DISCOVERIES IN AEROSOL SCIENCE:

Characterization of Nanoparticles Emitted From
Metallic Coils in Electronic Cigarettes

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Mentors

Jae Hong Park is currently an assistant professor in the School of Health Sciences at Purdue University. He is an aerosol scientist and a certified industrial hygienist with a background in mechanical engineering. Park's research is focused on aerosol

science, exploring the dynamics, synthesis, and control of aerosols. Since occupational aerosols (e.g., welding particles, nanoparticles, and bioaerosols)

decrease air quality and can cause adverse effects on human health, they must be measured for evaluating the air quality and then controlled for preventing exposure. Park's research goals are directed toward controlling aerosols to evaluate and improve air quality. He has focused on nanoparticle synthesis and collection for industrial hygiene, toxicological, environmental, and health purposes.

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Abstract

Electronic (e-) cigarettes, also known as electronic nicotine delivery systems, are battery-operated devices that are growing in popularity worldwide. Although e-cigarettes are known to be safer than traditional cigarettes, their potential health risks have not been extensively reviewed yet. In this study, aerosol nanoparticles generated from e-cigarette heaters were characterized. Kanthal A1 (iron + chromium + aluminum) coils, without nicotine solution or a wick, were installed in an e-cigarette atomizer. The operating conditions were varied coil resistances (0.1–1.0 Ω), a set applied power (10 W), and a stable duty cycle (50%: 5 s on, 5 s off). The size distribution and morphology of particles were collected by a scanning mobility particle sizer and evaluated by transmission electron microscopy, respectively. From an applied power of 10 W and a 50% duty cycle, the size and total number concentration (TNC) of particles emitted increased with greater coil resistances. Within these operating conditions, the average TNC of particles generated exhibited a steep decrease during the first 15 minutes of each trial. The TNC of particles decreased over time because surface oxidation (iron oxide layer) prevented further nanoparticle emission. As a result, a used coil may reduce the risk of the metal exposure from e-cigarettes. The results indicate that our designed and tested e-cigarette generation system is useful for future research that aims to investigate the health impacts of metallic particle inhalation on e-cigarette users.

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Keywords

aerosol, Kanthal coils, nanoparticles, electronic nicotine delivery systems, metal oxides, scanning mobility particle sizer

INTRODUCTION

Electronic (e-) cigarettes are battery-powered devices that generate and deliver vaporized nicotine, flavorings, and other additives (Figure 1). The variety of e-cigarettes available in the current market can be categorized into three broad types: a disposable cigarette-like device, a vape pen with a disposable tank, and an advanced personal vaporizer with a refillable tank (Zhao, Pyrgiotakis, & Demokritou, 2016). These products have been endorsed as alternatives to tobacco smoke and advertised as smoking cessation aids. Population surveys regarding the use of e-cigarettes have found that the majority of those surveyed believe that e-cigarettes are less harmful than regular cigarettes (Rutten et al., 2015; Goniewicz, Lingas, & Hajek, 2013). In the United States, there has been significant growth in availability, marketing, and use of e-cigarettes. Furthermore, the use of e-cigarettes by youths and young adults has increased at an exponential rate within the last decade (Farsalinos, Romagna, Tsiapras, Kyrzopoulos, & Voudris, 2014).

Figure 1. Different types of electronic nicotine delivery systems (FDA, 2019).

Conventional e-cigarette devices are mainly composed of a battery and an atomizer (Figure 2) with a chamber that houses a wick and nicotine solution. A heating element in the atomizer aerosolizes the e-liquid in its vaporized form to be inhaled by the user (Knorst, Benedetto, Hoffmeister, & Gazzana, 2014). Numerous studies have found that the combustion of tobacco in traditional cigarette smoking has been associated with several toxic, genotoxic, and carcinogenic properties. Since e-cigarettes do not produce the toxic combustion products found in regular tobacco cigarettes, the risk of known health effects caused by cigarette smoke could be reduced (Glasser et al., 2017). However, as one of the main components in e-cigarettes, the metallic coil could be a potential source of exposure to toxic metals.

Figure 2. Traditional e-cigarette atomizer.

