

Nanoparticle Formation from a Commercial Air Freshener at Real-exposure Concentrations of Ozone

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ABSTRACT

Occupational nanomaterial exposure is an important issue in the manufacture of such products. People are also exposed to various nanoparticles in their living environments. In this study, we investigated nanoparticle formation during the reaction of ozone and volatile organic compounds (VOCs) emitted from a commercial air freshener, one of many widely used consumer products, in a 1-m³ reaction chamber. The air freshener contained various VOCs, particularly terpenes. A petri dish containing 0.5 mL of the air freshener specimen was placed in the bottom of the chamber, and ozone was continuously injected into the center of the chamber at a flow rate of 4 L/min with an ozone concentration of either 50, 100 or 200 ppb. Each test was conducted over a period of about 4 h. The higher ozone concentrations produced larger secondary nanoparticles at a faster rate. The amount of ozone reacted was highly correlated with the amount of aerosol formation. Ratios of reacted ozone concentration and of formed particle mass concentration for the three injected ozone concentrations of 50, 100 and 200 ppb were similar to one other; 4.6 : 1.9 : 1.0 and 4.7 : 2.2 : 1.0 for ozone and aerosol mass, respectively.

Key words: Nanoparticle, Air freshener, Ozone, Volatile organic compounds, Nucleation burst, Condensational growth

1. INTRODUCTION

Air freshener use implies improved indoor air quality and healthier air for people to breathe. The use of air fresheners has recently been increasing. Although the volatile organic compounds (VOCs) released from air fresheners often have a pleasing aroma, numerous

risks have been associated with the inhalation of such VOCs, and these risks are often not listed on any product label (Steinemann, 2009). Several VOCs are considered to be health risks and are important factors in the production of photochemical smog and secondary aerosol formation. Many studies have been conducted to investigate indoor and outdoor VOCs, the results of which detailed warnings and preventive measures to improve living environments (Steinemann, 2009; Nazaroff and Weschler, 2004; Weschler, 2000).

Some of the VOCs emitted from air fresheners can react with ozone or air contaminants to form potentially harmful secondary products (Chen and Hopke, 2009; Langer *et al.*, 2008; Liu *et al.*, 2004; Nazaroff and Weschler, 2004). Ozone and other oxidants may also react with the dust particles or the compounds adsorbed on them, resulting in the formation of oxidation products. New particle formation may occur at low VOC concentrations and low or zero air exchange rates (Langer *et al.*, 2008; Sarwar and Corsi, 2007). The use of commercial air fresheners may, therefore, not improve indoor air quality but may actually contribute to increased indoor air pollution to ultimately produce adverse health effects.

Outdoor background-level ozone concentration is particularly high on hot and sunny days, and indoor ozone concentration is affected by outdoor ozone concentration from 30% to 70% (Weschler, 2000). In the National Ambient Air Quality Standard (NAAQS) of the USA, the outdoor 8-h ozone concentration limit is 75 ppb. Limonene and other VOCs react with ozone in the presence of 22-25 ppb ozone concentrations (Liu *et al.*, 2004). Langer *et al.* (2008) reported that secondary particle formation occurs at a 10-20 ppb ozone concentration and at an indoor limonene concentration of < 10 ppb. Therefore, even though an indoor ozone concentration may adhere to the standard, the levels may be sufficient to allow reactions with

