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Characterization of Volatilization of Filter-Sampled $PM_{2.5}$ Semi-Volatile Inorganic Ions Using a Backup Filter and Denuders

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ABSTRACT

The characteristics of the volatilization of semi-volatile inorganic ions sampled on a Teflon filter were investigated using a backup nylon filter and annular denuders. The volatilization ratio (VR) was defined as the fraction of the concentration at the backup filter and denuder to the sum of the concentrations collected at the Teflon filter, backup filter, and denuder. Particles whose aerodynamic diameters are less than or equal to 2.5 µm ($PM_{2.5}$) were sampled for 24 h each season from summer 2011 to spring 2012 at a site, about 35 km southeast of downtown Seoul. Increase in VR with increasing temperature was evident, but the effects of relative humidity were not clear, because seasonal variation in the two variables was correlated, and the variation in relative humidity was smaller than that in temperature. While VRs generally decreased with the amounts sampled on the Teflon filter, details of the volatilization behaviors varied among ions according to concentrations at the Teflon filter and VR. The removal of gaseous species at the upstream denuders affected VRs of semi-volatile ions by shifting the equilibrium among NH_4^+, NO_3^-, and SO_4^{2-}.

Keywords: Seasonal variations; Volatilization ratio; Meteorological parameters; Gas-particle equilibrium.

INTRODUCTION

Sampling atmospheric suspended particles on filter media is simple and inexpensive, and its processes are well known and established (Chow, 1995). Owing to these advantages, the filter sampling has been a standard method for the atmospheric particle measurement in many countries and regions, despite the development of a variety of new techniques (USEPA, 2004; Butterfield and Quincey, 2007; Chow *et al*., 2008). However, this sampling is labor intensive and unsuitable to investigate the variations over short time intervals. Moreover, it may cause positive artifacts to uptake gases by collected particles or by the filter medium; and negative artifacts to volatilize semi-volatile materials from the collected particles (Chow, 1995; USEPA, 2004).

These artifacts are intricate, because the filter sampling is designated as a standard method as mentioned earlier. Positive and negative artifacts can be reduced by installing upstream denuders to remove basic and acidic gases and installing backup filters and/or denuders to collect volatilized gases, respectively (USEPA, 1999; McMurry *et al*., 2004). However, complex configurations not only cause troubles in dealing with such system, but also weaken the advantages of being simple and inexpensive of the filter system.

The United States Environmental Protection Agency (USEPA) has established the air quality standards for particulate matter (PM) according to the size and has sought measures against problems associated with the filter sampling (Chow, 1995; McMurry *et al*., 2004; USEPA, 2004). In Europe, as the European Union introduced the standards for PM in the late 1990's, the INTERCOMP campaign has continued as a part of the second phase of the European Experiment on Transport and Transformation of Environmentally Relevant Trace Constituents in the Troposphere over Europe (EUROTRAC-2) to compare the results of filter sampling from different regions (Müller *et al*., 2004; Schaap *et al*., 2004). A number of studies have also been carried out in East Asia, including studies by Tsai and Perng (1998) and Pathak and Chan (2005) investigating particle–particle and gas–particle interactions to assess the artifacts during the filter sampling. In Korea, only a few studies (Kang and Lee, 2002; Kim and Kim, 2007) have paid attention to the filter sampling, because it was designated as a standard method along with introducing $PM_{2.5}$ air quality standard in 2000.

In this study, we sampled $PM_{2.5}$ using a Teflon filter at the Global Campus of the Hankuk University of Foreign Studies (Fig. 1) for a year between summer 2011 and spring 2012 to investigate the volatilization characteristics of inorganic ions. Reactive gases were collected by upstream denuders, and gases volatilized from particles on the filter were collected by a backup filter and a denuder. We