Traces of heavy metals have been identified and thoroughly characterized in traditional cigarette smoke as well. Metals include but are not limited to chromium, nickel, aluminum, cadmium, copper, lead, and manganese. Most of these metals are vital for physiological processes but can cause detrimental health effects when consumed in high doses. During cigarette combustion, these metals are released as aerosols, and if inhaled, the user may become susceptible to a number of diseases, including atherosclerosis, asthma, and cancer. Once entered into the bloodstream, the metals can translocate, deposit, and accumulate in several vital organs such as the brain, liver, and kidneys (Bernhard, Rossmann, & Wick, 2005).

As aforementioned, metals are also found in e-cigarette vapor. When electrical power is applied, the heating coils found in e-cigarette atomizers can emit metallic nanoparticles (particles less than 100 nm) through evaporation. The process of particle generation is similar to the one in glowing wire generator systems, where evaporated metals can condense and coagulate into nanoparticle clusters due to van der Waal interactions between individual particles (Boies, Lei, Calder, Shin, & Girshick, 2011). Because the coil is a metal alloy, there are a variety of metallic particles generated from the atomizer during the heating of the coil. For example, iron, aluminum, copper, chromium, nickel, and zinc have been previously found in relatively high concentrations in e-cigarette fumes (Olmedo et al., 2018). The concentration of each metal is dependent on the type of coil (Nichrome, Kanthal A1, SS316L, Titanium) used in the atomizer.

Kanthal A1, also known as Kanthal, coils are among the more common metal alloys found in e-cigarettes, mainly due to their abundance in the market, low

purchase/maintenance cost, and high electrical resistance. Kanthal is the commercial name of a metal alloy that is composed of iron, chromium, and aluminum. Recent studies have shown that heated Kanthal coils produce higher concentrations of iron, chromium, lead, and tin, contrary to other e-cigarette coils (Olmedo et al., 2018). In this study, Kanthal metal coils, without the use of a wick and nicotine solution, were heated to characterize the size distribution, total number concentration (TNC), and morphology of the metallic nanoparticles. Collecting and analyzing the particle-size distribution and TNC of generated metallic particles before contacting the wick or nicotine solution provides background data for future toxicological studies.

METHODS

The experimental setup consisted of an air supply system, an e-cigarette generation system, and a measurement setup (Figure 3). The air supply system was composed of an oil trap, a diffusion dryer, and a high-efficiency particulate air filter to remove oil contaminants, humidity, and particles, respectively. The dry particle-free air was controlled with a mass flow controller to be delivered to a glass chamber at 5 L/min. This creates a sheath of air surrounding the atomizer while it is functioning. The e-cigarette generation system consisted of an atomizer, a controller, a diaphragm pump, and a glass chamber. The measurement setup included a scanning mobility particle sizer, consisting of a classifier controller, a differential mobility analyzer, a condensation particle counter, and a soft X-ray aerosol neutralizer.

Figure 3. Experimental setup.

In sterile laboratory conditions, a Kanthal A1 coil with a diameter of 0.405 mm (26 gauge) and an inner diameter of 3 mm was twisted and installed in the atomizer. A box modified-type controller was used in the system. The diaphragm pump supplied the air from the atomizer to a 9-L sampling chamber at a flow rate of 2.5 L/min. The size distributions and TNC of the particles emitted from the atomizer were measured using the scanning mobility particle sizer. The size distributions and number concentrations

were simultaneously measured every 1 minute during a 30-minute sampling run.

The geometric mean diameter (GMD), geometric standard deviation (GSD), and TNC were calculated using an aerosol instrument manager. To examine the morphology and the elemental compositions of the generated particles, samples were collected onto a transmission electron microscope (TEM) grid using an ESPnano, which is an instrument that collects particulate matter. The elemental compositions of the particles were obtained using energy-dispersive X-ray (EDX) spectroscopy. Spectral peaks from the EDX were obtained and compared with standard peaks for the elements of interest.

A new Kanthal coil was tested for each trial. The experimental procedure test settings were adjusted to mimic various e-cigarette operating conditions. In the first test condition, the atomizer was turned on and off at a consistent rate of 0.1 Hz and 50% duty cycle (5 seconds on, 5 seconds off). This was conducted with an applied power of 10 W and under different coil resistance settings (0.1, 0.3, 0.5, and 1.0 Ω). Each test condition ran for 30 minutes. Three trials were conducted for each test condition, with the average of the trials used in the data analysis.

