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# AIRBORNE TOXIC METALS IN INDUSTRIAL COMPLEX AREAS of KOREA USING CCT-ICP-MS TECHNIQUE

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## ABSTRACT

The use of collision cell technology-inductively coupled plasma mass spectrometry (CCT-ICP-MS) is one of the strongly recommended choices for the accurate analysis of the toxic trace metals; this is because CCT-ICP-MS technique prevents polyatomic spectral interferences involved in the determination of As and Cr components. Taking advantage of this technique, the measurements of up to 20 metals were undertaken in this study. Based on the analysis of the metal and ionic compositions of PM10 samples, we attempted to explain the distribution characteristics of elements, to identify sources, and to assess their contributions to PM10 (by positive matrix factorization (PMF 2) method).

**Key Words:** PM10, trace metal, CCT-ICP-MS, source contribution, PMF

## 1. INTRODUCTION

Airborne particulate matters, especially PM10 (aerodynamic equivalent diameter, AED, less than 10 ㎛) fraction have been important. This is because of their potential for deposition on to the human respiratory system being accompanied by many harmful trace metals (such as As, Cd, Cr, Cu, Mn, Pb, Se, and Zn) (Dockery and Pope, 1994; Natusch et al., 1974). These trace metals originated from various source processes (soil, vehicle exhaust, combustion of fossil fuel, incinerator, and industrial process) affect the environment not only in locally limited region but also to a wide area (by long range transportation). The first step of figuring out the characteristics of airborne PM10 in the industrial complex area is to make long-term concentration profiles of PM10 and the associated components including toxic metals.

An analytical method aiming to the quantitative determinations of trace elements in airborne particulate samples should be sensitive and precise, while being potent enough to identify the diversity of elements simultaneously. However, studies have rarely been made either domestically or internationally to apply CCT-ICP-MS for the precise analysis of As and Cr components associated with airborne particulate matter. Hence, the use of CCT-ICP-MS is strongly recommended for the accurate analysis of these toxic trace metals, it can prevent polyatomic spectral interferences involved in the determination of As and Cr components (Tomas, 2002; Sakata and Kawabata, 1994). It was reported that more than 90% of the cancer risk from airborne toxic metals can be caused by those human carcinogens (Jang and Lee, 2002). Therefore, it is a very essential task to improve detection methods for those carcinogenic trace metals accurately.

In this study, we undertake the measurements of about 20 metals including 6 carcinogenic ones (using CCT-ICP-MS) and inorganic ion components (using Ion chromatography). Based on our measurement data, we characterize the concentration status of the study area. In addition, we concentrate on an application of receptor modeling for quantitative source apportionment as well as source identification of PM10 in the study area. Positive Matrix Factorization (PMF), new multivariate receptor modeling technique, developed by Paatero and Tapper (1994), is applied in the identification and quantification of air pollution sources. PMF has special features in that it uses realistic error estimates to weight the data values and the imposition of non-negative constraints in the factor computational process. PMF analysis was successfully applied in a number of prior studies to investigate the pollution sources (Hopke, 2003; Liu et al., 2003; Chueinta et al., 2000; Polissar et al., 1998).

## 2. EXPERIMENTAL

### 2.1 Site characteristics and sampling

The study area, Siwha and Banwall Industrial Complex, is one of the most polluted areas in Korea. This area is located approximately 70 km southwest of Seoul, the capital of South Korea (Fig. 1). It is a coastal area surrounded by a huge residential area. The characteristics of airborne PM10 in Siwha and Banwall Industrial Complex area are very complicated to figure out, because the pollutants are emitted from various types of stacks (about 2,000 sources). The collection of PM10 samples was made at two sampling stations located in both industrial areas and one residential sampling station. The two industrial stations in Siwha (SW) and Banwall (BW) are located about 2 km north-westerly from coast and the distance between the sites are about 8 km as shown Fig. 1. The residential sampling station Jungwang (JW) is located in the northeast direction of the SW station. This site is mostly affected by road traffic PM10 emissions and also by PM10 sources of industrial process from Siwha and Banwall Industrial Complex area.