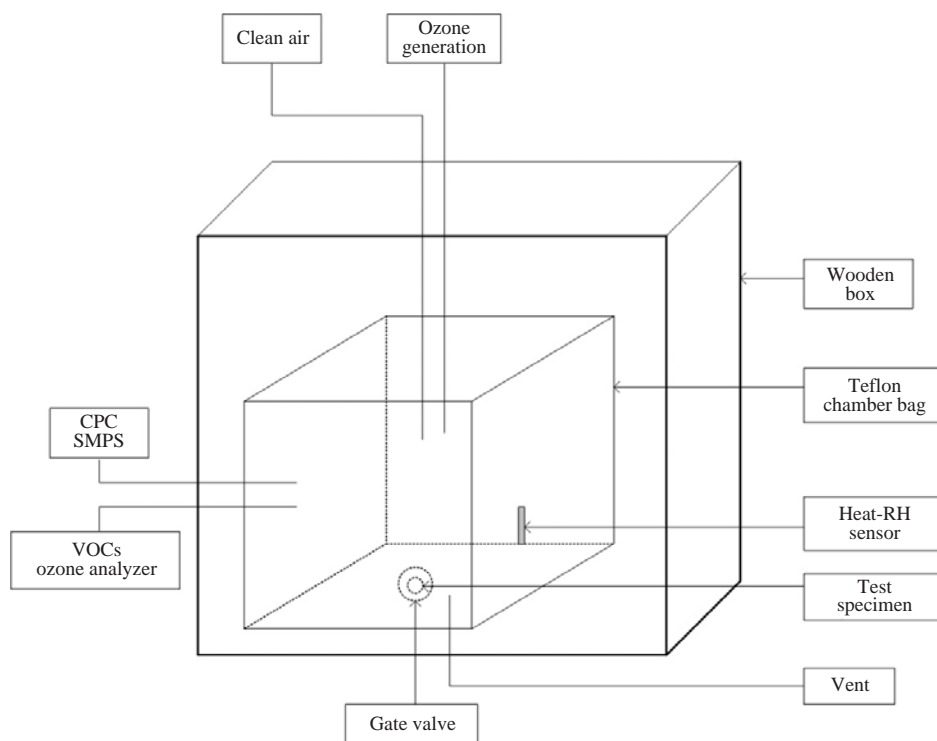


Fig. 1. Schematics of the experimental setup.

VOCs. Secondary pollutant formation depends on the concentrations of reactants such as VOCs and ozone. The evaporation of air fresheners was studied at temperatures less than 100°C (Aggarwala *et al.*, 1998). The higher is the indoor temperature, the faster is the evaporation of VOCs from air fresheners, and the higher is the formed particle concentration (Lamorena and Lee, 2008).

In this study, we investigated secondary aerosol formation during the reaction of ozone and VOCs emitted from a commercial air freshener in a 1-m^3 reaction chamber. Three ozone concentrations (50, 100 and 200 ppb) were used to observe the effect of ozone concentration level. The VOCs contained in the air freshener were identified, and the particle number size distribution was continuously monitored using a scanning mobility particle sizer. The consumed ozone was compared with the amount of aerosols formed during the test period.

2. EXPERIMENTAL

As shown in Fig. 1, the experimental system consisted of a reaction chamber, ozone and pure air generators and ozone and particle monitoring instruments. The volume of the cube-shaped reaction chamber made of 2-mil (about $51\ \mu\text{m}$) FEP Teflon film was 1

m^3 ($1 \times 1 \times 1\ \text{m}$), and the surface to volume ratio was $6.0\ \text{m}^{-1}$. The chamber was installed in a wooden box equipped with a door in order to avoid any light disturbances from the outside. A pure air generation system (Aadco Instruments 737-15) was used to supply zero air into the reaction chamber (Bae *et al.*, 2003). The confirmed concentrations of the impurities of O_3 , NO , NO_2 , NO_x and SO_2 in zero air were $< 1\ \text{ppb}$, and CO concentration was $< 0.05\ \text{ppm}$. Limonene, α -pinene, β -pinene and terpinene were not detected in the gas chromatograph/mass spectrometer (GC-MS, Shimadzu QP2010) analysis of the air.

Prior to each experiment, the chamber was flushed twice with pure air and ozone (200 ppb) for 3 h in order to minimize the effects of bag contamination. Thereafter, background particle and ozone concentrations of the air-filled chamber were measured for 4 h, resulting in an ozone production rate $< 0.01\ \text{ppb}/\text{min}$ and a particle mass concentration $< 0.06\ \mu\text{g}/\text{m}^3$.

