms of the capital letter H/L (between the battery charge rate of high (H) and low (L)) and the numerical value of 1/2 (between lighter (1) and heavier composition (2)). Accordingly, the results appear to be distinguished by both factors. If the mass of refill solution consumed per sampling volume is computed for each of four cases, the results (mg L^{-1}) are found on the order of H2(91.9) > H1(80.9) > L2(77.3) > L1(53.6). The effect of charge conditions is seen consistently as the results were compared without this kind of grouping (as seen in Fig 4A). With this type of grouping, the effects of the solution composition are now visible more evidently. The results in Fig 4B indicate that such factors are reflected, regardless of battery charge conditions so that the consumption rates of heavier compositions (2) are consistently higher than those of the lighter ones (1). Although the importance of the EC solution composition was predicted, such a factor has not been directly validated in previous work.[14] In this respect, the results of this study are very meaningful in that the role of the solution composition is validated as an a priori variable to assess the efficiency of A/V collection. Overall, the results of our study consistently show that the effects of all the selected variables in this work should be considered when the collection of EC smoke samples is carried out based on the principles of the MCT approach.

4. Conclusion

Employment of official sampling protocols is a prerequisite to obtain the measurement data to quantify the level of pollutants released from conventional cigarette smoking with confidence. In this respect, the absence of such protocols in the collection of EC smoke is a critical obstacle to evaluate the reliability and compatibility of measurement data between different studies.

To help establish such protocols, we investigated the potent role of key variables (flow rate, battery charge rate, and EC solution composition) in the collection of EC smoke samples using an MCT approach. The results of our study confirm that selection of the sampling flow rate or puff velocity exerts a great influence on the quantitation of pollutants released from EC smoking. Moreover, with the aid of MCT, it is possible to reliably check whether a mass balance is established between the pollutants released due to EC smoking and the actual quantity of EC-refill solution consumed via equivalent smoking steps. Knowing that the consumption rate of EC solution is controlled by a number of variables, quantitation results based on 'per puff' basis cannot be compared on a parallel basis. Hence, the use of this MCT approach can help resolve uncertainties remaining in the quantitation of pollutant emission rate that used to be computed mainly in terms of 'detected mass of pollutant per puff' basis without considering the actual consumption rate of refill solution.

Acknowledgement

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Table 1. Effect of the sampling flow rate of EC smoke on the mass consumption of EC solution.*

			Puffing conditions					
Order	Sampling flow rate		Duration	Interval	Number	Sampling volume (mL)		ΔEC Mass
	(mL/sec)	(L/min)	(s)	(s)	(N)	Aerosol	A+V	(mg)
1	0.83	0.05	1.98	10.0	5	8.3	50	7.0
2			1.97	10.0	10	16	100	13.4
3			1.99	10.0	30	50	299	39.2
4			1.98	10.0	50	83	499	62.1
5	8.33	0.5	1.97	10.0	5	82	498	6.4
6			1.99	10.0	10	166	998	17.7
7			1.99	10.0	30	498	2,999	66.0
8			1.99	10.0	50	828	4,993	172.6
9	16.7	1	1.99	9.94	5	166	994	14.7
10			1.96	10.0	10	326	1,995	32.0
11			1.98	10.0	30	992	6,001	104.2
12			1.98	10.0	50	1,654	9,997	179.1
13	25.0	1.5	1.98	10.0	5	248	1,495	14.0
14			1.97	10.0	10	493	2,993	28.1
15			1.97	10.0	30	1,478	8,978	90.8
16			1.97	10.0	50	2,463	14,963	121.5

*All tests were conducted using an EC solution with a PG:VG ratio of 7:3.

