Investigation of Atmospheric Pollution by Biomonitoring of Major and Trace Elements in an Industrial Region

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Abstract: Spatial variations and sources of atmospheric pollution by measuring of 48 major and trace elements were determined in Iskenderun industrial region using tree components samples (pine needle, bark, branch, stem and litter) and soil samples taken from 21 industrial sites and 6 background sites. The highest elemental concentrations were found at industrial sites and generally a decreasing trend with distance was observed. For most of the crustal elements, contamination factors (CF, element concentration in polluted area/element concentration in background area) were low (< 2). For Bi, Cr, Pb, Sn, and Zn, the Iskenderun region was classified as seriously contaminated ($8 < \langle CF \rangle \leq 27$), while for several elements, including Cd, Ag, Cr, As, Fe, Mn, Sn, Mo, Se, Pb, Ni, Sb and Zn the region was classified as moderately contaminated ($3.5 < \langle CF \rangle \leq 8$). Sources of trace elements in Iskenderun industrial region were investigated using Principal Component Analysis (PCA). PCA have identified four factors as crustal-resuspended particulate matter, iron-steel plant emissions, marine aerosol, and accumulation of nutrient elements on plants. Spatial variations of anthropogenic-based trace elements supported the finding that iron-steel facilities are their major sources in the Iskenderun industrial region. Results of this study confirmed that tree components and litter can be used as passive samplers to explore the geographical distribution of atmospheric pollution.

Keywords: Major and trace elements, Biomonitoring, Needle, Bark, soil.

1. INTRODUCTION

The presence some trace elements (*e.g*., Ni, Cd, Pb, As, Cr, Hg) in the environmental compartments raise a concern because of their adverse effects (Blackmore, 1998). Past and ongoing anthropogenic activities (*i.e*., industries, traffic, fossil-fuel combustion) emit considerable quantities of trace elements into the atmosphere. Since trace elements having adverse effects occur commonly, their concentrations should be monitored systematically to explore their spatial variations and sources. To determine the spatial distributions and sources of elements at multiple sites, using of the active samplers has some limitations (*i.e*., technical, physical and economical). Sampling and analyzing tree needles and leaves (Aboal *et al*., 2004; Baycu *et al*., 2006; Onder *et al*., 2006), and bark (Catinon *et al*., 2009; Gueguen *et al*., 2012) have been utilized as an alternative convenient method to determine the geographical variation of trace elements.

Trace element concentrations in the atmosphere have been determined by measuring the particulate matters directly in the air or in deposits, by constructing models describing the distribution of pollutants, or by using bioindicators (Aboal *et al*., 2004). The monitoring methods provide an approximate description of air quality. In this case it is easier and more effective to use the distribution models or bioindicators. Bioindicators ensure useful information on both the air quality and the effect of pollutants.

Trace elements in tree components are originated from the interactions of the trees with the atmosphere and soil (Guéguen *et al*., 2012; Kirchner *et al*., 2008). Previous studies imply different uptake mechanisms. The most efficient mechanism is uptake via the roots from the soil. High concentration cations are displaced by lower concentration ions having higher cation binding affinity in the xylem that acts as an ion exchange medium. Cations presented via the sap flow are sorbed to ligands contained in the xylem walls depending on the pH that changes seasonally and with soil pH (Kirchner *et al*., 2008). The following most efficient pathway is foliar uptake: dry deposited trace elements on needle/leaf surfaces are absorbed through the cuticle and epidermis, translocated by phloem with photosynthetic products to other tissues. The third and least effective uptake pathway is absorption through the bark into cambium. Once major and trace elements entered the sap or plant tissues, factors like the chemical properties of the specific element and pH of the sap determine their distribution in the stem (Kirchner *et al*., 2008).

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A previous study at Iskenderun industrial region in Turkey has indicated that soil is polluted substantially by persistent organic pollutants (POPs) and trace elements (Odabasi *et al*., 2010). The aim of this study was to establish the concentrations of major and trace elements in components of *Pinus brutia* and *Pinus pinea* and soil samples in Iskenderun region in Turkey. Therefore, the spatial variation and sources of atmospheric pollution were determined by using tree components (needle, branch, stem, bark and litter) as passive air samplers.

2. MATERIALS AND METHODS

2.1. Sampling Sites

The study area (36°19′-36°53′N and 35°40′-36°20′E) located at north of Iskenderun city. In this area, there are many significant industrial activities such as integrated steel plant (with coke ovens, a lime plant, a sintering plant, blast furnaces and steel mills), scrap processing iron-steel plants with electric arc furnaces (EAFs), rolling mills, cement plant, fertilizer plant, and also intense vehicular traffic and busy ports (Figure **1**).