During the experiments, the ozone concentration was monitored every one minute using a U.V. photometric O_3 analyzer (TEI 49i) with an O_3 lower detection limit of 1.0 ppb. The analyzer was calibrated with 700-ppb span gas. The particle number size distribution was determined using a scanning mobility particle sizer (SMPS), which consisted of an electrostatic classifier (TSI 3081) and a condensation particle counter (CPC, TSI 3010), over a scanning time of 5 min. The

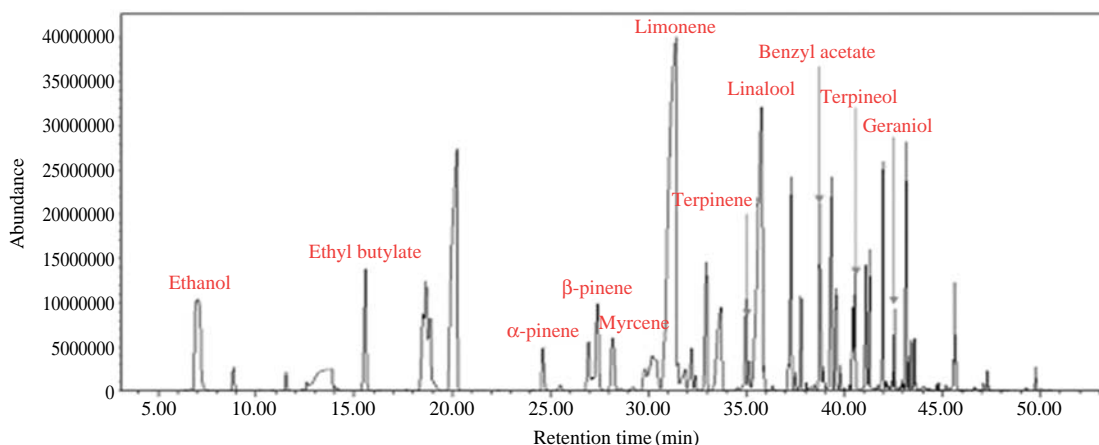


Fig. 2. Chromatogram of the volatile organic compounds emitted from the air freshener tested in this study.

aerosol and sheath flows in the electrostatic classifier were set to 0.3 and 3.0 L/min, respectively, in order to detect particles ranging from 13.8 to 723.0 nm in diameter. Total particle number concentration was also monitored using an ultrafine condensation particle counter (UCPC, TSI 3025) with a 1-sec response time in order to detect particles larger than 3 nm in size. The air temperature and relative humidity in the Teflon chamber were monitored every 5 min using a small sensor with a data logger (Sato Keiryoki SK-L200Th), the lower detection limit for relative humidity of which was 20%. This data logger was placed in the bottom of the chamber.

One Teflon sampling line was connected to the O_3 analyzer, and the SUS sampling line was connected to the SMPS and the UCPC. The total sampling flow rate for monitoring concentrations of particles and ozone in the chamber was controlled to be similar to the flow rate of ozone injected into the chamber. The control of inflow and outflow produced an approximate air exchange rate of 0.24 h^{-1} .

A 0.5-mL test specimen of a liquid-type air freshener was contained in a 50-mL glass petri dish with a diameter of 65 mm that was then placed in the bottom of the chamber, which was maintained at a temperature of $20 \pm 1^\circ\text{C}$ and a relative humidity of $< 20\%$. This specimen seemed to be dried out after about 2.5 h in the chamber at 20°C , implying that all of the VOCs from the air freshener had been emitted by this time. Ozone injection started 4 min after the test specimen was set and occurred into the center of the chamber through the top surface using a photometric O_3 calibrator (API 401). The ozone calibrator was operated at three different concentrations (50, 100 and 200 ppb) at a flow rate of 4.0 L/min. Each experiment lasted for about 4 h.

3. RESULTS AND DISCUSSION

3.1 Volatile Organic Compounds Emitted from an Air Freshener

A commercial liquid-type air freshener sold for home use was chosen as the tested product. A Teflon film bag 300 mm wide and 700 mm long was used to create about 10 L of volume through the supply of zero air. A cylindrical glass beaker containing a measured amount of liquid air freshener was inserted into the bag. The VOCs emitted from the air freshener were sampled into a Tenax TA adsorption tube for 10 min at a sampling flow rate of 200 mL/min. The chemical ingredients collected in the tube were desorbed using a thermal desorption system and analyzed using a GC-MS. The adsorbent tubes were thermally desorbed at 300°C for 5 min, and the analyzed compounds were cryogenically frozen at -10°C . A 60-m fused silica capillary GC column (internal diameter 0.32 mm; film thickness $1 \mu\text{m}$) was used to separate the compounds. The initial GC oven temperature was set at 40°C for 5 min and then increased from 40°C to 80°C at a rate of $5^\circ\text{C}/\text{min}$. The MS was scanned in full-scan mode from m/z 35 to 350 in 0.5-s scan intervals.