Table 2. Sampling conditions for EC smoke (aerosol plus vapor) with different refill solution compositions and battery charge conditions (Exp 2)

Order	EC Liquid composition (PG: VG)	A] High Battery Charge Conditions						B] Low Battery Charge Conditions					
					Puffing conditions						Puffing conditions		
		Battery charge (%)	Sampling Volume (mL)	ΔMass (mg)	Duration (s)	Interval (s)	Number (N)	Battery charge (%)	Sampling Volume (mL)	ΔMass (mg)	Duration (s)	Interval (s)	Number (N)
1	2 : 8	97.0	176	11.8	2.11	10.1	5	35	173	11.1	2.07	10.1	5
2		97.0	342	22.1	2.05	10.1	10	33	347	17.6	2.08	10.1	10
3		97.0	1,039	93.1	2.08	10.0	30	30	1052	63.2	2.10	10.1	30
4		95.0	1,724	139.3	2.07	10.1	50	26	1755	121.4	2.11	10.1	50
9	3 : 7	99.0	170	15.7	2.04	10.1	5	28	174	8	2.09	10.1	5
10		99.0	354	30.7	2.12	10.1	10	27	344	13.4	2.06	10.1	10
11		99.0	1,030	94.5	2.06	10.1	30	26	1027	30.6	2.05	10.1	30
12		98.0	1,751	101	2.10	10.1	50	30	1716	87	2.06	10.1	50
17	5 : 5	99.0	174	12.7	2.08	10.1	5	25	172	13.6	2.06	10.1	5
18		99.0	349	23.2	2.10	10.0	10	24	342	12.2	2.05	10.0	10
19		99.0	1,029	79.7	2.06	10.1	30	28	1083	43.7	2.17	9.8	30
20		98.0	1,727	172.2	2.07	10.1	50	28	1545	82.3	2.11	10.1	44
25	7 :3	97.0	177	15.9	2.12	10.1	5	31	174	11.8	2.09	10.0	5
26		97.0	349	29.8	2.09	10.1	10	30	353	24.6	2.12	10.1	10
27		97.0	1,036	104.7	2.07	10.1	30	27	1023	83.4	2.05	10.1	30
28		94.0	1,741	182.8	2.09	10.1	50	25	1735	138.6	2.08	10.1	50
33	8 : 2	96.0	174	13.9	2.09	10.1	5	29	174	11.3	2.09	10.1	5
34		95.0	350	33.6	2.10	10.3	10	28	350	22.2	2.10	10.3	10
35		93.0	1,076	82.1	2.15	10.1	30	27	1076	78.9	2.15	10.1	30
36		91.0	1,779	145.6	2.13	10.2	50	26	1779	134.9	2.13	10.2	50

* (Quartz filter +Sorbent tube+Teflon line)