The sampling sites were selected representing regions influenced by the emissions of local industries (n = 21) and the background areas (n = 6) (Figure **1**). The site selection procedure was carried out based on initial field trips and the results of a previously conducted soil pollution study in the region (Odabasi *et al*., 2010). Tree components (bark, branch, stem, pine needles), litter from two pine species, *Pinus brutia* and

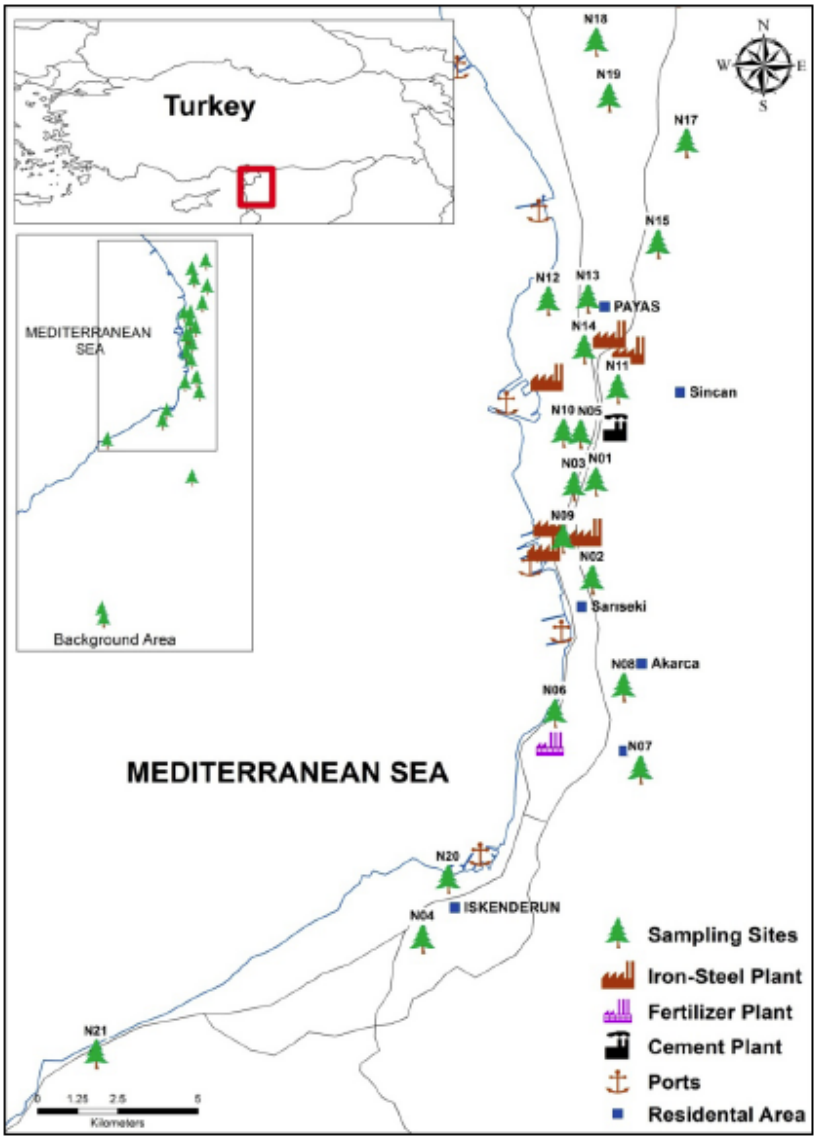


Figure 1: Locations of the sampling sites, major industries, and residential areas.

Pinus pinea and soil samples were collected in period of November 2010.

2.2. Sampling Procedure

2.2.1. Soil Sampling

Surface soil samples (10 replicates over a ~100 m^2 area) were collected from the upper 5 cm of surface, and then they were combined for each sampling site. Then, all samples were sieved (2.0 mm mesh sieve) to purge stones and plant residues. ~0.5 gr of samples for chemical analysis and 10 g of samples for determination of moisture content were used. Soil pH was determined by mixing 10 g of sample with 25 mL of deionized water, letting stand overnight, and measuring the pH of the liquid phase using a pH meter (InoLab pH 730).

2.2.2. Tree Components and Litter Sampling

Litter was mainly composed of fallen pine needles on ground. Four duplicates of litter were sampled by gathering the layer above the soil (each from 10x10 cm area). Barks were sampled by the help of a bark knife, from top 0.5 cm layer, and at 1.5 m height. Bark samples scraped from the four different sides of the stem were combined. Needle and branch samples were cut from the upper 1/3 of the tree crown using a pruning stick. Needle samples were grouped into two fractions: the 1-year (~7 months old) and 2-year needles (~1.6 years old). Tree stems were sampled using an increment borer. All collected samples were carried to the laboratory in their thoroughly cleaned glass containers, and stored at 4°C until analysis procedures. Moisture contents of the soil and litter/tree component samples were measured gravimetrically after drying subsamples for 24 h at 105°C and 60°C, respectively.

