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#### Short communication

## Are glass fiber particles released during the use of electronic cigarettes? Development of a semi-quantitative approach to detect glass particle emission due to vaping $^{\star}$



Jae-Won Shin<sup>a</sup>, Sang-Hee Jo<sup>a</sup>, Ki-Hyun Kim<sup>a,\*</sup>, Hee-Nam Song<sup>b</sup>, Chang-Hee Kang<sup>c</sup>, Nanthi Bolan<sup>d</sup>, Jongki Hong<sup>e,\*</sup>

- a Department of Civil and Environmental Engineering, Hanyang University, 222 Wangsimni-ro, Seoul 04763, Republic of Korea
- <sup>b</sup> ACEN Co., Ltd, Yeongtong-Gu, Dukyong Dearo 1556-16, Suwon-Si, Gyeonggi-Do 16670, Republic of Korea
- <sup>c</sup> Department of Chemistry, Jeju National University, 66 Jejudaehakno, Jeju 690-756, Republic of Korea
- <sup>d</sup> Global Center for Environmental Remediation (GCER), Faculty of Science, University of Newcastle, Callaghan, NSW 2308, Australia
- <sup>e</sup> College of Pharmacy, Kyung Hee University, 26 Kyungheedae-ro, Seoul 02447, Republic of Korea

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#### ABSTRACT

This study investigated the emission characteristics of glass particles resulting from smoking electronic cigarettes (ECs). First, the most suitable filter for the collection of glass particles was explored by examining the performance (reliability) of various types of filters. A polycarbonate filter was determined as the optimum choice to collect glass particles in EC aerosol. A cartomizer was filled with EC refill solution composed of an equal volume of propylene glycol (PG) and vegetable glycol (VG). To simulate the potential conditions for glass particle emission, EC vaped aerosols were collected at three distinctive puffing intervals: (1) 0–10 puffs, (2) 101–110 puffs, and (3) 201–210 puffs (flow rate of 1 L min<sup>-1</sup>, 2 s per puff, and 10 puffs per sample). Glass particles were observed as early as after 100 times puffing from certain products, while after 200 from others. Thus, glass particles were generated by increasing the number of puffs and usage of the EC cartomizer. The analysis of glass particles collected onto polycarbonate filters by scanning electron microscopy (SEM)/energy dispersive X-ray spectroscopy (EDS) confirmed the presence of glass particles in samples collected after puffing 100–200 times. The study demonstrated that the possibility of glass particle emissions from the EC device increased considerably with the increasing number of total puffs.

#### 1. Introduction

Electronic cigarettes (ECs) are designed to produce the same effect as conventional cigarettes through vaporization of EC solution (Goniewicz et al., 2013). Propylene glycol (PG) and vegetable glycerin (VG) are the main ingredients of EC refill solution. Since the introduction of ECs, the EC devices have continued to evolve into many different forms. In general, EC devices can be classified into three generations: (1) first- generation devices consisting of a single-use cartomizer (a combination of cartridge and atomizer) (Dawkins et al., 2015), (2) second-generation devices consisting of a tank-type cartomizer with refillable EC solution (Dawkins et al., 2015), and (3) third-generation devices consisting of a tank-type cartomizer with refillable EC solution that also include large-capacity batteries to generate or regulate voltage or power (Farsalinos and Polosa, 2014).

In 2003, ECs were introduced for the first time in China as a safer alternative to conventional tobacco (Adkison et al., 2013; Schripp et al., 2013). Since then, ECs have been widely used due to the convenience of purchase (e.g., through the internet) and the availability of various options (e.g., flavor and form). However, the potential harmfulness of EC aerosol has gained attention over the past years. According to recent studies, EC aerosol and refill solution are suspected to be hazardous because they contain and release various harmful compounds such as carbonyls and volatile organic compounds (VOCs) (Jo and Kim, 2016; Marco and Grimalt, 2015). In addition, emission of metal/silicate particles and nanoparticles generated from first-generation ECs has also been reported (Williams et al., 2013). These emissions could potentially lead to respiratory diseases, as EC aerosols can be inhaled deeply into the lungs (Chen et al., 2008; McCauley et al., 2012). Furthermore, as a significant amount of ultrafine particles was found in EC aerosol, their

E-mail addresses: kkim61@hanyang.ac.kr (K.-H. Kim), jhong@khu.ac.kr (J. Hong).

<sup>\*</sup> Capsule: Evidence is established to show that consumer of E-cigarette will be systematically exposed to glasses released via its vaping.

<sup>\*</sup> Corresponding authors.

presence was suspected to induce serious diseases like lung cancer for active or passive smokers inhaling EC vapor (Fuoco et al., 2014; Scungio et al., 2018). Moreover, some glass particles were reported to cause inflammation of the bronchi through the production of toxic oxygen and superoxide dismutase (Cho and Paik, 2016). The incidence of respiratory disease is thus expected to increase if smokers are continuously exposed to glass particles in EC smoke.

In this study, a simple method was developed for effective sampling and analysis of glass particles released during smoking second-generation ECs. The battery of third generation ECs can be interchanged with the cartomizer of second-generation ECs. Hence, the sampling and analytical method for aerosol developed for the second generation EC devices should also be applicable to those for the third-generation EC devices (Farsalinos and Polosa, 2014). The emission characteristics of glass particles from EC devices were also investigated according to two criteria: (1) selection of a proper sampling filter to effectively capture nanoparticles from EC aerosol and (2) characterization of nanoparticle generation in relation to an increase in the number of puffs. The mechanisms involved in the generation of glass particles (or nanoparticles) by EC use were also explored. The results of this study will be helpful to find a better method to measure the potential risks associated with the consumption of ECs.