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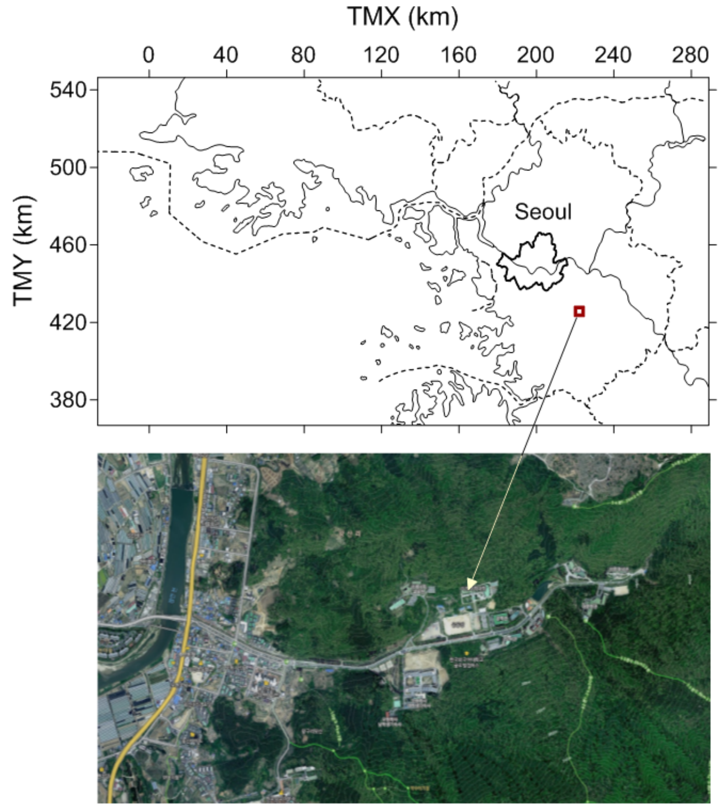

Fig. 1. Location of the measurement site at the Global Campus of Hankuk University of Foreign Studies. A 4-lane national road, about 1.4 km to the west from the measurement site, is shown as orange line in the left of the lower panel.

investigated the effects of factors affecting the volatilization of inorganic ions sampled on the filter and attempted to interpret the results in terms of the equilibrium between gas and particulate phases.

METHODS

Sampling

The sampling site was located at the rooftop of a five-story building on a hill (37.34°N, 127.27°E, 167 m above sea level), about 35 km southeast of downtown Seoul. The site was in the valley upsloping to the east; the west side was a rural area where small-sized buildings, farmlands, and open spaces are scattered on the sides of a four-lane road and river. We have measured particle masses and chemical compositions since the mid-2000s to investigate the effects of emission from the metropolitan Seoul as well as the Asian Continent at the site (Won *et al*., 2010); the Seoul Metropolitan Government Institute of Health and Environment operated a monitoring station within the same campus for studying the pollution characteristics downwind of Seoul for two years from March 2008 (Choi *et al*., 2014).

$PM_{2.5}$ was sampled on a Teflon filter (Zefluor, Pall) with a low volume air sampler consisting of a cyclone (2.5-µm size cut, URG-2000-30EH), two upstream denuders (annular, URG-2000-30x242-3CSS), Teflon filter, a backup filter, and a backup denuder in series (Yu *et al*., 2006). The first denuder in the front of the filter pack was coated with a solution containing 0.1 g of Na_2CO_3 and 0.1 g of glycerol dissolved in 50 mL of deionized water (DW) and 50 mL of methanol to collect nitric acid, hydrochloric acid, SO_2, and other acidic gases. The second denuder was coated with a solution containing 0.1 g of phosphorous acid dissolved in 90 mL of methanol and 10 mL of DW to collect ammonia. Nylon filter (Nylasorb, Pall) following the Teflon filter in the filter pack captured acidic gases, and another phosphorous acid-coated denuder captured ammonia gas, volatilized from particles sampled on the Teflon filter.

Sampling started around 10:00 a.m. with replacing filters and denuder samples and continued for 24 h at a flow rate of 16.7 L/min. Before sampling, the filters were conditioned at 40% relative humidity (RH) for 24 h and weighed using an electronic balance (DVG215CD, Ohaus). Nylon filter soaked in DW in a 125 mL vial was sonicated using an ultrasonic extractor (Power Sonic # 420, Hwashin Tech.) for an hour and cleaned using DW and methanol to remove contaminants. Denuders were dried using compressed N_2 gas after cleaning with DW and filled with coating solutions for 20 min. Temperature and RH were observed using an on-site automatic weather station (Useem Instrument) at 10 min intervals.

