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TRACE METAL LEVELS IN VEHICLE AIR AND POLLEN FILTER DUSTS BY THE BCR EXTRACTION PROCEDURE

OCENA ZAWARTOŚCI METALI ŚLADOWYCH W POWIETRZU SAMOCHODOWYM I W ODFILTROWANYCH PYŁACH Z WYKORZYSTANIEM PROCEDURY EKSTRAKcji BCR

Abstract: The availability of Cd, Co, Cr, Cu, Mn, Ni, Pb, Fe and Zn of dust from vehicle air and pollen filters were investigated by four-step BCR (European Community Bureau of Reference) sequential extraction procedure. The acid-soluble, reducible, oxidizable, residual extracts were measured by inductively coupled plasma-optical emission spectrometry (ICP-OES). The results indicated that both of air and pollen filter dusts contained higher concentration of Fe. To estimate the accuracy of the method the standard reference material BCR 701 was used. The results for recovery all the elements were found in the range 95.4-101.3%. The mean concentrations [µg/g] of trace elements in dusts from air filters/pollen filters were: cadmium 16.72/17.56; cobalt 24.22/23.72; chromium 46.02/55.44; copper 44.92/37.67; iron 1868.03/1854.92; manganese 231.2/213.64; nickel 38.89/45.27; lead 60.99/67.17; zinc 199.58/201.25. The results obtained are in agreement with data reported in the literature.

Keywords: vehicle air filter, vehicle pollen filter, dust, trace metals

Introduction

Trace metals in the atmosphere may be present as dust particles [1]. This contamination is mostly caused by automobile engines and industry [2, 3]. Some of these trace metals important for enzymatic activities and the others were toxic when in excessive concentrations [4, 5]. Toxic elements can lead to some diseases for human health and also have an impact on plant growth [6]. They can be transported to human bodies via air dusts and plants take up by foliage [7-9]. Many techniques are available to determine the atmospheric trace metal contamination. Coskun et al [10] reported moss biomonitoring technique based on microwave digestion and flame atomic absorption spectrometry to study atmospheric deposition of heavy metals.

Halek et al [11] used glass fiber filters for collecting the air dust samples for PM trace elements analysis by inductively coupled plasma based on microwave digestion. Wu et al [12] analyzed air dust samples by three stage sequential leaching procedure and inductively

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coupled plasma optical emission spectrometry. Quiterio et al [13] reported metals in airborne particulate matter by acid digestion and inductively coupled plasma atomic emission spectrometry.

Four step BCR sequential extraction procedures have been used to extract heavy metals in dust, sediments, soil etc. [14-16]. The chemical reagents provide selectively destroyed metal bounds with targeting carbonate bound metals, iron and manganese oxide associated metals, metals bound to sulphide and organic phases, and mineral phases respectively [17]. The four fractions representing water and acid soluble, easily reduced, oxidizable and residual fraction [18].

Sakarya is an industrial city of Turkey. Air trace metal content resulting from industrial activity may affect the urban areas. In this study trace metal components in dust from vehicle air and pollen filters were investigated. These filters are useful tools to collect air dust. Filters were collected from ten different vehicle used in Sakarya region in 2010-2011 time interval.

Materials and methods

All the samples were oven-dried at 105°C for 24 h. Dried dust samples were passed through a sieve having a 200 mesh size. All the reagents used to prepare the extracting solutions were products of analytical grade quality (Merck, Germany). BCR 701 Standard Reference Material was used to certify the experimental results obtained and to evaluate the reliability of the method used [19]. ICP-OES (Spectro Analytical Instruments, Kleve, Germany), Nuve SL 350 model shaker, Nuve NF 400 model centrifuge and Schott CG 840 model pH meter were used throughout the experiments.