2.3. Sample Processing and Analysis

About 0.5 g of samples for all components were digested for 20 min at 190°C in a microwave digester. In digestion procedure, acid mixture of 8 mL HNO_3, 3 mL HCl and 1 mL H_2O_2 were used. Then, the samples were cooled to room temperature, their volume was adjusted to 50 mL by adding deionized water (18.2 MΩ cm^{-1}), and the extracts were filtered using a PTFE filter (Millipore, pore size: 0.45 µm). Forty eight major and trace elements (Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cu, Dy, Er, Eu, Fe, Ga, Gd, Hg, Ho, K, La, Li, Lu, Mg, Mn, Mo, Na, Nd, Ni, P, Pb, Pr, Rb, Sb, Se, Sm, Sn, Sr, Tb, Th, Tl, U, V, Y, Yb, and Zn) were analyzed with an Inductively Coupled Plasma-Mass Spectrometer (Agilent 7700x) equipped with a HMI system.

The samples were processed and analyzed under strict quality control/quality assurance procedures. Validity of the initial calibration during analysis runs was routinely checked using a verification standard solution. Blanks were processed along with samples to determine the presence/amounts of analyzed elements in the reagents. Mean blank quantity plus three standard deviations were used to calculate the method detection limit (MDL).

3. RESULTS AND DISCUSSION

Elemental levels determined in soil, litter, and tree components are shown in Tables **1**, **2** and S1. For different tree components, usually the lowest trace element levels were found in stem samples and the highest ones were found in bark. Mean concentration ratios of 2-year/1-year needles were generally higher than 1.0 (range: 0.6-10.3) that could be ascribed to higher accumulation amounts on 2-year needles because of their longer contact to the atmospheric pollutants (Odabasi *et al*., 2015; Ratola *et al*., 2010). Crustal elements (Mg, Ca, Na, Fe) had the highest levels and anthropogenic-based elements like Cd, Cu, Pb, Cr, Ni, V, and Zn followed them. Rare earth elements and lanthanides were the elements having the lowest measured levels (Tables **1**, **2** and **S1**). Appreciably higher element concentrations were found in soil and litter samples compared to those observed in tree component samples. This observation may be ascribed to the longer contact of soil and litter to pollutants in the air. Elements are directly deposited (dry/wet) from the atmosphere and also carried to soil by falling leaves/needles. As a result, soil behaves as the final sink for atmospheric trace elements. pH of the soils (7.48 ± 0.11) in Iskenderun region are slightly alkaline, as a result most of the elements have low mobility, and they are accumulated in soil.

Several previous studies conducted around the world have measured and reported trace/major element levels in tree components, especially in bark (Al-Alawl *et al*., 2007; Berlizov *et al*., 2007; Birke *et al*., 2018; Catinon *et al*., 2009; El-Hasan *et al*., 2002; Fujiwara *et al*., 2011; Gueguen *et al*., 2012; Kuang *et al*., 2007; Schelle *et al*., 2008) and needles (Aboal *et al*., 2004; Lehndorff and Schwark, 2010). Their findings were similar to the present study, *i.e*., lowest

Table 1: Trace and Macro Element Concentrations [a] in Tree Components and Litter (mg kg^{-1})