Analysis

The sampled Teflon filters were also conditioned at 40% RH for 24 h and weighed. Then, the filters soaked in a mixture of 1 mL ethanol and 14 mL DW were sonicated for 30 min to dissolve water-soluble ions. The Nylon filters were soaked in an eluent for anion analysis and sonicated by the same method as the Teflon filters. Each denuder was extracted by rotating 10 mL DW on the collection surfaces for 20 min. All the extracted samples were stored in a vial and kept in a freezer prior to ion chromatography (IC) analysis.

A Metrohm IC was used to analyze cations (Na^+, NH_4^+, K^+, Mg^{2+}, and Ca^{2+}) and anions (Cl^-, NO_3^-, and SO_4^{2-}) after removing impurities using a syringe filter. Cations were analyzed using a Metrosep C4-150/4.0 column with a solution of 4 mM HNO_3 as eluent, and anions were analyzed using a Metrosep A Supp 5-150/4.0 column with a solution of 3.2 mM Na_2CO_3 and 1.0 mM $NaHCO_3$ as eluent. The minimum detection limits in $\mu g/m^3$ (uncertainties in %) for the IC analysis were 0.040 (4.9) for Cl^-, 0.058 (4.2) for NO_3^-, 0.057 (3.3) for SO_4^{2-}, 0.062 (21.4) for Na^+, 0.010 (15.5) for NH_4^+, 0.010 (8.8) for K^+, 0.007 (10.8) for Mg^{2+}, and 0.045 (7.4) for Ca^{2+}, which was determined by analyzing seven blank filters (Hong *et al*., 2012).

RESULTS AND DISCUSSION

Seasonal Characteristics

The analysis results from the samples collected on the filters and denuders are listed in Table 1 with meteorological parameters. A total of 105 daily 24-h samples were collected by season. The ionic balance was checked by the Global Atmospheric Watch (GAW) criteria suggested by Allan (2004). Total number of valid daily data was 90% of the total number of days during the sampling period except summer. In the summer, despite 33 days in total, sampling was performed on 22 days, because of frequent precipitation including heavy rain. Moreover, five samples were lost because of incomplete sampling, and 17 samples were obtained.

Table 1 shows that the average temperature is high in summer and low in winter. RH is also high in summer and low in spring and winter, but the range of the variation in RH is smaller than that in temperature. The diurnal ranges of temperature and RH are both larger in spring and fall and smaller in summer. The mass concentration of $PM_{2.5}$ sampled on the Teflon filter is high in spring and winter and low in summer; however, the fraction of ions to the total mass is the highest in summer (62% compared to 47–49% in other seasons). Although SO_2 concentration at the upstream denuder is low in summer, SO_4^{2-} concentration is higher than other seasons, indicating an active photochemical production. In contrast, the concentrations of NO_3^- and Cl^- are the highest in winter, because low temperatures are favorable for their partitioning in the particulate phase.

SO_4^{2-} and NO_3^- were neutralized mostly by NH_4^+. Cl^-/Na^+ equivalent ratios were as low as 0.14 and 0.37 in summer and fall, respectively, whereas they were as high as 2.20 and 2.40

Table 1. Seasonal means and standard deviations of meteorological parameters and concentrations at filters and denuders.