A four-step BCR sequential extraction procedure applied to dust samples is summarized below. 1.0 g dust was placed into 50 cm³ polypropylene centrifuge tubes. 40 cm³ of 0.11 M of acetic acid was added to the tube which was then shaken for 16 h at of 22 ±5°C and a speed of 200 rpm. The extract was separated from the solid phase by centrifugation at 4000 rpm for 15 min. The supernatant liquid was stored at 4°C prior to analysis. 40 cm³ of 0.1 M hydroxylamine hydrochloride (adjusted to pH 2 with 2 M HNO₃) was added to the residue from the bound to carbonates metals fraction (exchangeable and acid soluble). After shaking the mixture for 16 h at 22 ±3°C at a speed of 200 rpm, it was centrifuged for 15 min and then decanted into a beaker. 10 cm³ of 8.8 M H₂O₂ (pH of 2-3) was added carefully, in small aliquots, into the residue from bound to iron and manganese oxide metals fraction (reducible) in the centrifuge tube. The tube was covered loosely and digested at room temperature for 1 h with occasional shaking. The tube was then continuously digested for 1 h at 85 ±2°C in a water bath with occasional shaking for the first 30 min and the volume was then reduced to around 2-3 cm³ by further heating of the uncovered tube. Again, the covered tube was heated to 85 ±2°C and digested for 1 h before the volume in the uncovered tube was reduced almost to dryness after cooling, 25 cm³ of 1 M ammonium acetate (adjusted to pH 2 by adding of concentrated HNO₃) was added to the residue and the tube was shaken for 16 h at room temperature. The extract was separated from the solid phase by centrifugation and separated from the solid phase by centrifugation and decantation as described above and stored at 4°C. The residue (bound to mineral phases metals) from bound to sulfide and organic phases metals fraction (oxidizable) was digested in a mixture (3:1) of concentrated HNO₃ and HCl. The samples evaporated to dryness.

Again, 10 cm³ aqua regia added and the same procedure was repeated. Solutions were filtered blue band filter paper. Filtrates was completed to 50 cm³ with nitric acid and stored at 4°C [7, 20-23].

Results and discussions

In order to validation of the sequential extraction method results BCR-701 standard reference material was analyzed. The recoveries in each fraction are showed in Table 1. The results were in agreement with the certified values for all metals. The recoveries were greater than 95.4% for bound to carbonates, 96.6% for bound to iron and manganese oxide and 96.4% for bound to sulfide and organic matters fractions.

Table 1
Results of extractable trace metals [µg/g] on BCR 701 in comparison with certified values

| | Analyzed value | Certified value | Recovery [%] |
|--------------|----------------|-----------------|--------------|
| Cd | | | |
| Acid soluble | 7.67 ±0.16 | 7.34 ±0.35 | 95.7 |
| Oxidizable | 3.83 ±0.04 | 3.77 ±0.28 | 98.4 |
| Reduced | 0.28 ±0.16 | 0.27 ±0.06 | 96.4 |
| Cr | | | |
| Acid soluble | 2.32 ±0.26 | 2.26 ±0.16 | 97.4 |
| Oxidizable | 46.0 ±0.2 | 45.7 ±2.0 | 99.3 |
| Reduced | 145 ±4 | 143 ±7 | 98.6 |
| Cu | | | |
| Acid soluble | 51.7 ±2.4 | 49.3 ±1.7 | 95.4 |
| Oxidizable | 125 ±1 | 124 ±3 | 99.2 |
| Reduced | 55.0 ±0.6 | 55.2 ±4.0 | 100.4 |
| Ni | | | |
| Acid soluble | 15.8 ±1.3 | 15.4 ±0.9 | 97.5 |
| Oxidizable | 27.5 ±1.5 | 26.6 ±1.3 | 96.7 |
| Reduced | 15.1 ±2.1 | 15.3 ±0.9 | 101.3 |
| Pb | | | |
| Acid soluble | 3.30 ±1.36 | 3.18 ±0.21 | 96.4 |
| Oxidizable | 128 ±3 | 126 ±3 | 98.4 |
| Reduced | 9.6 ±1.4 | 9.3 ±2.0 | 96.9 |
| Zn | | | |
| Acid soluble | 206 ±2 | 205 ±6 | 99.5 |
| Oxidizable | 118 ±3 | 114 ±5 | 96.6 |
| Reduced | 46.9 ±1.3 | 45.7 ±4.0 | 97.4 |

Total metal contents of Cd, Co, Cr, Cu, Mn, Ni, Pb, Fe and Zn in the dusts from vehicle air and pollen filters are shown in Table 2. The results show that Fe was most abundant metal in both of filters. The concentrations of metals in air filter dust samples were in the following order: Fe > Mn > Zn > Pb > Cr > Cu > Ni > Co > Cd. These results were similar to levels found in pollen filter dust samples except Ni. The concentration of Cu was lower than Ni in pollen filter dust samples and the concentrations of metals in the following order: Fe > Mn > Zn > Pb > Cr > Ni > Cu > Co > Cd. There is a good agreement between this work and other similar studies reported in the literature. Highest concentrations of Fe also have been reported by Saracoglu et al [7] and Siddique et al [24]. The results

obtained from air and pollen filter dust samples compared air dust, soil, sediments analysis works made in Turkey in Table 2. The concentration of Cd, Co, Cr, Cu and Pb were found in vehicle air and pollen filter dust samples in this study higher than the other literature works except outdoor dust work made in Sakarya. Mn and Ni values in this study lower than sediment and outdoor dust works and Zn concentration lower than street and outdoor dust works made in Sakarya.