For the collection of airborne particulates, a PM10 high-volume air sampler (Kimoto Model-121FT, Japan) was equipped with Whatman 41 air filter (8"×10"). This filter is a fibrous filter made of cellulose with a small pressure drop and good mechanical strength. A total of 86 samples in two industrial stations (SW, BW) and 56 samples in residential station (JW) were collected from September 2003 to January 2005. The fundamental meteorological conditions (including rainfall, temperature, relative humidity (RH), wind direction, horizontal wind velocity, etc.) were recorded at hourly intervals during the study period. Air samples were drawn at a flow rate of 0.85 $m^3 min^{-1}$ for the duration of about 24-hour. The total volume of air passed through a filter was computed using the flow recorder for each sampling period.

Whenever the motor brush of sampler was changed, flow rate was re-adjusted by orifice calibrator (GMW-25). The collected samples were pre-stored for 24-hour in a controlled atmosphere (20℃, 50% relative humidity).

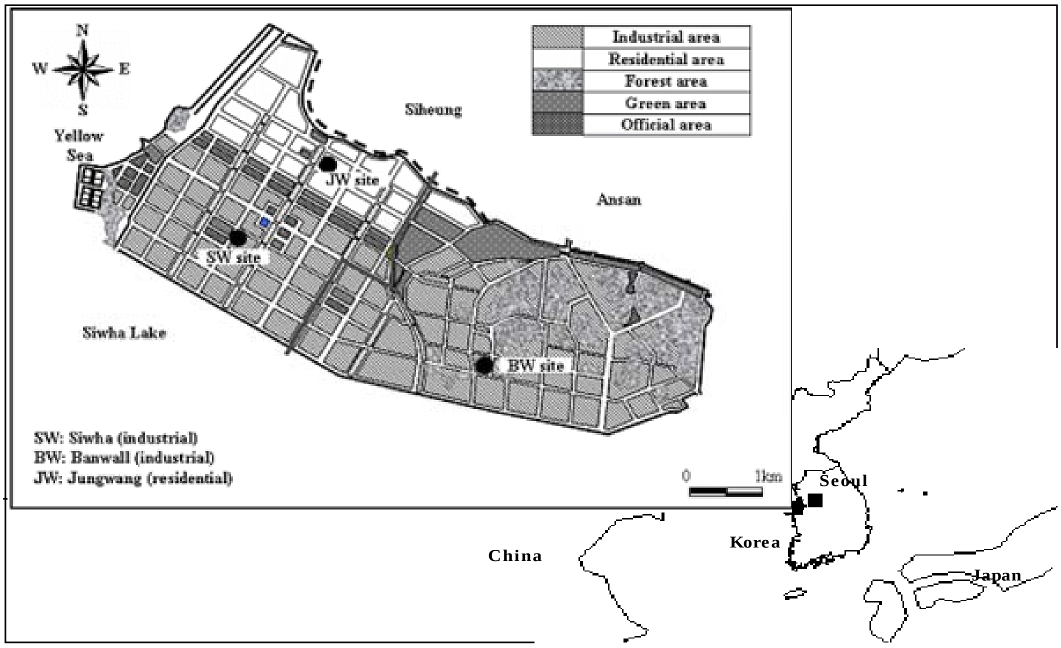


Figure 1. Study area and the sampling sites in Siwha and Banwall Industrial Complex area

## 2.2 Chemical analysis

The concentration of Al, As, Ba, Ca, Cd, Co, Cr, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, Se, Ti, V, and Zn associated with PM10 were determined on a CCT-ICP-MS (X-7 mounting series model with hexapole collision cell, Thermo Elemental), which can remove polyatomic spectral interferences affected by plasma gas (Ar), matrix components, and solvent acid (Tomas, 2002; Tanner, 1995; Sakata and Kawabata, 1994; Jiang et al., 1988). A portion of the sampled filters (1/10) was decomposed by microwave digestion with 5mL nitric acid. All the extracts were pooled and brought in to a final weight of 50 g in polyethylene bottle using 1% pure $HNO_3$ solution. The instrumental conditions used were basically similar to those reported by the instrument manufacturer. The results obtained by the standard calibration curve method were in general in line with those obtained by the standard addition method (r value above 0.999). The final concentrations were corrected with the combined reagent and filter blanks.