Bark					1-Yr needle				2-Yr needle			
Industrial			Background		Industrial		Background		Industrial		Background	
	AVG	SD	AVG	SD	AVG	SD	AVG	SD	AVG	SD	AVG	SD
As	0.77	0.89	0.11	0.05	0.14	0.10	0.06	0.03	0.28	0.28	0.12	0.04
B	13.4	3.2	9.9	2.49	43.2	13.8	26.5	11.2	48.5	24.1	22.6	11.9
Ba	23.7	19.6	6.7	3.6	4.9	2.6	3.9	2.8	7.0	3.9	5.7	4.1
Ca	8687	2180	5603	1870	5628	1716	4413	1981	7934	2379	7912	3107
Cd	0.9	1.4	0.15	0.09	0.15	0.08	0.14	0.15	0.26	0.23	0.15	0.14
Cu	9.0	10.9	2.96	0.766	4.2	1.6	3.8	0.5	3.9	1.2	3.0	0.38
Hg	0.021	0.013	0.014	0.003	0.053	0.029	0.053	0.021	0.031	0.024	0.056	0.011
K	334	106	262	34	4993	1153	4093	934	4464	1727	3653	1019
Na	191	131	205	201	446	357	675	680	902	710	1259	958
Pb	38.7	77	1.9	1.0	4.4	3.0	1.04	0.71	9.7	10.9	2.18	1.17
V	4.6	3.7	1.7	0.764	0.38	0.15	0.42	0.16	0.7	0.3	0.99	0.25
Zn	530	1765	5.8	5.6	59.1	37.9	28.6	14.2	210	594	37.6	26.5
Branch					Stem				Litter			
Industrial			Background		Industrial		Background		Industrial		Background	
	AVG	SD	AVG	SD	AVG	SD	AVG	SD	AVG	SD	AVG	SD
As	0.241	0.192	0.441	0.067	0.020	0.017	0.016	0.010	3.1	3.3	1.11	1.00
B	68.9	82.6	359.9	42.2	7.4	3.81	7.1	3.6	1858	1203	1302	403
Ba	5.2	2.6	2.0	2.0	1.7	0.7	1.4	1.81	34.3	32.1	16.1	15.4
Ca	7118	3278	4383	2534	1725	436	1732	492	16085	7209	11473	5286
Cd	0.31	0.25	0.18	0.23	0.041	0.030	0.07	0.081	0.9	0.9	0.25	0.35
Cu	4.0	1.3	2.6	0.63	1.25	0.42	1.3	0.5	8.2	5.8	3.9	2.6
Hg	0.014	0.008	0.011	0.004	0.0033	0.0049	0.0191	0.0252	0.030	0.015	0.013	0.005
K	1912	654	1281	404	520	86	664	89.6	1114	572	742	275
Na	231	278	271	262.8	178.1	61.7	122.0	47.9	437	368	426	356
Pb	6.2	7.0	0.53	0.22	0.30	0.42	0.16	0.08	29	29	5.5	7.33
V	1.1	1.0	3.0	0.595	0.122	0.337	0.054	0.046	21.8	25.0	14.45	13.7
Zn	58.8	144	5.3	4.1	11.7	9.2	11.0	4.9	428	988	85.0	141

[a] These elements were selected to include examples of macro/micro nutrients, crustal, and anthropogenic elements.

anthropogenic element concentrations were measured in background areas when the highest levels were observed at sites near the anthropogenic activities (urban and industrial areas).

Spatial distributions for selected major and trace element levels in 2-year pine needles (As, B, Pb, Ba, Ca, Hg, Cd, V, Cu, K, Na, Zn) in Iskenderun region are presented in Figure **2** while variations for other sample types (*i.e*., soil, litter, 1-year needles, branch, stem, and bark) are included in Figures **S1** through **S12**. For plants, P, K, Ca, and Mg take part in metabolic processes (major elements) while Fe, Cu, Zn, Mo, Mn, B, Ni, Na, and V are classified as micro and beneficial elements (or nutrients) (Graham *et al*., 2005; Havlin *et al*., 2013). In all sample types, generally the lowest trace element concentrations were found in background sites, far from local pollutant emitters when the high levels were determined at sampling sites in the near of local sources (Table **1**, Figures **2**, **S1**-**S12**).

Table 2: Trace and Macro Element Concentrations of in Soil (mg kg^{-1})

	Industrial		Background	
	AVG	SD	AVG	SD
As	41.0	37.0	27.1	5.0
B	1477	798	464	430
Ba	108	62	59	82.4
Ca	59714	41384	30763	35929
Cd	1.1	1.2	0.27	0.44
Cu	26.7	12.8	17.7	4.5
Hg	0.04	0.03	0.014	0.008
K	2293	1352	1680	1474
Na	135	69	1704	1710
Pb	66	71	6.4	6.2
V	208	87.7	246	36.1
Zn	303	437	20.9	24.6

Prevalent winds in the region are southerly (Odabasi *et al*., 2010). As a result, highest levels were generally observed at sites located north of the primary sources. These findings were especially true for anthropogenic-based elements (Pb, As, Hg, Cu, Cd, V, and Zn). Although some major elements (*i.e*., B, Ca, K, and Na) indicated spatial variations resembling to anthropogenic-based elements, the differences in their levels at different sites were not distinct. Spatial variability of these elements might be ascribed to the unpaved road fugitive particulate matter emissions surrounding the industries in the region (Kara *et al*., 2015b).

In agreement with the studies reported previously (Berlizov *et al*., 2007; Catinon *et al*., 2009; Lehndorff and Schwark, 2010; Sun *et al*., 2010), the results of this study have implied that trace element levels observed in tree components (especially bark and needles) reveal the atmospheric pollution level and they may be utilized as passive samplers to explore the geographical distributions of atmospheric trace elements in a region. The results have also suggested that branch samples that were not studied previously could also be used for passive sampling of trace elements.