Table 2

Comparison of trace metals [$\mu\text{g/g}$] concentration in vehicle air and pollen filter dusts to other studies made in Turkey

| Type | Cd | Co | Cr | Cu | Mn | Ni | Pb | Fe | Zn | Reference |
|----------------------------------|------------------------|---------------------|------------------------|-------------------------|----------------------|------------------------|---------------------|-----------------------------|----------------------|------------|
| Vehicle air filter dust | 16.72 ± 0.05 | 24.22 ± 0.13 | 46.02 ± 0.06 | 44.92 ± 0.12 | 231.20 ± 0.25 | 38.89 ± 0.12 | 60.99 ± 0.12 | 1868.03 ± 0.09 | 199.58 ± 0.14 | This study |
| Vehicle pollen filters dust | 17.56 ± 0.04 | 23.72 ± 0.07 | 55.44 ± 0.09 | 37.67 ± 0.18 | 213.64 ± 0.19 | 45.27 ± 0.09 | 67.17 ± 0.08 | 1854.92 ± 0.21 | 201.25 ± 0.21 | This study |
| Roadside soil | - | - | - | - | - | 58.67 | 47.54 | - | - | [25] |
| Street dust | 0.7 | - | 10.2 | 14.2 | - | 40.2 | 25.9 | - | 205.1 | [26] |
| River sediment | 2.62 ± 0.17 | 21.21 ± 0.18 | 36.96 ± 0.20 | 37.54 ± 0.20 | 772.00 ± 0.20 | 82.96 ± 0.19 | 41.31 ± 0.16 | 2023 ± 0.21 | 76.67 ± 0.20 | [27] |
| Outdoor dust | 21 | - | 340 | 1054 | - | 718 | 1185 | - | 1862 | [28] |
| Vehicle air filter dust | 15.58 | - | - | 33.54 | 180 | - | - | 1625 | - | [7] |
| Lake sediments | - | 3.0 | 11.5 | 19.2 | 99.8 | 19.2 | 8.05 | 3276 | 34.6 | [29] |
| House dust | 1.4 ± 0.1 | 3.0 ± 0.3 | 30.9 ± 2.4 | 62 ± 5 | 48.4 ± 4 | - | 42.8 ± 3.7 | 1974 ± 18 | 225 ± 20 | [30] |
| Highway soil | 4.31 ± 4.32 | 19.7 ± 11.8 | 44.6 ± 42.4 | 22.9 ± 18.1 | 555 ± 340 | 132 ± 180 | 31.2 ± 14.1 | 2.554 ± 1.411 (%) | 103 ± 57 | [31] |
| Street dust | 2.53 | 16.5 | 29.0 | 36.9 | 237 | 44.9 | 74.8 | - | 112 | [32] |
| Faculty/ House/ Jean-market dust | 4.27/ 3.75/ 2.54 | - | 38.7/ 53.5/ 53.5 | 119.8/ 66.2/ 62.4 | - | 64.8/ 37.8/ 97.8 | - | - | - | [33] |

The mean levels and abundance percentages of the metal concentrations in air and pollen filters were shown in Table 3. The highest percentages of Cd in both of filters were found in the bound to carbonates fraction. The lowest abundance of Cr and Fe in air and pollen filters was found in this fraction. The results indicated that Mn was most abundant metal in bound to carbonate fractions. The highest release of Mn and Zn in air and pollen filters was found in this fraction. Relative abundance in each fraction of all elements in the air and pollen filter dust samples were shown in Figure 1. Cr, Fe and Pb were presents at the higher level in the reducible fraction than exchangeable fraction. Fe was most abundant metal in bound to iron and manganese oxide fractions. Cd, Co, Cu, Mn, Ni and Zn were presents at the lower concentrations under reductive condition than acidic condition. The relative abundances were of all metals in the oxidizable fraction higher than reducible and exchangeable fractions except Mn and Pb. The lowest releases of Mn in air and pollen filters were found in oxidizable fraction. The mean concentration of Pb in the oxidizable

fraction was lower than reducible fraction and higher than exchangeable fraction in air filter dust samples. The lowest concentration of Pb was found in the oxidizable fraction in pollen filters. The highest release of Cu in pollen filters was found in oxidizable fraction and presents higher concentration level than exchangeable and reducible conditions in air filter dust samples. The mean Cd, Co, Cr, Ni and Zn levels within the both of filters were higher than bound to carbonates and bound to iron and manganese oxide fractions. The highest concentrations of Co, Cr, Fe, Ni and Pb in both of filter dust samples were found in residual fraction. The results indicated that Fe was most abundant metal in bound to sulphide and organic phases fractions. The lowest Cd levels were found in both of filters in residual fraction. The highest release of Cu in air filters was found in residual fraction and presents lower concentration level than oxidizable conditions in pollen filter dust samples. The lowest level of Zn in pollen filter dust samples were found in this fraction and the level in air filters higher than reducible fraction.