Period	Summer (7.21–8.23)	Fall (9.14–10.11)	Winter (11.28–1.4)	Spring (4.26–5.30)
Number of samples	17	24	32	32
Temperature (°C)				
Mean	25.1 ± 1.6	15.5 ± 4.2	–0.9 ± 5.1	16.9 ± 2.2
Diurnal range	8.37 ± 2.6	13.8 ± 3.8	10.1 ± 2.8	13.6 ± 3.7
Relative humidity (%)				
Mean	79.0 ± 7.2	70.5 ± 7.8	63.9 ± 10.1	62.4 ± 9.8
Diurnal range	34.7 ± 11.1	55.6 ± 15.1	45.4 ± 13.4	56.2 ± 13.3
Upstream denuder ($\mu g/m^3$)				
HCl	0.19 ± 0.11	0.36 ± 0.17	0.15 ± 0.13	1.01 ± 0.48
HNO_3	1.67 ± 1.00	2.36 ±1.19	1.16 ± 0.92	3.85 ± 2.11
SO_2	1.19 ± 1.37	4.03 ± 3.27	6.64 ± 6.17	7.40 ± 4.86
NH_3	2.64 ± 0.93	2.02 ± 0.63	0.83 ± 0.52	1.87 ± 0.92
Teflon filter ($\mu g/m^3$)				
Total mass	24.1 ± 17.8	31.7 ± 16.8	42.4 ± 23.5	42.6 ± 20.2
Cl^-	0.04 ± 0.08	0.12 ± 0.09	0.85 ± 0.86	0.74 ± 0.19
NO_3^-	1.40 ± 1.91	2.99 ± 3.67	7.71 ± 6.48	5.43 ± 5.42
SO_4^{2-}	9.14 ± 6.32	8.02 ± 5.04	5.84 ± 5.14	8.89 ± 5.66
Na^+	0.18 ± 0.08	0.21± 0.12	0.25 ± 0.16	0.20 ± 0.07
NH_4^+	3.78 ± 2.51	3.75 ± 2.49	4.60 ± 3.48	5.11 ± 3.32
K^+	0.22 ± 0.12	0.33 ± 0.19	0.38± 0.28	0.26 ± 0.13
Mg^{2+}	0.01 ± 0.02	0.04 ± 0.02	0.06 ± 0.05	0.10 ± 0.08
Ca^{2+}	0.06 ± 0.04	0.10 ± 0.04	0.15 ± 0.08	0.22 ± 0.11
Backup nylon filter ($\mu g/m^3$)				
Cl^-	0.07 ± 0.04	0.12 ± 0.07	0.21 ± 0.20	0.16 ± 0.07
NO_3^-	1.33 ± 0.89	1.99 ± 1.59	0.49 ± 0.51	3.28 ± 1.98
Backup denuder ($\mu g/m^3$)				
NH_4^+	0.93 ± 0.66	0.53 ± 0.77	0.29 ± 0.35	1.61 ± 0.94

in winter and spring, respectively. If Na^+ originated from sea salt as NaCl, low equivalent ratios of Cl^-/Na^+ in summer and fall can be attributed to the chloride depletion by SO_4^{2-} and NO_3^- (Zhuang *et al.*, 1999). For a high equivalent ratio of Cl^-/Na^+ in winter and spring, additional emissions of Cl^- from biomass burning and incineration can be presumed (Won *et al.*, 2010).

Fig. 2 shows the seasonal variation in the volatilization ratio (VR) of semi-volatile ions. VR for ion A is the ratio of the concentration at the backup filter and denuder, $[A]_{backup}$, to the sum of $[A]_{backup}$ and the concentration at the Teflon filter, $[A]_{Teflon}$, as follows:

$$\mathrm{VR}(\%) = \frac{[A]_{backup}}{[A]_{backup} + [A]_{Teflon}} \qquad (1)$$

In Fig. 2, VR of Cl^- is high in summer and low in spring, whereas that of NO_3^- is high in summer and low in winter. VR of NH_4^+ is high in spring and low in winter, but its variation is relatively smaller than Cl^- and NO_3^-. Because semi-volatile substances prefer the particulate form when temperature is low and humidity is high, VR should be low under those conditions (Seinfeld and Pandis, 1998). High VRs of Cl^- and NO_3^- in summer are due to high temperature despite a high RH, indicating that temperature is more effective in the variation in VR, to be discussed later. Clearly, the low VRs of NO_3^- and NH_4^+ in winter are also because of low temperature.