The NIST standard reference material (SRM: NIST, the National Institute of Standards and Technology, U.S.A., SRM 2783, air particulate on filter media) was used for the analytical quality control of CCT-ICP-MS analysis. It was found that relative errors (against SRM values) of Al, As, Cr, Fe, Mg, Mn, Pb, Sb, V, and Zn fell below 20%, while those of Ca, Si, and Ti above 20%.

The filters were also extracted using ultra-pure water using by ultrasonic instrument (Branson 8210, U.S.A.) for 30 minutes. Ion chromatography was also used to determine the concentrations of $SO_4^{2-}$, $NO_3^-$, and $Cl^-$. IC (ΩMetrohm, 761 Compact

IC model) consists of a separation column (IC SI-90 4E, Shodex, Japan), a guard column, and suppressor (SCX Membrane PCR, SeQuent, Sweden) using sulfuric acid. For the IC analysis, the pooled standard deviation for peak area of standard solution was about 4 %, recovery of spiked anions ranged between 97-105 %, and the average precision determined from duplicate analysis ranged between 1.5-7% for three anions. Therefore, the stability and reproducibility of the analytical condition can be considered highly stable.

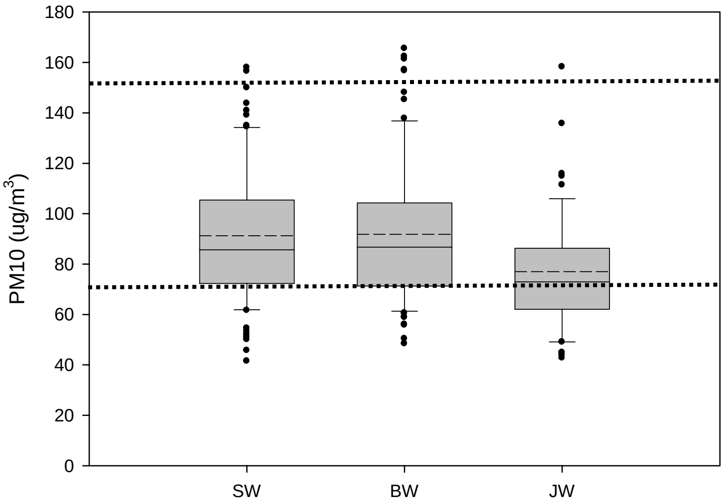
## 3. RESULTS AND DISCUSSION

### 3.1 Mass concentration of PM10

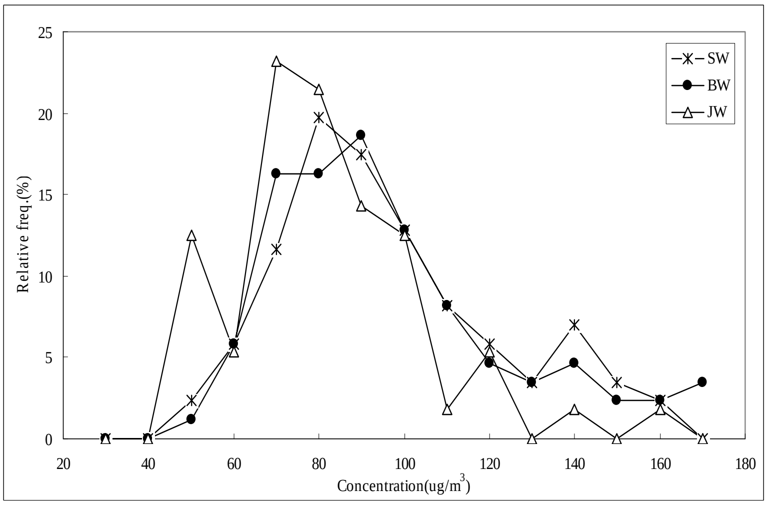
The seasonal values for PM10 measurements are shown in Table 1. The annual average of PM10 was 91.2 $\mu g/m^3$ (SW) and 91.8 $\mu g/m^3$ (BW), whereas the counterpart at JW was 77.0 $\mu g/m^3$. The similar temporal variation of PM10 was observed from three sampling sites, showing the highest concentration in fall while the lowest in summer. The distribution of PM10 concentration is also shown as box-whisker plot and relative frequency patterns in Fig 2. The mean annual concentrations of PM10 at all sites exceeded 70 $\mu g/m^3$ of Korean annual limit, and also 2 times at SW, 3 times at BW, and 1 times at JW exceeded the daily PM10 limit value, 150 $\mu g/m^3$. The daily PM10 concentrations between three sites have a strong correlation coefficient, which SW to BW was 0.77, SW to JW was 0.73, and BW to JW was 0.74. This may imply that PM10 concentrations in study area are affected to each other.