The tested Kanthal wire materials were analyzed by inductively coupled plasma mass spectrometry (ICP-MS). Next, the test coil was digested using a Discover SP-D Microwave Digester and diluted 45 times to get a final HNO_3 concentration of 2% (Luo, Hsu, & Shen, 2006). ICAP-Q ICP-MS was conducted simultaneously with an ESI SC-4DXS Autosampler to measure up to 39 elements present in the test coil. All samples were collected in the kinetic energy discrimination mode by using high-purity helium as the collision gas. Finally, online internal standard addition was performed using 50 $\mu\text{g/L}$ scandium and an SC FAST Valve. The ICP-MS was conducted with a dwell time of 0.01 seconds for all analytes, with 25 sweeps and 3 main runs per sample.

RESULTS AND ANALYSIS

To evaluate the characteristics of the particles emitted from Kanthal coils, particle size distributions and TNC of particles were collected in correspondence to the various operating conditions: coil resistances (0.1, 0.3, 0.5, and 1.0 Ω), an applied power of 10 W, and a 50% duty cycle.

The particle mobility diameter of the generated particles was log-normally distributed and ranged

between 30 nm and 70 nm (Figure 4). These particulates would be classified as nanoparticles because their diameters were measured to be less than 100 nm. The parabolic distributions in Figure 4 of the TNC of particles/ cm^3 have GSDs ranging from 1.2 to 1.6. Therefore, the GSD values depict the plots in Figure 4 as being unimodally distributed.

Figure 4. Particle size distributions with varying resistances.

The GMD of the particles emitted from Kanthal coils increased throughout the experiment (Figure 5). The average GMD was relatively stable during the sampling period at a coil resistance of 0.1 Ω . However, at higher resistances, the size of the particles increased within 10 to 15 minutes into the sampling period before stabilizing. For instance, as the coil resistance increased from 0.1 Ω to 0.3 Ω , the GMD of the particles increased by about 10% in diameter. A similar trend of the growing particle diameters from 41 nm to 46 nm was evident when the coil resistance increased from 0.3 Ω to 0.5 Ω . As the resistance of the coil increased from 0.5 Ω to 1.0 Ω , the particle size increased from 51 nm to 60 nm, and the GMD increased by approximately 27%. On average, the stabilization of the GMD occurred approximately 10 minutes into the sampling period of all trials. The generation of larger particle diameters could be attributed to the condensation of the vaporized metal after the coil was heated. Additionally, the electrostatic properties of the emitted metal nanoparticles could have enhanced the aggregation into larger-size particles.

The average TNC of particles generated from all tested resistances was highest at the start of the trials

Figure 5. Geometric mean diameter over sampling time.

(Figure 6). The magnitude of the initial TNC of particles increased by about two-and-half fold, from 0.1 Ω to 0.3 Ω . Between 0.3 Ω and 0.5 Ω , there was a half-fold increase in the initial TNC of particles. Finally, from 0.5 Ω to 1.0 Ω , the initial TNC of particles more than doubled. However, the TNC exponentially decreased after the first few minutes of the sampling period in all trials. For example, the TNC of a Kanthal coil tested with a resistance of 0.5 Ω decreased from an initial value of 1.1×10^6 particles/cm³ to 2.4×10^5 particles/cm³ within the span of 5 minutes. Overall, the TNC exhibited a rapid rate of decay over the sampling period, decreasing by half the concentration about every 3.5 minutes.

The compositions of the Kanthal coils were captured by a TEM and evaluated by EDX analysis. According to the TEM image, the collected particle appears aggregated, which was formed from much finer primary particles (Figure 7). The TEM image of the particle was combined with EDX maps, and the analysis corresponded to iron, oxygen, aluminum, silicon, magnesium, and chromium, respectively. Interestingly, some elements coagulated and formed an aggregated particle, such as the one boxed in red, as shown in Figure 7. The colored dots in these images indicate the presence of different aerosolized elements. More specifically, the aggregation was identified as a mixture of iron and oxygen nanoparticles because they were concentrated within the outline of the boxed aggregate. However, some particles were dispersed due to the melting of the

Figure 6. Total number concentration of particles over sampling time.

metals from the heated coil. The ICP-MS analysis of the coil revealed that Kanthal is primarily composed of iron and aluminum, along with smaller amounts of chromium and magnesium.

Figure 7. TEM and EDX images of Kanthal A1 coils.