The VOC chromatogram is illustrated in Fig. 2. The air freshener contained many volatile organic compounds, such as α -pinene, β -pinene, myrcene, limonene, terpinene, linalool, terpineol, benzyl and geraniol. Some of these VOCs are known to readily react with ozone, forming secondary indoor pollutants (Jo *et al.*, 2008; Lamorena and Lee, 2008; Langer *et al.*, 2008; Sarwar and Corsi, 2007; Fan *et al.*, 2005).

3.2 Background Level under Pure Air Reaction

Prior to conducting the VOCs/ozone reaction, we

needed to know the supplied ozone concentration in the chamber and its background potential to form secondary aerosol particles. A mass balance equation was considered to calculate the variation in ozone concentration in the chamber. The theoretical ozone concentration in the chamber was calculated using Eq. (1).

$$V \frac{dC_t}{dt} = Q_1 C_0 - Q_2 C_t \quad (1)$$

where V = volume of the chamber (L)

C_0 = injected ozone concentration (ppb)

C_t = ozone concentration at time t (ppb)

Q_1 = inlet flow rate (L/min)

Q_2 = outlet flow rate (L/min)

In this equation, it was assumed that the inlet flow rate (Q_1) was equal to the outlet flow rate (Q_2). If V and C_0 are constant and the initial ozone concentration ($t=0$) is zero, then the theoretical ozone concentration in the chamber at time t is calculated using Eq. (2) (Jung *et al.*, 2004).

$$C_t = C_0 (1 - e^{-Q_1 t / V}) \quad (2)$$

The theoretical ozone concentration curves obtained using Eq. (2) for the three experimental conditions (50, 100 and 200 ppb) at the injected ozone flow rate of 4.0 L/min are plotted in Fig. 3. Each experimental ozone concentration curve showed fair agreement with the theoretical values. The slightly lower experimental values might have been caused by the incomplete mixing of the ozone, loss of ozone due to adsorption to the chamber wall and reaction with the VOCs in the chamber filled with pure air. The measured ozone concentrations inside the chamber for 50, 100 and 200 ppb experiments reached 26, 50 and 108 ppb, respectively, after 4 h.

To investigate the effect of background contamina-

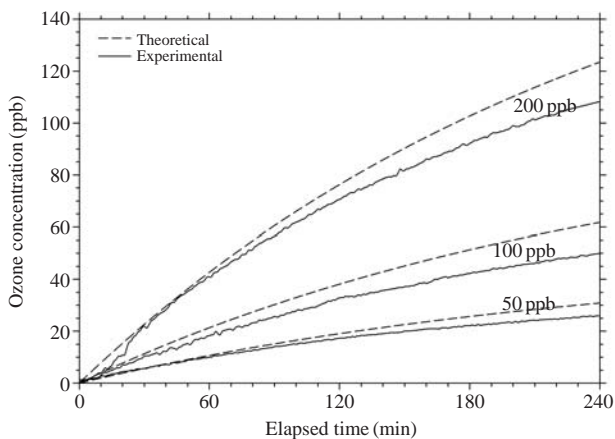


Fig. 3. Comparison of experimental and theoretical ozone concentrations.

tion on the formation of secondary aerosols, we conducted experiments on the reaction of ozone with pure air. Changes in particle number and mass concentration with elapsed time are plotted in Fig. 4(a) and 4(b), respectively. Since secondary organic particles are mostly spherical (Lee *et al.*, 2005), we calculated particle mass from the particle number size distribution data obtained using the SMPS measurements and assuming that particle density was 1 g/cm³. Fig. 4(a) shows that particle formation clearly started at about 105 and 75 min for injected ozone concentrations of 100 and 200 ppb, respectively. Thereafter, secondary aerosols were continuously formed, and the particle number concentrations for 100 and 200 ppb reached 668 and 1,798 particles/cm³, respectively, after 240 min. In the case of the 50-ppb ozone concentration, however, aerosol formation was not observed.