Contamination factor (CF, element concentration in polluted area/element concentration in background area) has been used to assess the degree of air pollution (Berlizov *et al*., 2007). For most of the crustal elements CF values were low (< 2) according to a previously proposed scale (Fernandez and Carballeira, 2001). However, for Bi, Cr, Pb, Sn, and Zn, the Iskenderun region was classified as seriously contaminated (8 < ⟨CF⟩ ≤ 27), while for several elements, including Ag, As, Bi, Ba, Cd, Fe, Cr, Mn, Ni, Mo, Sb, Pb, Se, Sn, and Zn the region was classified as moderately contaminated (3.5 < ⟨CF⟩ ≤ 8).

Correlation analysis was used to find out the relationships of elemental levels observed in different types of samples. The correlations between tree components, litter, and soil were generally significant ($p < 0.01$) pointing out to their interactions. Significant correlations were found especially for anthropogenic-based elements (*i.e*., Ag, Cd, As, Cu, Sn, Pb, Hg, Mo, Sb, Se, and Zn) and for some crustal elements (Na, Ca, and Sr). For the majority of the elements, significant correlations were observed between bark, 1-year needles, and 2-year needles. Soil generally associated with litter but less with bark, branch and needles. This suggests that, atmospheric deposition is a more influential mechanism than soil uptake, especially for anthropogenic element accumulation on plants in Iskenderun region. Correlations of stem sample trace elemental levels with other tree components were weak. This could be ascribed to the fact that observed trace elemental levels are those integrated over the lifetime of the plant and they do not reflect the current levels like bark, branch, and needles that contain newly deposited particles from the atmosphere.

To assess the relative importance of anthropogenic and terrestrial trace element sources in different samples, the enrichment factors (EFs) were calculated as:

$$EF = CX/CAlPlant CX/CAlSoil \quad (1)$$

where C_X is the concentration of the element being investigated and C_{Al} is the reference element (Al) concentration in a soil or plant sample. Al was used as the reference element since it is one of the major components of the Earth's crust and its importance is limited for metabolic processes of plants (Achotegui-Castells *et al*., 2013). Average values of the EFs are presented in Figure **S13**. Most of the crustal elements and lanthanides had EF values ≤ 1.0 indicating no enrichment relative to local soils. In general, the largest EFs were observed for major (Ca, K, Mg, and P) or micro (B, Cu, Mo, Mn, and Zn) plant nutrient elements. Other elements like Ag, Cd, Ba, Bi, Hg, Pb, Ga, Sn, Rb,