Nevertheless, it is difficult to explain a lower VR of Cl^- in spring than in winter, because a higher temperature and lower RH in spring are unfavorable for Cl^- partitioning in the particulate phase. In the case of NH_4^+, VR is not so high even in summer, because 91% of NH_4^+ in equivalent concentration was bound to stable SO_4^{2-} (Keck and Wittmaack, 2005; Kim and Kim, 2007).

Major Factors Affecting the Volatilization Ratio

Table 2 lists the correlation coefficients between VRs of semi-volatile ions and selected parameters. All VRs in Table 2 are positively correlated with temperature, but the correlation coefficient for NO_3^- is especially high. A low correlation coefficient for NH_4^+ is attributable to its relatively low volatility in summer, resulting from a large fraction of NH_4^+ bound to stable SO_4^{2-}, as described. In fact, VR of NH_4^+ is negatively correlated with SO_4^{2-} concentration on the Teflon filter (*t*-test, $p < 0.01$). Although a high RH is favorable for partitioning in the particulate phase, VRs of Cl^- and NO_3^- show a positive correlation to RH. It may be due to the characteristics of the climate in Korea where RH is high in summer at high temperatures. To distinguish the RH effect, the partial correlation coefficients of RH excluding the effect of temperature was calculated to be 0.387**, –0.034, –0.394** for VRs of Cl^-, NO_3^-, and NH_4^+, respectively (**, $p < 0.01$). Despite the fact that the positive correlation decreases (0.413 to 0.387 for Cl^-) and the negative correlation increases (–0.131 to –0.394 for NH_4^+), compared to the correlation coefficients shown in Table 2, a negative correlation of VR with RH is not distinct.

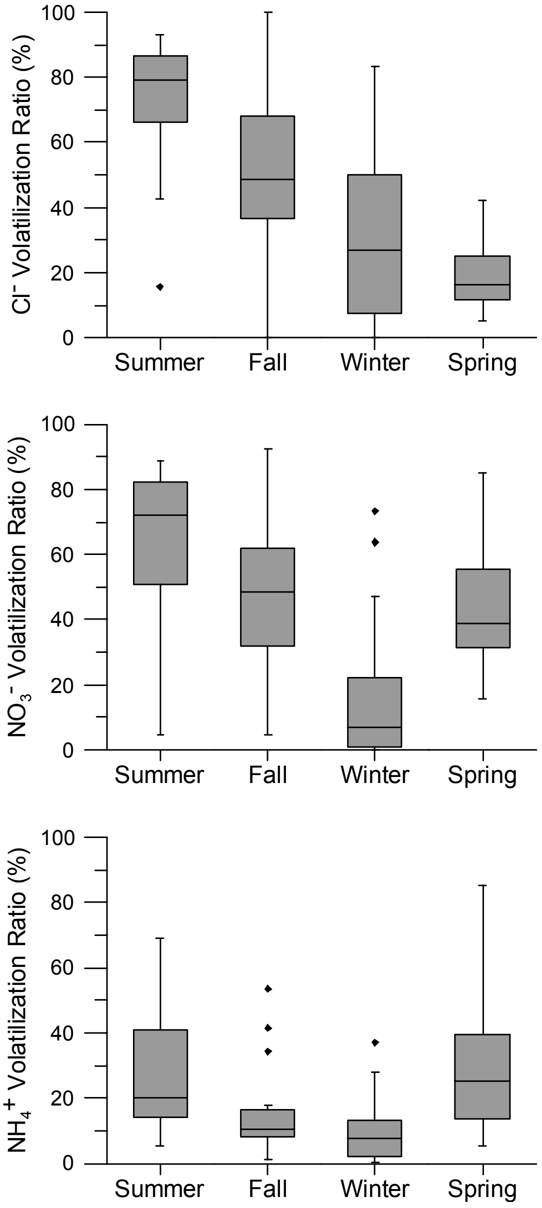

Fig. 2 Seasonal variation in the volatilization ratio of semi-volatile inorganic ions. Boxes represent the 75th, 50th, and 25th percentiles from the top. Upper and lower whiskers represent maximum and minimum excluding outliers. Outliers are the data fall below $Q_L - 1.5\ (Q_U - Q_L)$ or above $Q_U + 1.5\ (Q_U - Q_L)$, where Q_U and Q_L are the 75th and 25th percentiles, respectively. Solid symbols denote the outliers.