Fig. 4(b) shows that the formed particle mass also increased for both 100 and 200 ppb conditions. Particle mass concentrations due to reactions of unknown

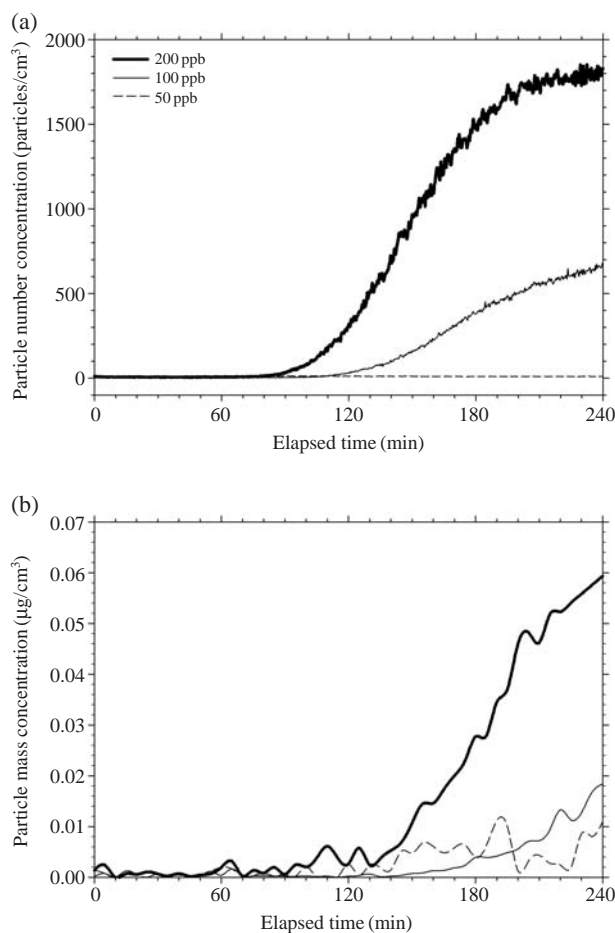


Fig. 4. Background particle formation under pure air reactions for 50, 100 and 200 ppb ozone concentrations, (a) particle number, (b) particle mass.

substances in pure air and ozone in the chamber were 0.02 and 0.06 $\mu\text{g}/\text{m}^3$ for injected ozone concentrations of 100 and 200 ppb, respectively, after 4 h. In the case of the 50-ppb ozone concentration, although small amounts of formed particle mass were observed after 120 min, no increasing trend was observed and the particle mass concentration was maintained under 0.01 $\mu\text{g}/\text{m}^3$. This concentration was thus considered the noise level of particle mass concentration. Thus, the concentration of formed particles was insignificant at the injected ozone concentration of 50 ppb.

3.3 Ozone Reduction and Particle Formation under the Air Freshener Reaction

It is well known that the reactions of VOCs/ozone form secondary aerosol particles. The amount of newly formed particles depends on various factors such as concentrations of VOCs and ozone, air temperature, relative humidity and the chemicals in the VOCs (Lamorena and Lee, 2008; Singer *et al.*, 2006; Fan *et al.*, 2005; Liu *et al.*, 2004). In this study, it was observed that ozone concentration had an effect on secondary aerosol formation.

When ozone was injected into the chamber, the ozone reacted with pre-existing VOCs emitted from the test specimen. Therefore, the ozone concentrations in the presence of the air freshener specimen were lower than those in the pure air reaction, as shown in Fig. 3. Fig. 5 compares these differences among the three ozone concentration levels. As expected, the ozone concentrations in the air freshener experiment significantly deviated from those for the experiment of the pure air reaction, indicating that ozone is consumed during reaction with VOCs.

The ozone consumption rate might also have changed with elapsed time. In Fig. 5, there were no obvious

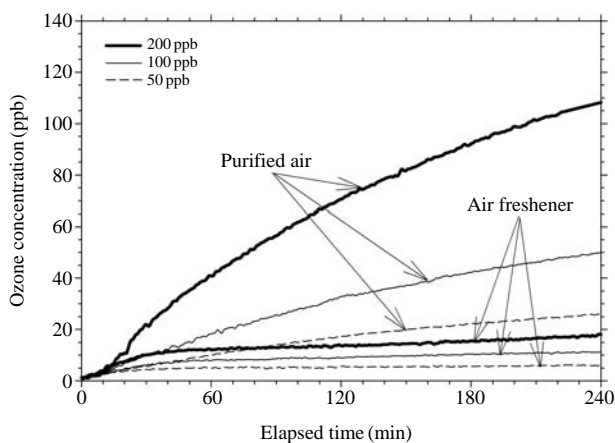


Fig. 5. Comparison of ozone concentrations between the pure air and air freshener experiments during the chemical reaction.