Table 1. A statistical summary of PM10 concentration divided in spatial and seasonal base (unit: $\mu g/m^3$).

| | | Mean | S.D. | S.E. | Min | Max | N |
|---|---|---|---|---|---|---|---|
| SW | Spring | 95.5 | 18.7 | 5.0 | 61.6 | 134.9 | 14 |
| | Summer | 63.5 | 12.5 | 3.3 | 45.7 | 88.5 | 14 |
| | Fall | 100.2 | 31.8 | 5.9 | 41.5 | 158.0 | 29 |
| | Winter | 93.6 | 21.0 | 3.9 | 61.9 | 143.7 | 29 |
| | All period | 91.2 | 26.7 | 2.9 | 41.5 | 158.0 | 86 |
| BW | Spring | 92.0 | 24.2 | 6.5 | 63.2 | 157.1 | 14 |
| | Summer | 65.5 | 10.4 | 2.8 | 48.3 | 83.8 | 14 |
| | Fall | 104.4 | 33.2 | 6.2 | 58.9 | 165.5 | 29 |
| | Winter | 91.9 | 21.9 | 4.1 | 58.8 | 137.8 | 29 |
| | All period | 91.8 | 28.2 | 3.0 | 48.3 | 165.5 | 86 |
| JW | Spring | 84.2 | 24.8 | 6.6 | 66.9 | 158.2 | 14 |
| | Summer | 55.0 | 10.4 | 2.8 | 42.7 | 73.8 | 14 |
| | Fall | 89.7 | 20.3 | 5.4 | 58.5 | 135.7 | 14 |
| | Winter | 79.1 | 16.1 | 4.3 | 59.8 | 111.4 | 14 |
| | All period | 77.0 | 22.5 | 3.0 | 42.7 | 158.2 | 56 |



(a) box-whisker plot



(b) relative frequency patterns

Figure 2. The distribution patterns of PM10 concentrations in Siwha (SW) and Banwall (BW) industrial area; (a) box-whisker plot for inspecting level of PM10 (upper line means daily PM10 limit value, while lower line means annual PM10 limit value of Korea) and (b) relative frequency for shape of distribution of PM10.

### 3.2 Concentration of metals and ionic species

To constrain uncertainties involved in our measurements, we eliminated some outlying data sets prior to statistical analysis by employing the two independent screening steps (Kim et al., 2002). As a first step, about 7.7 % of data sets were removed on the basis of signal-to-noise (S/N) ratio of less than 2. Following the initial screening, the data sets with the values exceeding ±3 SD from the mean were also excluded to rule out the possibility that the distribution patterns of certain components are distorted by the presence of a few extreme values. Loss of data was about 1.5 % at this secondary stage. The concentration of metals and three ionic species in the study area after this screening procedure was summarized in Table 2.

From the results of the quantitative analysis for airborne PM10 samples by CCT-ICP-MS, it was found that the concentration of metals associated with crustal sources (such as Al, Ca, Fe, K, Mg, and Na) were much higher than that of any other toxic metals. The distribution patterns of different metals were clearly distinguished with their concentrations ranging across five orders of magnitude. Based on a simple comparison of the metal concentrations by their magnitude, the data sets could be grouped into five different categories: 1) $< 10^{-1}$ ng/m$^3$: Be; 2)$< 10^0$ ng/m$^3$: As, Cd, Co, Se, and V; 3) $< 10^1$ ng/m$^3$: Ba, Cr, Mn, Ni, Ti, and Sb; 4) $< 10^2$ng/m$^3$: Al, Cu, Mg, Na, Pb, Si, and Zn, and 5) $> 10^3$ng/m$^3$: Ca, Fe and K.