DISCUSSION

From the collected data and analysis, it is evident that there was an increase in the GMD, which corresponded to the generation of larger-size particles over time. The size of the generated nanoparticles increased due to the spontaneous coagulation that took place in the sampling chamber. Smaller particles would be expected if sampling occurred closer to the source of generation, in this case proximal to the e-cigarette generation system. Also, there was an exponential decrease in TNC of particles over a 30-minute sampling trial. This pattern of aerosol generation was noticed in all tested coil resistances. The TEM and EDX analysis of the coil contents revealed that the decreasing TNC of particles during each trial was caused by the oxidation of iron particles.

The presence of iron oxide was expected because the filtered air was used as a carrier gas to generate congregated metallic particles (Khan et al., 2014). Over time, there would be layered coats of iron oxides on the Kanthal coil as the particulates deposit on one another. This phenomenon supports the fact that the initial TNC of particles was highest during the start of sampling and exponentially decreased over time. Furthermore, these results indicate that used or ceramic-coated metal coils could emit fewer metal particles than a new coil. The rate at which oxidation occurs is dependent on several factors, one being the amount of iron or aluminum in the coil. For example, the oxidation rate of the material initially increases with a greater percentage of aluminum present, after which increasing aluminum content results in a decreasing oxidation rate over usage period. Exposure to a temperature higher than 1,000°C will result in an increased rate of oxidation (Sauer, Rapp, & Hirth, 1982).

If inhaled, the remaining generated nanoparticles may translocate into the respiratory system and to other organs. According to the International Commission on Radiological Protection respiratory model, particles less than 0.1 μm are susceptible to alveolar lung tissue deposition (Figure 8). Studies have shown that over time, nanoparticles can penetrate the interstitial space and elicit an inflammatory response (Srinivas et al., 2012). Furthermore, nanoparticles can translocate from the lungs to the lymphatic system, where they can accumulate in the tracheobronchial lymph nodes as well as in the circulatory system. Once in the blood vessels, they can gather at the site of an atherosclerotic plaque buildup (Miller et al., 2013). However, limited information makes it difficult to conclude that nanoparticles produced from e-cigarette coils can cause severe toxic effects. To consider the potential toxicity of metallic nanoparticles in humans, more detailed information such as the mass-based dose, chemical form, solubility, and surface area of particles should be evaluated.

Figure 8. International Commission on Radiological Protection respiratory model.

The results of this study need to be viewed in the presence of several limitations. First, the particles were generated using a modified e-cigarette system under controlled conditions. The air supplied to the system was filtered and dehumidified. Since humidity affects the oxidation rate of the metal, e-cigarette use under normal operating conditions may exhibit a different particle-generation rate. Additionally, the duty cycle set for the e-cigarette system in this study may not correspond with actual e-cigarette usage patterns because particles were generated intermittently based on duty cycle time, rather than a continuous process. Moreover, the metallic nanoparticles generated from the coil may act as a seed for the growth of fumes emitted from the e-cigarette solution, which results in differences in the diameter of the particles. The possibility also exists for particles to be generated from sources other than the e-cigarette coils.

CONCLUSION

The overall aim of this project was to characterize the size distribution and TNC of particles released from metallic coils in a custom-built e-cigarette generation system. The Kanthal coil was heated, without nicotine nicotine solution and a wick, to be further examined by a TEM and an EDX, which capture and collect the elemental compositions of the metal particles, respectively. The size distribution and the GMD of the emitted particles ranged from 30 to 70 nm. Since these particles are classified as nanoparticles, they can enter the body and be translocated to other organs such as the respiratory tract and eventually into the circulatory system. The TNC of particles generated initially increased and then decreased over the testing period due to iron oxidation coating the surface of the tested coils. The TEM images show that aggregation is visible; however, the particles seem dispersed since metals are melted from the heat produced by the coil. In addition to metal oxidation, the EDX analysis revealed that iron, silicon, and oxygen contents were mainly found in particles emitted from the coil. Increasing the resistance of the e-cigarette coil increases the number of particles generated; thus, the user would have an increased exposure to metallic nanoparticles. The use of low-resistance coils, along with an applied power of less than 10 W, is recommended to help minimize metal exposure from e-cigarettes. With a working e-cigarette generation system developed from this project, future inhalation toxicological studies can focus on analyzing the particle emissions from e-cigarettes that contain a nicotine solution, a wick, and test the different metal coils: Nichrome, SS316L, and Titanium.

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