differences between the two ozone concentration curves for the first 10 min. Thereafter, the ozone concentrations slowly increased, resulting in significant deviations from the ozone concentration curves under the pure air reaction. Four time periods (0-10 min, 11-114 min, 115-180 min and 181-240 min) were created according to the ozone consumption rate. As shown in Table 1, ozone was heavily consumed during the second period, after which, the consumption rate decreased. The ozone consumption rate was higher at higher ozone concentration conditions. For example, ozone consumption rates for 50, 100 and 200 ppb experiments during the second period were 0.11, 0.22 and 0.54 ppb/min, respectively. The ozone consumption rate seemed to be proportional to the injected ozone concentration in these experimental conditions. Although ozone was injected into the chamber at concentrations of 50, 100 and 200 ppb, the measured ozone concentrations were significantly lower at the end of experiment, 6, 11 and 18 ppb, respectively.

Typical particle formation and the growth phenomena are shown in Fig. 6(a) and 6(b) for 200-ppb ozone concentration. Fig. 6(a) shows that nanoparticle formation began after about 10 min. The nanoparticle concentration increased very abruptly and quickly reached its maximum peak (82,285 particles/ cm^3) at about 18 min. This rapid formation is well known as the nucleation burst phenomenon. After reaching peak concentration, the particle number concentration decreased due to both coagulation and wall loss. The coagulation effect is calculated to be comparable to the wall loss effect when the particle number concentration is about 80,000 particles/ cm^3 for a particle size of about 40 nm (Hinds, 1999). However, the coagulation phenomenon becomes relatively unimportant when the particle concentration decreases to 50,000 particles/ cm^3 or when particles grow larger than 100 nm, because decrement in particle concentration under these conditions can be explained only by wall loss (Lee *et al.*, 2004).

Condensable vapors formed during the reaction of ozone and VOCs contributed to nanoparticle formation in the early stage of this experiment. After the nuclea-

Table 1. Reaction rate of ozone according to the elapsed time period.

Elapsed time (min)	Reaction rate of ozone (ppb/min)		
	50 ppb	100 ppb	200 ppb
0-10	NC	NC	NC
11-114	0.11	0.22	0.54
115-180	0.08	0.14	0.33
181-240	0.06	0.11	0.29