The mean concentrations of several toxic metals (including As, Mn, Se, V, and Zn) in the SW site were measured as 7.06±3.34, 93.5±49.5, 3.17±1.87, 7.54±5.14, and 451±304 ng/m$^3$, respectively. In the case of BW site, they were measured to be 5.34±3.26, 79.4±42.5, 4.08±2.86, 6.53±6.33, and 624±394 ng/m$^3$, respectively. In the JW, they were 5.65±2.92, 39.0±24.1, 3.23±1.69, 8.66±6.10, and 287±194 ng/m$^3$, respectively.

The mean concentrations of three inorganic ions ($Cl^-$, $NO_3^-$, and $SO_4^{2-}$) were also measured from those three areas as well. In the SW site, the results were 4.91±2.76, 10.5±6.05, and 9.91±6.42 $\mu$g/m$^3$, respectively. In the BW site, they were 9.01±6.13, 10.1±6.89, and 10.0±7.53 $\mu$g/m$^3$, respectively. In the JW site, they were 2.79±1.92, 9.46±5.64, and 10.4±4.64 $\mu$g/m$^3$, respectively. The sum of anionic species in BW was contributed about 29.0 % of total PM10 mass concentration, while sum of those species in SW and JW are about 25 %, respectively.

Comparison of the aerosol compositions in relation with crustal composition can help discriminate the contribution of man-made source processes from other source processes (Senaratne and Shooter, 2004; Gao et al., 2002). The concept of enrichment factor (EF) is based on the fact that some metals originating from well-defined sources (such as Al or Fe) mainly originating from the earth's crust) can be distinguished from other metals derived by different source processes. On the basis of arbitrary EF value criteria of 50, we were able to sort out such metals as As, Cd, Co, Cr, Cu, Ni, Pb, Sb and Se to be enriched in PM10 samples of our study sites. It was noteworthy that most of those metals are the major anthropogenic source components.

### 3.3 Source contributions

Based on the metal and ionic composition data for PM10 samples, we attempted to identify sources, and to assess their contributions to PM10 by positive matrix factorization (PMF 2) method. Basic equation of PMF model is shown equation (1). The method is to find the unknown matrix, G and F by the solution of a least square method iteratively

$$X = GF + E \qquad (1)$$

Where, X(m×n) is the data matrix consisting of the m chemical components analyzed in n samples, G(n×p) is the source contribution to the each sample. F(p×m)

Table 3. Concentration of metals and three ionic species measured from the two industrial stations (SW and BW) and one residential area (JW) during 2003-2005. The concentration unit of three ions are $\mu g/m^3$, while all the rest are in $ng/m^3$.