Yu *et al.* (2006) found that VR of NO_3^- increased with the diurnal ranges of temperature and RH. Kim and Kim (2007) reported that the sampling artifact of Cl^- in spring and fall was negative during the daytime, but became slightly positive during the nighttime as a result of an increase in the particulate phase with increasing RH. Both studies indicated an effect of the diurnal change in RH. Table 2 shows that VR of Cl^- decreases with diurnal range of RH. The dependence of VR of Cl^- on RH in Table 2, different from Yu *et al.* (2006), is probably because the diurnal range of RH is small in summer with high VR (Table 1). This

Table 2. Correlation coefficients between volatilization ratios of semi-volatile inorganic ions and selected parameters.[a]

	Volatilization ratio (%)		
	Cl^-	NO_3^-	NH_4^+
Mean temperature	0.405**	0.712**	0.396**
Diurnal temperature range	–0.306**	0.061	0.111
Relative humidity (RH)	0.431**	0.301**	–0.131
Diurnal RH range	–0.335**	–0.071	0.051
Upstream denuder HCl	–0.307**	0.227*	0.229*
Upstream denuder HNO_3	–0.131	0.220*	0.056
Upstream denuder SO_2	–0.308**	–0.244*	–0.171
Upstream denuder NH_3	0.285**	0.557**	0.131
Teflon filter sum	–0.367**	–0.413**	–0.476**
Teflon Cl^-	–0.660**	–0.506**	–0.084
Teflon NO_3^-	–0.516**	–0.636**	–0.380**
Teflon SO_4^{2-}	–0.044	–0.026	–0.414**
Teflon NH_4^+	–0.328**	–0.372**	–0.483**
Backup filter Cl^-	–0.018	–0.203*	–0.232*
Backup filter NO_3^-	–0.090	0.283**	–0.087
Backup denuder NH_4^+	–0.143	0.140	0.659**

[a] Statistical significance was tested by the student's *t*-test: * $p < 0.05$; ** $p < 0.01$.

assertion is corroborated by negative correlation of diurnal range of RH with temperature, although the correlation coefficient is small at –0.139. In summary, temperature is the most important parameter in determining VR among meteorological parameters in this study. The effect of RH is not as clear as temperature, because its range of variation is smaller than, and/or its variation is related to, that of temperature. Due to the latter, the partial correlation coefficient excluding the effect of temperature did not help much examine the effect of RH only.

VR of every ion in Table 2 is negatively correlated with the concentration on the Teflon filter, indicating that the more the amount of ions is collected on the Teflon filter, the less is the ratio of gas to particulate phase, resulting in a lower VR. Because of this, Chang *et al.* (2000) proposed the use of a particle concentrator to reduce the sampling artifact by NO_3^- loss. Table 2 also shows that VR of an ion is most negatively correlated with the concentration of the same ion on the Teflon filter. Because of the difference in the gas–particle equilibrium among ions, VRs of Cl^- and NO_3^- are less correlated with the concentration of stable SO_4^{2-}. By the same reasoning, VR of NH_4^+ is less correlated with the concentration of Cl^-, because NH_4^+ is mainly combined with more stable NO_3^- and SO_4^{2-}.

According to the definition of VR given in Eq. (1), VR of an ion should be proportional to the concentrations of that ion at the backup filter and denuder. However, VR of Cl^- is negatively correlated with the concentration at the backup filter along with the largest negative correlation to the concentration at the Teflon filter. The seasonal variations in Cl^- concentrations in Table 1 show that the coefficient of variation at the Teflon filter is larger than that at the backup filter (0.95 vs. 0.42). As a consequence, VR of Cl^- is mainly determined by the concentration at the Teflon filter, and is negatively correlated, because the concentration at the Teflon filter is in the denominator in Eq. (1). In contrast, VR of NH_4^+ tends to be determined by the concentration at the backup denuder since the coefficient of variation at the backup denuder is larger than that at the Teflon filter (0.69 vs. 0.15).