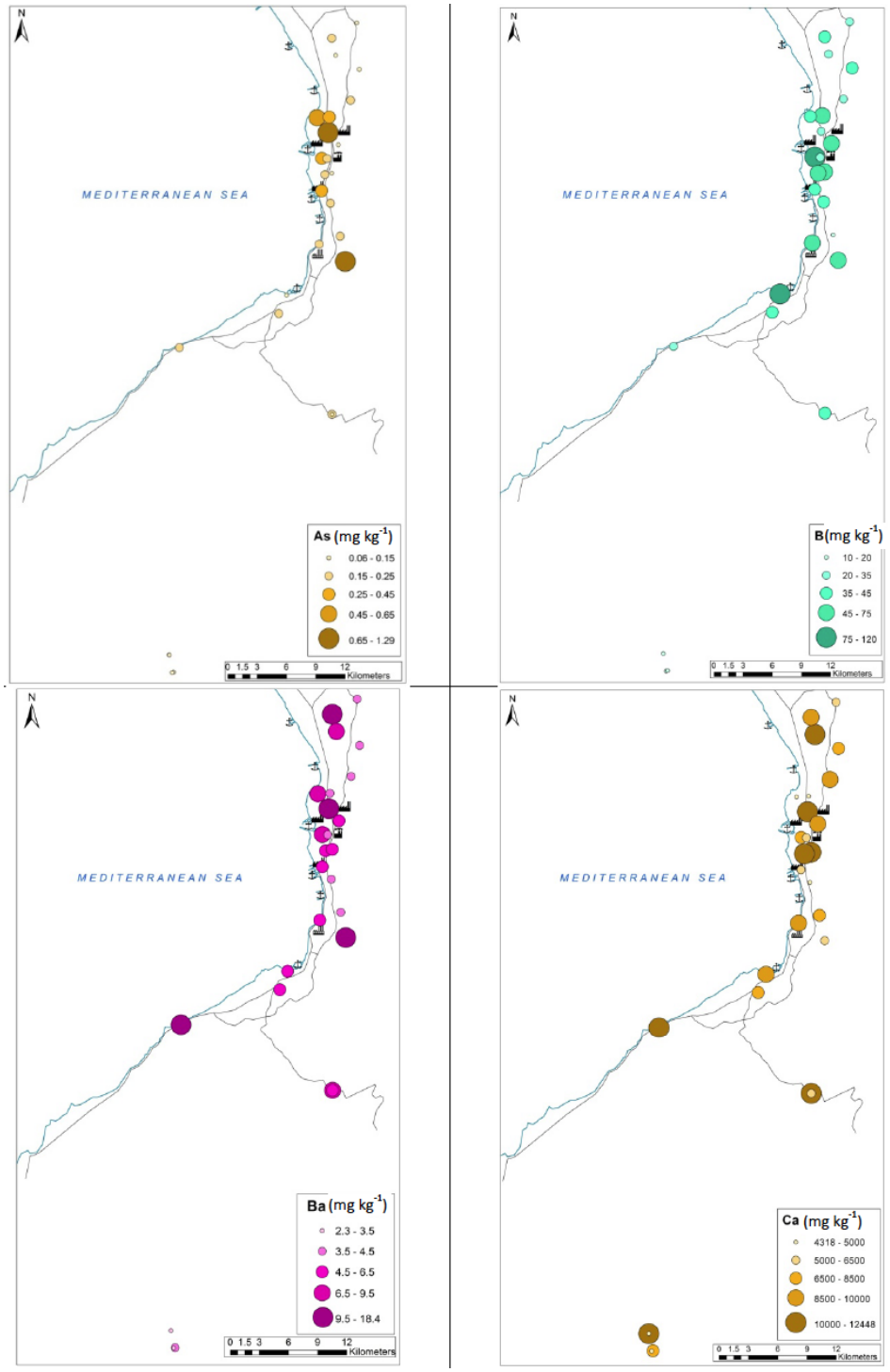
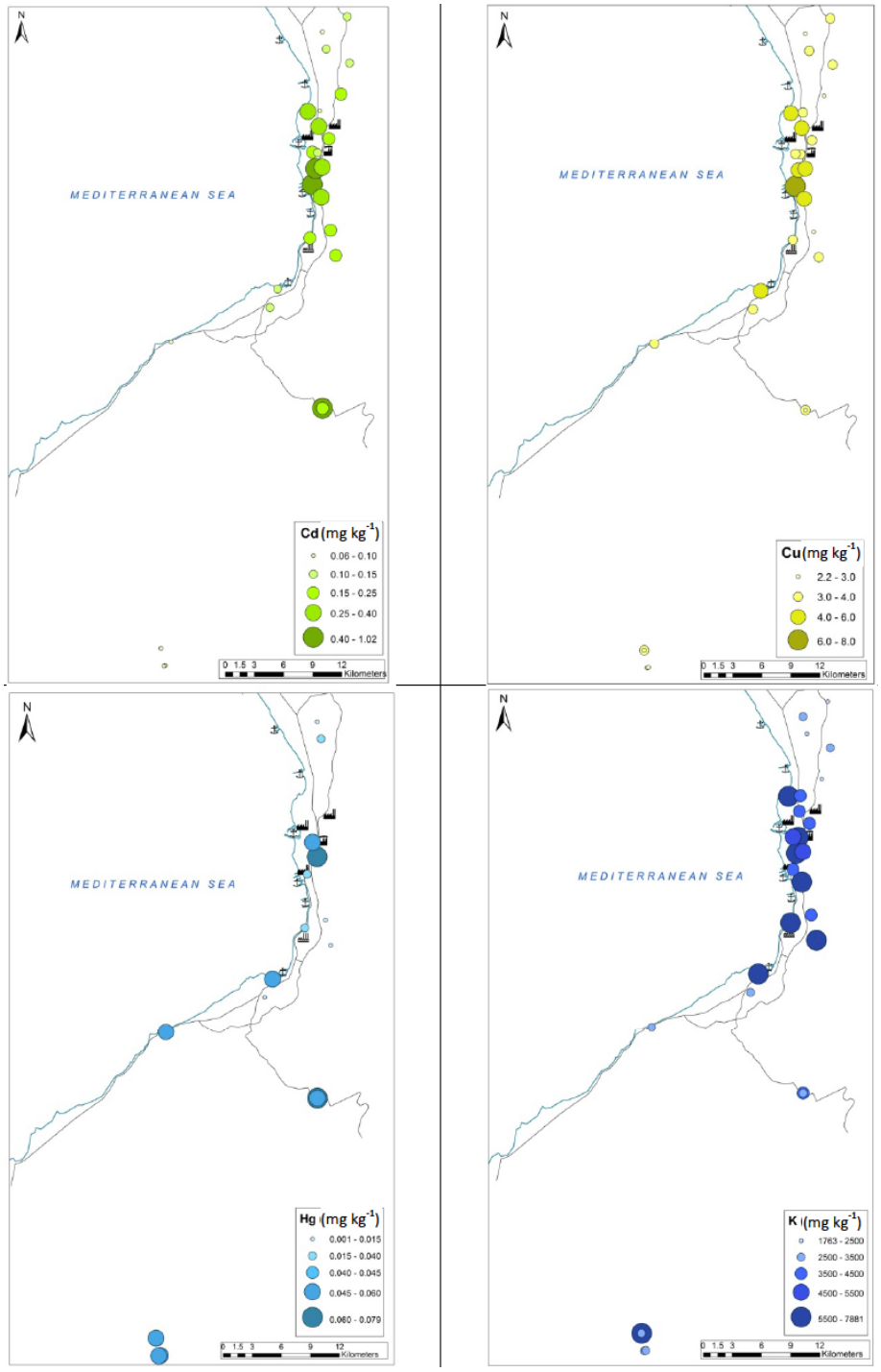