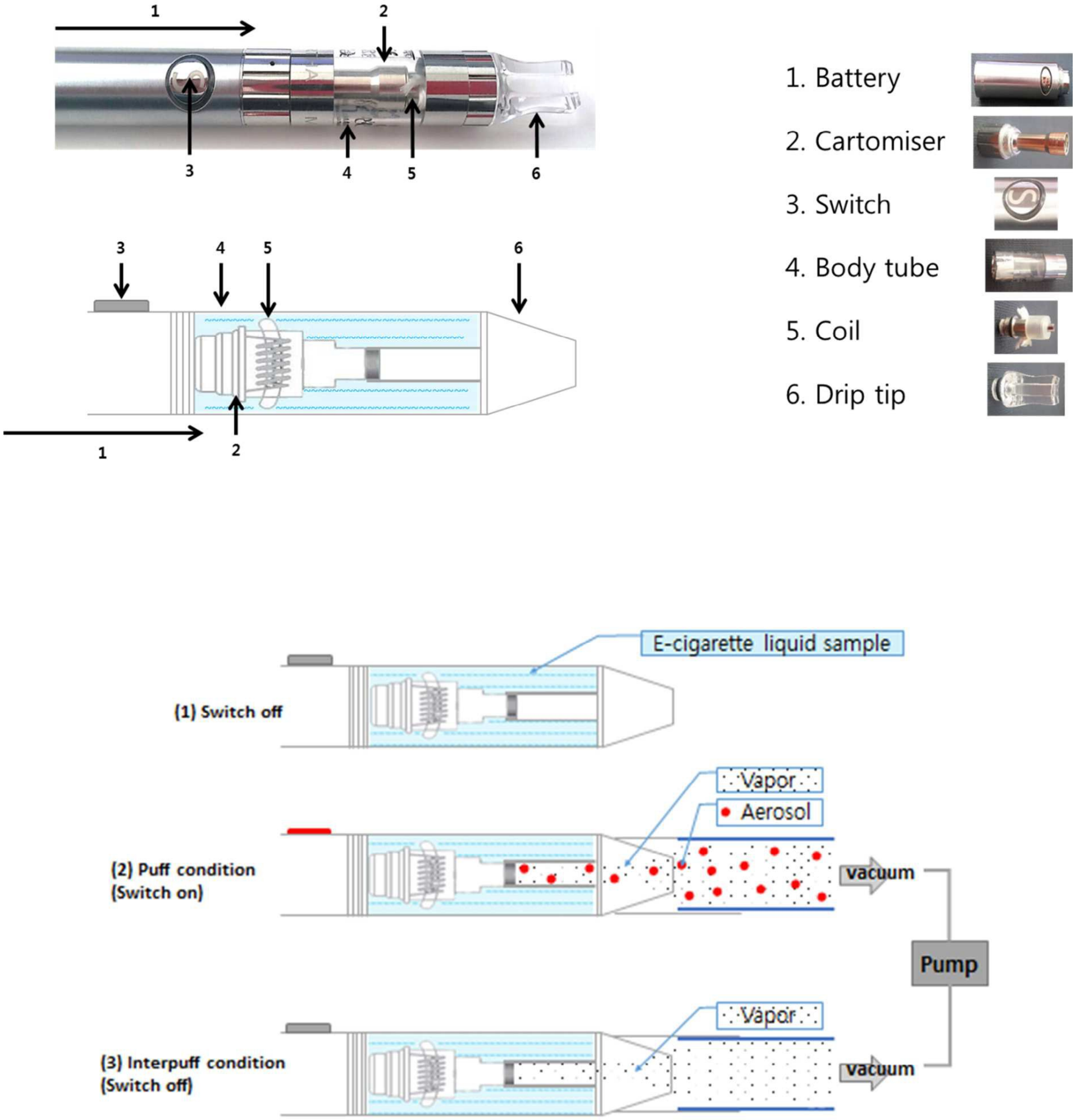


Figure 1. Schematics of the EC system and types of sample (vapor and aerosol particles) with the on and off switch modes