#### 2. Materials and methods

#### 2.1. Selection of filter for sampling

Selection of an appropriate sampling medium is one of the essential steps for collecting particles in aerosol including EC smoking. For this purpose, a preliminary experiment was performed to find a suitable filter not only for sampling of glass particles in EC aerosol but also for subsequent characterization with scanning electron microscopy (SEM)/ energy dispersive X-ray spectroscopy (EDS). Since the maximum length of the EI emission glass particles is in the micrometer range, filters with a pore size of 2 µm or less were selected. Next, four types of filters containing no silicate or its components were selected among the various filters surveyed, including (1) one type of mixed cellulose membrane filter, (2) two types of polytetrafluoroethylene (PTFE) membrane filters, and (3) one type of polycarbonate filter. Since filters that are exposed to dust can complicate SEM/EDS analysis, filters that frequently generated static electricity were excluded. Filters with an irregular weave pattern that could make it difficult to distinguish glass particles in SEM analysis were also excluded. Likewise, filters that deformed at 80 °C (i.e., the optimum conditions for drying water vapor) were excluded. Through several pre-tests in consideration of these processes, the key obstacles in sampling and pretreatment processes were found and excluded. Eventually, the polycarbonate filter was selected as the optimal medium to collect and analyze glass particles in EC aerosol.

#### 2.2. Sampling and pretreatment of EC samples for SEM/EDS analysis

To reduce contamination of silicate during the sampling and pretreatment procedure, all tools used in this experiment (including filter holders and tweezers) were made of Teflon. In addition, the cartomizer consisting of a glass tube, metal ring, and atomizer (Fig. 1) was cleaned with methanol. The cartomizer was then filled with a refill solution made of PG /VG, which is the main ingredient of EC refill solution, prepared at a fixed ratio of 5:5 and left for two hours or more to wet the coil sufficiently. In order to focus on the formation of glass particles due to the use of the EC devices, experiments were conducted based on EC liquids prepared using reference standard grade, instead of various types of commercial EC liquids. The EC aerosol samples were generated with an EC auto-sampler machine (Chemtekins, Korea) under a fixed puffing condition (1 L min<sup>-1</sup> of purging flow rate, 2 s puffing time, 10 s interval between puffs, and 10 puffs) (Fig. 2). This sampling was carried

out by following the basic procedures for electronic cigarette smoke analysis recommended by the National Institute of Food and Drug Safety Evaluation in Korea (NIFDS, 2016). After aerosol sampling, the filters were dried for 2 h at 80  $^{\circ}\text{C}$ . The temperature of 80  $^{\circ}\text{C}$  was selected because water vapor on the filter dried without affecting the filters at this temperature. The SEM/EDS system was used to examine the dried filters for glass particles.

To assess whether glass particles were generated without heating of the coil, a sample of background air was collected after it passed through the cartridge without the coil or refill solution. Puffs were also collected without vaporizing EC solution. To compare the effect of an increasing number of puffs on sample collection, the EC vaped aerosol samples were collected at three different puffing intervals: (1) samples collected between the 1st and 10th puffs, i.e., just after replacing the coils with new ones (A-10); (2) samples collected between the 101st and 110th puffs (A-110); (3) samples collected between the 201st and 210th puffs (A-210). To detect the release of glass due to the consumption of EC, one needs to collect excessive number of samples. To facilitate the detection of glass released from EC, we simplified our sampling intervals to represent early stage (A-10) vs. moderately consumed stage conditions (A-110 or A-210).

In addition, as a simple means to verify the reliability of the developed sampling method, further research was carried out by testing the method against two other commercially used products (B and C) (Table 1). To allow comparison of glass particle generation patterns, we purchased and tested using three EC devices (A, B, and C). Basic information on the sample codes and their relevant puff conditions is presented in Table 1. Among all samples, the most important three samples collected between the 201st and 210th puffs that were rich in glass particles were used for detailed analysis. These samples were demonstrated to clearly show generation of glass particles by EC products. In addition, to make our experiments consistent in voltage, a battery (power supply) compatible with all three products was used.

#### 3. Results and discussion

#### 3.1. Selection of a filter type for glass particle sampling

To select the best filter for sampling of glass particles emitted during EC use, four types of filters were purchased and examined independently. Basic information about and pictures of the filters prepared for this study are presented in Table 2 and Fig. 3, respectively. The mixed cellulose membrane filter (MCF-0.3) was frequently exposed to dust due to static electricity, and it deformed during drying at about 80 °C (e.g., unsuitable filter). Two other types of PTFE membrane filters were found to be unsuitable for this study because these filters exhibited an irregular wavy pattern that made detection of the target glass particles difficult (Fig. 4). In addition, PF-0.45 attracted dust particles due to static electricity and deformed at about 80 °C. The polycarbonate filter (PF-2.0) did not exhibit any of the disadvantages of the other filters. As a result, the most suitable sampling filter for SEM/EDS analysis and sampling of glass fiber particles in EC aerosol was determined to be the polycarbonate filter. Its advantages included: (1) low exposure to dust, (2) heat resistance at 80 °C, (3) relatively even surface pattern (Fig. 4), and (4) nanometer-sized pores. When samples of aerosol were collected by a PF-2.0, the filter facilitated detection of glass particles in the SEM/EDS system.

### 3.2. Background air samples when collected with or without heating of the solution or coil

In this study, background air samples were collected without heating of the coil, which functioned like the wick in first-generation ECs. This sample was then analyzed to see if glass particles were emitted from the coil of the second-generation EC. The presence of large particles was observed in the filter surface by SEM analysis. These

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