NC: not calculated

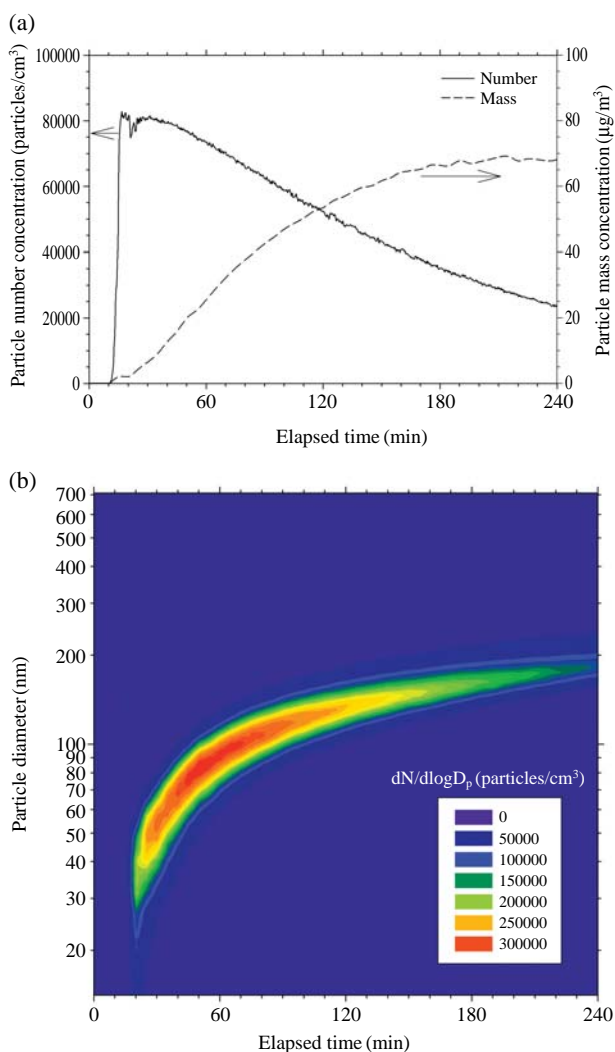


Fig. 6. Particle formation and growth phenomena due to ozone reaction with volatile organic compounds emitted from an air freshener at a 200-ppb ozone concentration, (a) particle number and mass, (b) particle size distribution.

Table 2. Effect of ozone concentration on particle production.

Ozone concentration injected (ppb)	50	100	200
Ratio of ozone concentration supplied	1	2	4
Ratio of ozone concentration reacted	1	1.9	4.6
Ratio of particle mass concentration produced	1	2.2	4.7

tion burst, the nucleated nanoparticles acted as seed sites, and the condensable vapors were deposited onto the pre-existing nanoparticles. As shown in Fig. 6(b), this caused the particles to increase in size because of the condensational growth phenomenon (Heaton *et al.*, 2007; Simchi *et al.*, 2007; Park *et al.*, 2002). During the condensational growth period, total particle mass concentration increased continuously, as shown in Fig.

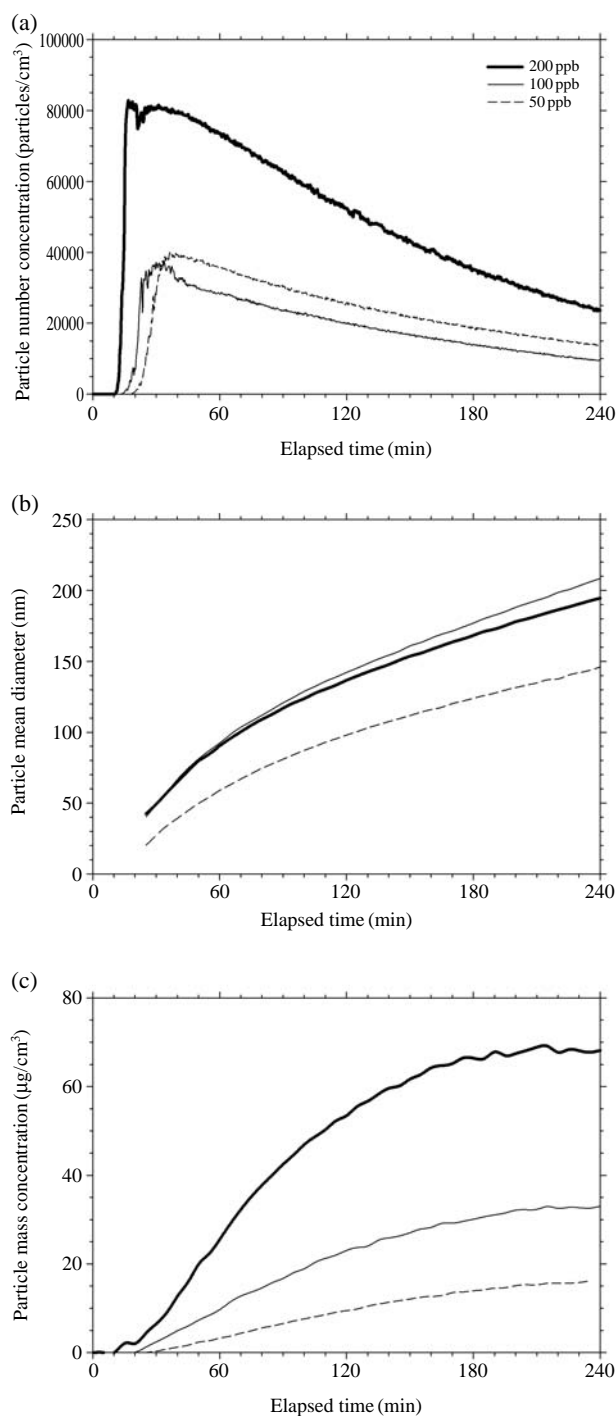


Fig. 7. Effect of ozone concentration on particle formation, (a) particle number, (b) particle mean diameter, (c) particle mass.