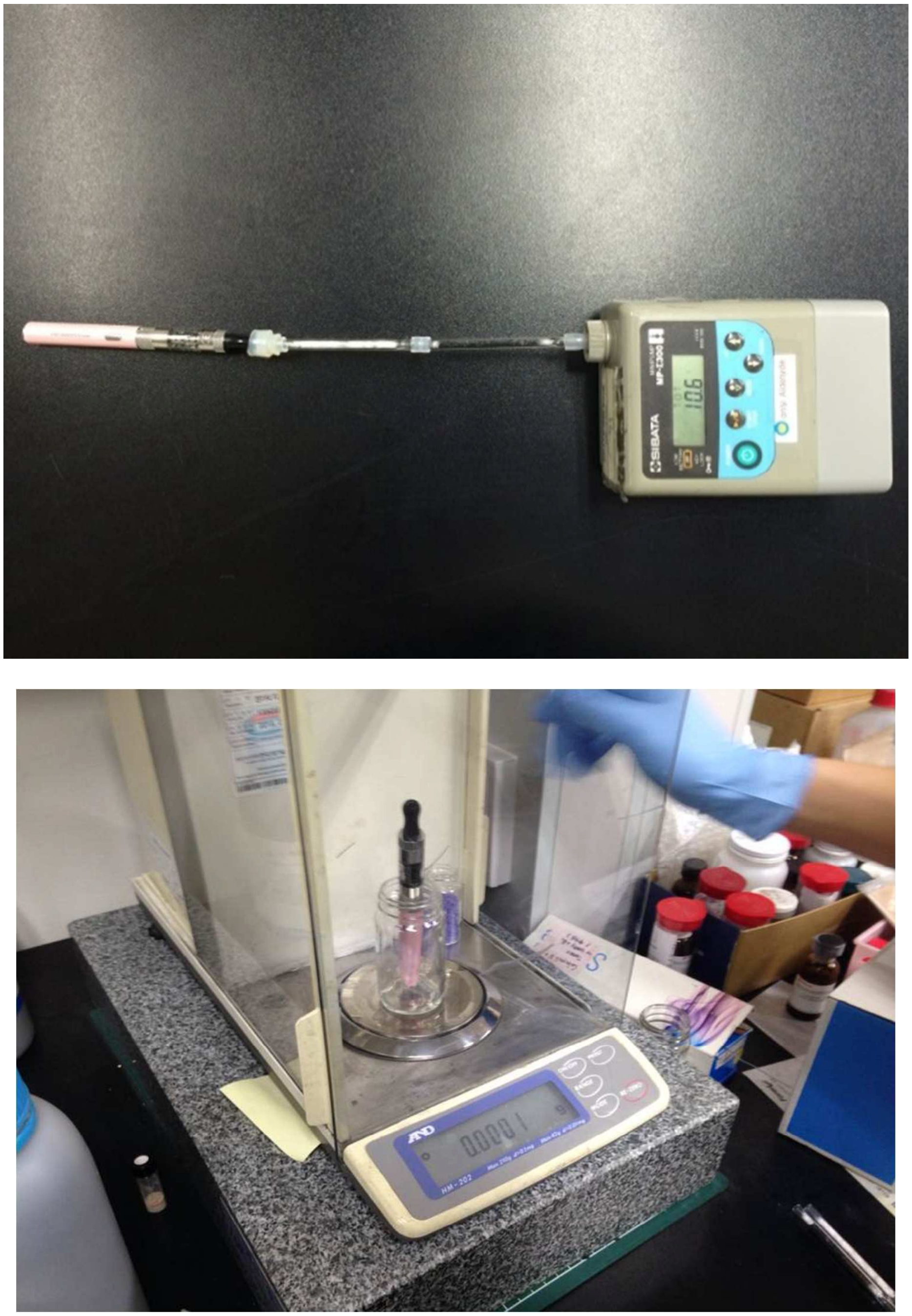


Figure 2. Schematic of the experimental setups for collecting EC smoke (A) and measuring the mass change in EC solution at every step (B).