| | (1)SW | | | (2)BW | | | (3)JW | | |
|---|---|---|---|---|---|---|---|---|---|
| | Mean | Min | Max | Mean | Min | Max | Mean | Min | Max |
| Al | 1090 | 29 | 2691 | 1294 | 260 | 6527 | 1394 | 43.0 | 9494 |
| As | 7.8 | 1.6 | 28.6 | 6.6 | 0.9 | 21.3 | 7.6 | 1.4 | 18.7 |
| Ba | 35.3 | 4.4 | 218.3 | 40.3 | 3.5 | 207.4 | 36.6 | 6.5 | 103 |
| Be | 0.06 | 0.01 | 0.21 | 0.04 | 0.01 | 0.09 | 0.06 | 0.02 | 0.19 |
| Ca | 995 | 482 | 2293 | 796 | 52.6 | 2236 | 522 | 38.1 | 3536 |
| Cd | 7.0 | 0.4 | 31.7 | 6.4 | 0.4 | 33.1 | 4.6 | 0.5 | 14.4 |
| Co | 1.4 | 0.1 | 6.4 | 1.5 | 0.1 | 5.9 | 0.9 | 0.1 | 2.7 |
| Cr | 78.8 | 5.5 | 468 | 20.9 | 1.9 | 83.4 | 10.9 | 1.7 | 37.8 |
| Fe | 1331 | 236 | 3979 | 1495 | 190 | 5917 | 1135 | 103 | 7422 |
| K | 1396 | 427 | 13075 | 1141 | 90.7 | 3825 | 1193 | 63.6 | 3736 |
| Mg | 348 | 50.5 | 1585 | 398 | 43.8 | 1646 | 376 | 30.0 | 2618 |
| Mn | 137 | 5.7 | 643 | 111 | 12.3 | 421 | 63.9 | 5.5 | 318 |
| Na | 578 | 157 | 1592 | 617 | 38.2 | 4601 | 686 | 112.9 | 2102 |
| Ni | 44.3 | 4.0 | 360 | 29.7 | 3.5 | 298 | 12.0 | 1.8 | 39.0 |
| Pb | 382 | 19.7 | 1672 | 487 | 8.2 | 2891 | 229 | 19.2 | 1662 |
| Sb | 8.9 | 0.9 | 32.0 | 8.4 | 0.6 | 59.9 | 6.5 | 0.5 | 17.5 |
| Se | 3.3 | 0.5 | 11.9 | 4.9 | 0.6 | 25.3 | 2.9 | 0.7 | 7.2 |
| Si | 520 | 243 | 1309 | 492 | 146 | 1430 | 391 | 74 | 967 |
| Ti | 22.6 | 3.4 | 71.4 | 30.6 | 2.5 | 93.7 | 28.9 | 1.6 | 168 |
| V | 6.5 | 0.4 | 23.9 | 5.7 | 0.7 | 21.7 | 6.3 | 0.5 | 17.2 |
| Zn | 519 | 38.1 | 3017 | 1078 | 68.7 | 4756 | 306 | 27.1 | 2685 |
| $Cl^-$ | 4.7 | 0.4 | 16.8 | 8.5 | 0.8 | 25.9 | 2.9 | 0.2 | 8.1 |
| $NO_3^-$ | 9.0 | 1.4 | 23.1 | 9.4 | 2.1 | 26.9 | 8.3 | 1.6 | 22.1 |
| $SO_4^{2-}$ | 8.3 | 2.5 | 30.0 | 8.8 | 2.2 | 29.2 | 8.8 | 3.1 | 20.9 |

is the matrix of source profile. E presents the residual matrix of calculation, and the main process of the PMF is minimizing the Q-value, which is defined in the equation (2) below as the sum of square of the residuals ($e_{ij}$) weighted inversely with error estimates ($s_{ij}$) of the data point (Paatero, 1997; Paatero and Tapper, 1994).

$$Q(E) = \sum_{i=1}^{m} \sum_{j=1}^{n} (e_{ij} / s_{ij})^2 \qquad (2)$$

The Q value can be used to determine the optical number of factor. The theoretical Q value should be approximately equal to the degree of freedom of datum in the data set. In the almost all cases, however, calculated Q value is higher than theoretical Q value because of missing and/or below detection limit data points. The more details of PMF are referred to the literatures.

The data set was then used for PMF2 to identify the possible sources of PM10 in the study area. The measured data were used directly, and the expanded uncertainty in 99 % confidence interval of each data value was used as an error estimate. For those data below the detection limit (DL), values were replaced with a half value of DL for each element, and the value of DL was used as an error estimate. For missing data, geometric mean of elemental concentration was used, and four times (of the geometric mean) were used as an error estimate to obtain large standard deviation.

A common nine sources for PM10 in the three sampling sites were found based on Q value, FPEAK, and scaled residual (<±2). Scaled residual values less than ±2 were more than 80% in the three data sets. The results of correlation analysis using observed versus predicted PM10 mass concentrations indicated that the resolved factors effectively accounted for the total mass. Correlation coefficients between reconstructed and measured portions of three data sets were higher than 0.75 in the study area. On average, the extracted eight factors from the data accounted for 97% with respect to the corresponding measured PM10 concentrations.