In the ambient air, materials in the gas and particulate phases are in equilibrium. Therefore, the volatilization of a material in the particulate phase on the Teflon filter should increase if that material in the gas phase is removed by the upstream denuder (Yu *et al.*, 2006). However, as shown in Table 2, only VR of NO_3^- is positively correlated with HNO_3 concentration at the upstream denuder ($p < 0.05$) (Liu *et al.*, 2014). Even VR of NO_3^- is more closely related to NH_3 concentration at the upstream denuder ($p < 0.01$), indicating that particulate NO_3^- on the Teflon filter is volatilized, because of the NH_3 deficiency (to form NH_4NO_3) by removing NH_3 at the upstream denuder. The same is true for negative correlation of VR of NO_3^- to SO_2 concentration at the upstream denuder. It seems that removing SO_2 suppresses the production of SO_4^{2-} (Pathak and Chan, 2005), leaves more NH_3 free without forming $(NH_4)_2SO_4$, and secures NH_4NO_3 from volatilization. A similar phenomenon was expected in VR of Cl^-, because a considerable amount of Cl^- emitted from the biomass burning and incineration can combine with NH_3. However, in the case of VR of Cl^-, a negative correlation with HCl concentration at the upstream denuder is obvious. This result is primarily because the amount collected at the backup filter in spring is much smaller than that collected at the upstream denuder (Table 1), but the detailed mechanism for the phenomenon is not certain.

SUMMARY AND CONCLUSIONS

The characteristics of volatilization of semi-volatile inorganic ions sampled on the Teflon filter were investigated using denuders and a backup filter from summer 2011 to spring 2012. Considering a large seasonal variation in meteorology for the study area, we analyzed 105 daily 24-h samples covering full one year. We attempted to understand a detailed behavior of volatilization of each ion by investigating the effects of important factors comprehensively. This was because an accurate understanding of volatilization is prerequisite to quantifying the artifacts and minimizing them more closely.

Among meteorological parameters, the effect of temperature was prominent, and VRs of Cl^- and NO_3^- were the highest in summer. The seasonal variation in VR of NH_4^+ was relatively small, mainly because of a low VR in summer caused by combing NH_4^+ with stable SO_4^{2-}. In contrast to temperature, the effect of RH on VR was not distinct. This was due to the fact that VRs of semi-volatile ions were mostly high in summer when high RH with a smaller diurnal range was unfavorable for the volatilization. More precisely, despite opposite effects of temperature and RH on VR, they were generally correlated in this study. Moreover, the variation in RH is smaller than that in temperature. As a result, it was difficult to distinguish the effect of RH even with a partial correlation coefficient excluding the effect of temperature. Similarly, the effects of diurnal ranges of temperature and RH were not clear.

As indicated by Chang *et al.* (2000) and Yu *et al.* (2006), VRs of semi-volatile ions were negatively correlated with their concentrations at the Teflon filter. However, the volatilization behaviors were different among ions. In the case of Cl^- having lower concentration at the Teflon filter and higher VR, VR increased significantly with decreasing concentration at the Teflon filter. However, in the case of NH_4^+ having higher concentration at the Teflon filter and lower VR, the variations in both VR and the concentration at the Teflon filter were relatively small.

Removing gases at the upstream denuder lowers the gas fraction from the gas-particle equilibrium in the ambient air, and thus, the volatilization from particles on the Teflon filter may increase. However, such a phenomenon was observed only for NO_3^-. Even in NO_3^-, the increase in VR was more distinct when removing NH_3 than HNO_3, because NH_4NO_3 was limited by NH_3. Similarly, VR of NO_3^- decreased by removing SO_2 at the upstream denuder, because NH_3 free from SO_4^{2-} would be produced from SO_2, inhibited the volatilization of NH_4NO_3. Notably, the removal of gaseous species at the upstream denuders affected VRs of semi-volatile ions by shifting the equilibrium between NH_4^+, NO_3^-, and SO_4^{2-}.

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