Figure 2: Spatial distribution of selected trace and macro elements in 2-year needles.

Sb, Sr, and Tl also had high EFs. Among the enriched elements there are several anthropogenic ones (Ag, Bi, Hg, Mn, Cd, Mo, Cu, Sb, Pb, Sn, and Zn). Enrichment of anthropogenic-based elements suggests that deposition from the atmosphere is the most efficient pathway for their accumulation on plants in the Iskenderun region.

Trace element sources in Iskenderun region were further investigated using Principal Component Analysis (PCA). PCA was run for sample types that all analyzed elements were detected (*i.e.*, 2-year needles, bark, branch, litter, and soil) and using the SPSS software (v20.0, SPSS Inc.). The extraction method was PCA, and the raw calculated factor loading coefficients were rotated by Varimax. During the



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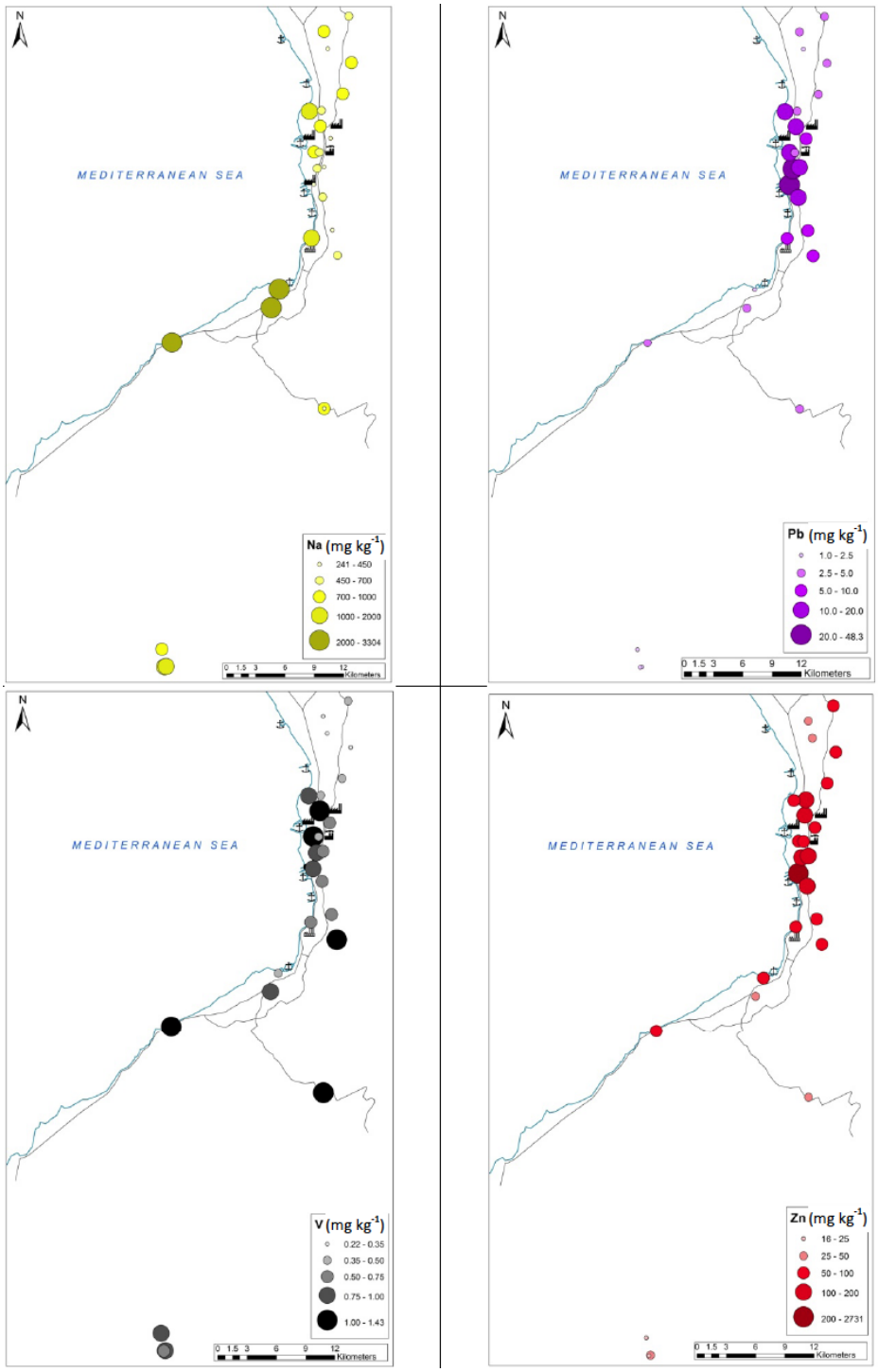
analysis, only principal components (PCs) were retained that have eigen values > 1.0 and clusters with factor loadings higher than 0.5.