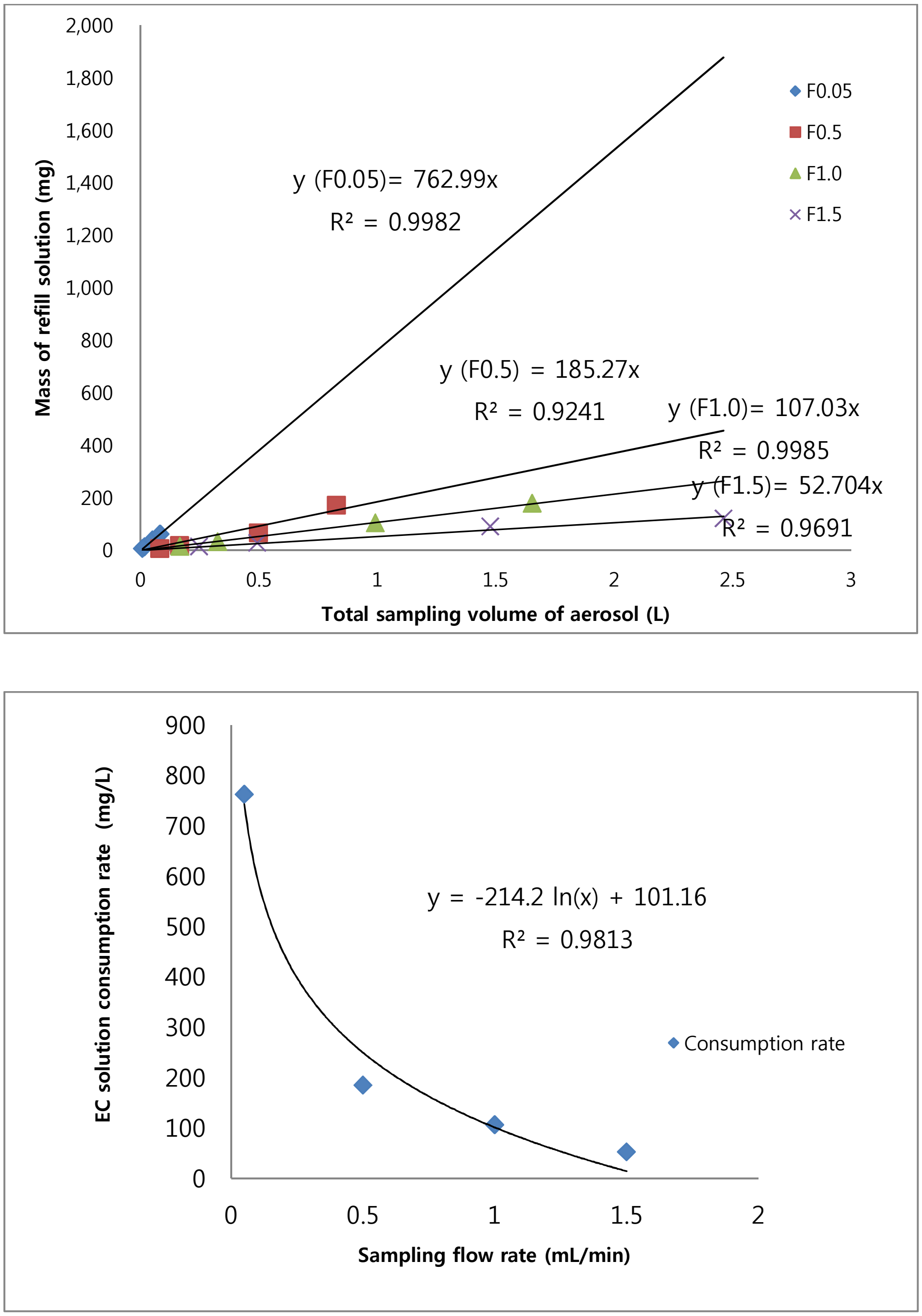
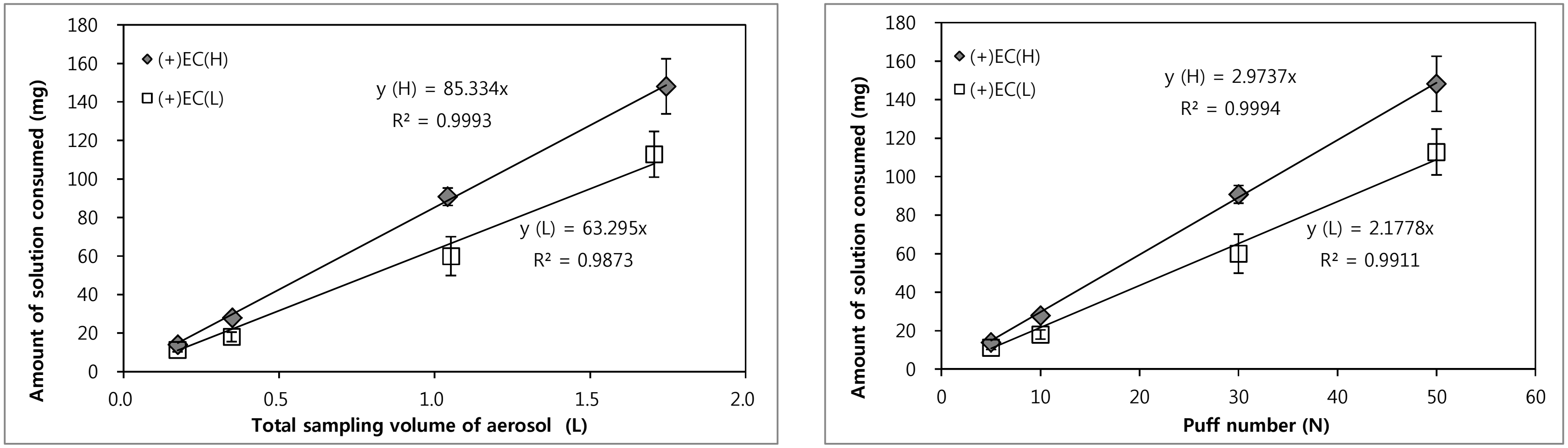
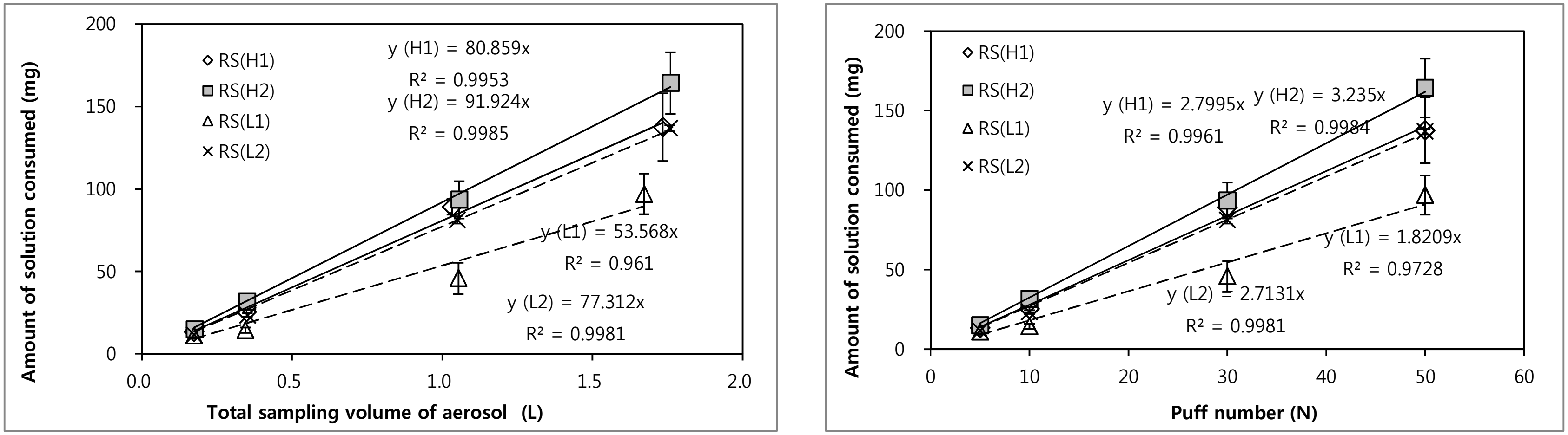


Fig 3. Effect of the sampling flow rate of EC vapor/aerosol particles and the consumption rate of the solution: (A) upper: Relationship between total sample volume and consumed amount of refill solution and (B) lower: Relationship between the sampling flow rate and refill solution consumption rate.



A. Comparison of sampling and EC solution consumption in relation to battery charge rate between high (H) and low (L) levels.



B. Comparison with solution composition conditions: PG percentage less than or equal to 50% (L1 and H1) vs. above 50% (L2 and H2).

Fig 4. The effect of battery charge rate and solution composition on EC sampling rate (Error bars denote standard error).