The natural sources of soil dust and sea salt were enriched by Al, Ca, Fe, K, Mg, and Si and Na, $Cl^-$, and Mg, respectively. The secondary aerosol source has a high concentration of $NO_3^-$ and $SO_4^{2-}$, being suspected to be the major sources of air particulate matter. The sources associated with the transportation were divided into road dust and vehicle exhaust. The former was defined as contaminated soil dust on the pave road by vehicle exhaust, particles created through tire, brake, and vehicle wear process as marker of $NO_3^-$, $SO_4^{2-}$, Ba, Cr, Sb, Si, and Ti (Abu-Allaban et al., 2003). On the other hand the latter ones were vehicle exhaust directly emitted from tailpipes as marker of Sb, Zn, Cd, Pb, and $Cl^-$. The incinerator source contains high concentration of Cd, Pb, Sb, and Zn. The elements originating from burning of coal and fuel oils (like As, Se, Mn, V, and Zn) were used for the identification of these combustion source processes (Song et al., 2001; Huang et al., 1994; Ondov et al., 1982).

In the three sites, the number of air pollution sources analyzed by PMF2 is found to consist of nine different ones. The sources included both the natural and anthropogenic ones including soil dust, sea salt, road dust, secondary sulfate, soil dust, secondary nitrate, incinerator, vehicle exhaust, coal combustion, and oil combustion. The average source contributions of each source to the PM10 concentration in each sampling sites were compared in Table 3. The results of PMF2 revealed very well the characteristics of sampling sites as its contribution for the PM10 concentration. It suggested important roles of both industrial and natural source processes, despite the fact that the study site is located at the industrial complex.

## 4. CONCLUSIONS

The airborne concentrations of PM10 and their components covering 20 metals and 3 anions were investigated from the industrial complex area in Korea. The elemental concentrations in PM10 were determined on a CCT-ICP-MS, It was found that relative errors (against SRM values) of Al, As, Cr, Fe, Mg, Mn, Pb, Sb, V, and Zn fell below 20%, while those of Ca, Si, and Ti above 20%.

Table 3. Average and standard error of source contributions for each factor at each site. (unit: $\mu g/ m^3$)

| Sources | SW | BW | JW |
|---|---|---|---|
| Road dust | 20.5±2.54 | 21.1±2.3 | 11.2±0.75 |
| Secondary sulfate | 15.3±1.65 | 15.7±1.7 | 14.5±0.50 |
| Soil dust | 10.7±0.68 | 10.4±1.0 | 14.0±0.68 |
| Secondary nitrate | 9.4±1.14 | 14.4±2.0 | 11.5±2.54 |
| Sea-salt | 7.2±0.75 | 4.3±0.6 | 4.0±0.58 |
| Incinerator | 6.5±0.92 | 7.0±1.0 | 5.0±1.14 |
| Vehicle exhaust | 6.3±1.02 | 5.9±1.0 | 3.6±1.02 |
| Coal combustion | 6.2±0.58 | 4.8±0.7 | 4.3±1.65 |
| Oil combustion | 5.8±0.50 | 6.9±1.2 | 7.8±0.92 |
| Observed PM10 | 92.2±3.12 | 91.8±3.04 | 77.0±3.0 |
| Predicted PM10 | 86.2±3.05 | 89.7±3.44 | 75.9±4.5 |

The concentration of metals such as Al, Ca, Fe, K, Mg, and Na associated with crustal sources were much higher than that of any other toxic metal. The distribution patterns of different metals were clearly distinguished with their concentrations ranging across five orders of magnitude. Based on the analysis of the metal and ionic compositions of PM10 samples, we tried to identify sources, and to assess their contributions to PM10 by positive matrix factorization (PMF 2) method. In all three sites, the number of air pollution sources analyzed by PMF2 is found to consist of nine different ones. The sources included both the natural and anthropogenic ones including soil dust, sea salt, road dust, secondary sulfate, soil dust, secondary nitrate, incinerator, vehicle exhaust, coal combustion, and oil combustion. Results of PMF2 modeling also suggested important roles of both industrial and natural source processes, despite the fact that the study site is located at or near at the very active industrial complex.

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