Generally four principal components were extracted for major and trace elements. These components accounted for the 67.82% (2-year needles) to 93.34% (bark) of the total variance (Table **S2**). The first factor (25.5-48.9% of the variance) had high loadings on Al, Be, Ce, Ga, Gd, K, Li, Lu, Pr, Rb, Sm, Tb, Th, Tl, U, V, Y, and Yb. These elements are crustal elements and they could be accumulated by either uptake from soil or by the deposition of resuspended particulate matter onto plant surfaces. Therefore, this factor represents crustal sources. The second factor (17.6-37.4% of the variance) was dominated by Ag, Hg, As, Cu, Bi, Cd, Sn, Cr, Fe, Tl, Mn, Se, Mo, Pb, Sb, and Zn. The elements are markers for iron-steel production (Kara *et al*., 2015b; Thurston *et al*., 2011; Yatkin and Bayram, 2008) and the second factor might be ascribed to the



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iron/steel plants in the study area. The third factor (8.6-15.1% of the variance) had high contributions from Ca, Mg, Na, Sr. Recently, in a similar region (Aliaga, Turkey) the highest elemental concentrations in sea water were measured for Na, Mg, K, Ca, Sr (Kara *et al*., 2015a). Therefore, factor 3 could be associated with marine aerosol. The fourth factor (3.0-11.0% of the variance) had high loadings on B, Na, P, and Sr. B and Na are micro nutrients while P is a macro nutrient for plants. Thus, this factor represents the selective uptake from soil and accumulation of these elements in sampled plants.

PCA results obtained in this study agree well with previous studies conducted in similar regions (Cetin *et al*., 2007; Kara *et al*., 2014a, b; Kara *et al*., 2015b) indicating that one of the most important emitters for anthropogenic trace elements are the iron-steel plants.

CONCLUSIONS

Spatial variations and sources of several major and trace elements (n = 48) were explored in Iskenderun region in Turkey using pine needle, branch, bark, stem, litter, and soil samples gathered at several sites. The highest concetrations were determined at sites affected by industrial activities and generally a decreasing trend with distance was observed.

For most of the crustal elements, contamination factors (CF, element concentration in polluted area/element concentration in background area) were low (< 2). For Bi, Cr, Pb, Sn, and Zn, the Iskenderun region was classified as seriously contaminated (8 < ⟨CF⟩ ≤ 27), while for several elements, including Cd, Ag, Bi, As, Ba, Mn, Cr, Fe, Mo, Pb, Ni, Sb, Se, Sn, and Zn the region was classified as moderately contaminated (3.5 < ⟨CF⟩ ≤ 8).

Sources of trace elements in Iskenderun area were investigated using Principal Component Analysis (PCA). PCA have identified four factors as crustal-resuspended particulate matter, iron-steel plant emissions, marine aerosol, and accumulation of nutrient elements on plants.

Findings of the study confirmed that tree components and litter might be utilized as passive air samplers to explore the variations and sources of atmospheric pollution in an industrial region.

SUPPLEMENTARY MATERIAL

Supplementary material associated with this article can be found in the online version.

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