6(a) (Sarwar and Corsi, 2007), implying that the condensable vapors contributed continuously to the gas-to-particle conversion process. Here, the particle mass concentration seemed to increase relatively rapidly until 180 min, after which the increase was markedly

slowed. This might be due to the drying out of the test specimen after about 150 min. The particle mass concentration increased to $68.2 \mu\text{g}/\text{m}^3$ at 240 min. Considering that the 24-h $\text{PM}_{2.5}$ standard of NAAQS in the USA is $35 \mu\text{g}/\text{m}^3$, this concentration is not negligible for indoor environments.

3.4 Effect of Ozone Concentration on Particle Formation

Reactions of ozone with constituents containing VOCs emitted from an air freshener produce secondary aerosol particles. Most of these potentially reactive chemicals are terpenes or terpene-related compounds. Fan *et al.* (2003) reported that constituents of VOCs affect the particle mass concentration at a fixed ozone concentration. It is known that ozone concentration affects particle mass formation (Coleman *et al.*, 2008; Lamorena and Lee, 2008). In this study, we investigated the effect of ozone on secondary aerosol formation during the reactions of ozone and VOCs in the chamber. Three low levels of ozone concentration, 50, 100 and 200 ppb, were chosen to simulate real-world situations.

Fig. 7(a) shows that the nucleation burst was delayed as the ozone concentration was decreased. Namely, nanoparticle formation occurred at the elapsed times of 17, 13 and 10 min for 50, 100 and 200 ppb, respectively. The peak concentrations at 50 and 100 ppb were about half of that at 200 ppb. Fig. 7(b) shows the variation in mean diameter of newly formed aerosol particles according to ozone concentration. The trends of particle mean diameter for 100 and 200 ppb were similar to each other. The amount of secondary aerosol formation can be best expressed in terms of particle mass. Fig. 7(c) illustrates that particle mass concentration increased continuously and seemed to be approximately proportional to ozone concentration. Most of the particle mass (about 85%) was produced at the elapsed time of 20-180 min. Leungsakul *et al.* (2005) reported that about 60% of the particle mass was produced in the initial stage. The rates of aerosol mass production were 0.09, 0.19 and $0.42 \mu\text{g}/\text{m}^3/\text{min}$ between 11-180 min for ozone concentrations of 50, 100 and 200 ppb, respectively. Ratios of mass concentrations produced were similar to the ratios of ozone concentration to VOCs, as presented in Table 2. Here, the ozone concentration was a key parameter in the production of secondary particles in the presence of a fixed amount of air freshener.

4. CONCLUSIONS

With regard to environmental health, exposure to

nanoparticles has been extensively studied. In this study, we conducted a reaction chamber experiment to quantify the amount of nanoparticles produced from the reactions of ozone and VOCs. The source of VOCs was an air freshener, a popular consumer product. Three low levels of injected ozone concentration, 50, 100 and 200 ppb, were selected to investigate the aerosol formation process.

From the pure air experiments, we found that measured ozone concentrations reached 26, 50 and 108 ppb after 4 h inside the reaction chamber into which an ozone flow of the concentrations of 50, 100 and 200 ppb, respectively, was continuously injected. The amount of aerosols formed ($< 0.06 \mu\text{g}/\text{m}^3$) was negligible compared to the results ($> 10 \mu\text{g}/\text{m}^3$) of the air freshener experiments.

In the air freshener experiments, we found that ozone was significantly consumed during the reaction period. The nucleation burst occurred early at a higher ozone concentration, and the mean particle diameter increased from $< 40 \text{ nm}$ to $> 100 \text{ nm}$ due to the condensational growth phenomenon. The concentration of secondary aerosols produced seemed to be proportional to the injected ozone concentration. Namely, the rates of aerosol mass production were 0.09, 0.19 and $0.42 \mu\text{g}/\text{m}^3/\text{min}$ from 11-180 min for injected ozone concentrations of 50, 100 and 200 ppb, respectively.

The results imply that VOCs emitted from an air freshener have the potential to produce nanoparticles under low ozone concentrations similar to those in actual living environments. Further studies are needed to quantify the VOC species emitted from consumer products and to classify the production rate of aerosol mass according to standard test methods.

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