Table 3
Relative amount of metals extracted from vehicle air filters of the BCR sequential extraction

| Elements | Type of filters | Acid-Soluble ^a | Ratio [%] | Reducible ^a | Ratio [%] | Oxidizable ^a | Ratio [%] | Residual ^a | Ratio [%] | Total ^b |
|----------|-----------------|---------------------------|-----------|------------------------|-----------|-------------------------|-----------|-----------------------|-----------|--------------------|
| Cd | Air Filter | 6.21±0.01 | 37.14 | 3.73±0.06 | 22.31 | 4.33±0.07 | 25.90 | 2.45±0.04 | 14.65 | 16.72±0.05 |
| | Pollen Filter | 7.54±0.06 | 42.94 | 3.90±0.14 | 22.21 | 4.07±0.10 | 23.18 | 2.05±0.06 | 11.67 | 17.56±0.04 |
| Co | Air Filter | 5.24±0.03 | 21.64 | 4.21±0.13 | 17.38 | 6.40±0.05 | 26.42 | 8.37±0.12 | 34.56 | 24.22±0.13 |
| | Pollen Filter | 5.67±0.07 | 23.90 | 4.17±0.01 | 17.58 | 5.94±0.12 | 25.04 | 7.94±0.05 | 33.47 | 23.72±0.07 |
| Cr | Air Filter | 3.57±0.05 | 7.76 | 5.64±0.09 | 12.26 | 10.27±0.09 | 22.32 | 26.54±0.06 | 57.67 | 46.02±0.06 |
| | Pollen Filter | 4.20±0.02 | 7.58 | 5.50±0.10 | 9.92 | 9.47±0.12 | 17.08 | 36.27±0.09 | 65.42 | 55.44±0.09 |
| Cu | Air Filter | 7.51±0.14 | 16.72 | 3.72±0.15 | 8.28 | 16.54±0.16 | 36.82 | 17.15±0.24 | 38.18 | 44.92±0.12 |
| | Pollen Filter | 9.12±0.09 | 24.21 | 2.47±0.02 | 6.56 | 14.54±0.20 | 38.60 | 11.54±0.21 | 30.63 | 37.67±0.18 |
| Fe | Air Filter | 34.58±0.07 | 1.85 | 218.94±0.45 | 11.72 | 487.23±0.38 | 26.08 | 1127.28±0.12 | 60.35 | 1868.03±0.09 |
| | Pollen Filter | 42.14±0.21 | 2.27 | 194.28±0.36 | 10.47 | 567.25±0.15 | 30.58 | 1051.25±0.46 | 56.67 | 1854.92±0.21 |
| Mn | Air Filter | 86.71±0.25 | 37.50 | 52.17±0.14 | 22.56 | 36.76±0.11 | 15.90 | 55.56±0.31 | 24.03 | 231.20±0.25 |
| | Pollen Filter | 91.54±0.16 | 42.85 | 47.27±0.10 | 22.13 | 28.49±0.05 | 13.34 | 46.34±0.20 | 21.69 | 213.64±0.19 |
| Ni | Air Filter | 4.82±0.09 | 12.39 | 3.27±0.06 | 8.41 | 8.63±0.08 | 22.19 | 22.17±0.15 | 57.01 | 38.89±0.12 |
| | Pollen Filter | 5.94±0.15 | 13.12 | 3.10±0.07 | 6.85 | 7.69±0.11 | 16.99 | 28.54±0.09 | 63.04 | 45.27±0.09 |
| Pb | Air Filter | 5.40±0.13 | 8.85 | 9.47±0.10 | 15.53 | 7.61±0.14 | 12.48 | 38.51±0.13 | 63.14 | 60.99±0.12 |
| | Pollen Filter | 8.57±0.05 | 12.76 | 9.50±0.16 | 14.14 | 8.54±0.08 | 12.71 | 40.56±0.05 | 60.38 | 67.17±0.08 |
| Zn | Air Filter | 64.32±0.21 | 32.23 | 37.91±0.20 | 18.99 | 52.64±0.23 | 26.38 | 44.71±0.12 | 22.40 | 199.58±0.14 |
| | Pollen Filter | 71.76±0.20 | 35.66 | 42.50±0.21 | 21.12 | 47.27±0.22 | 23.49 | 39.72±0.19 | 19.74 | 201.25±0.21 |

^a mean [µg/g], standard deviation (SD)

^b total concentration of the four fraction ±SD

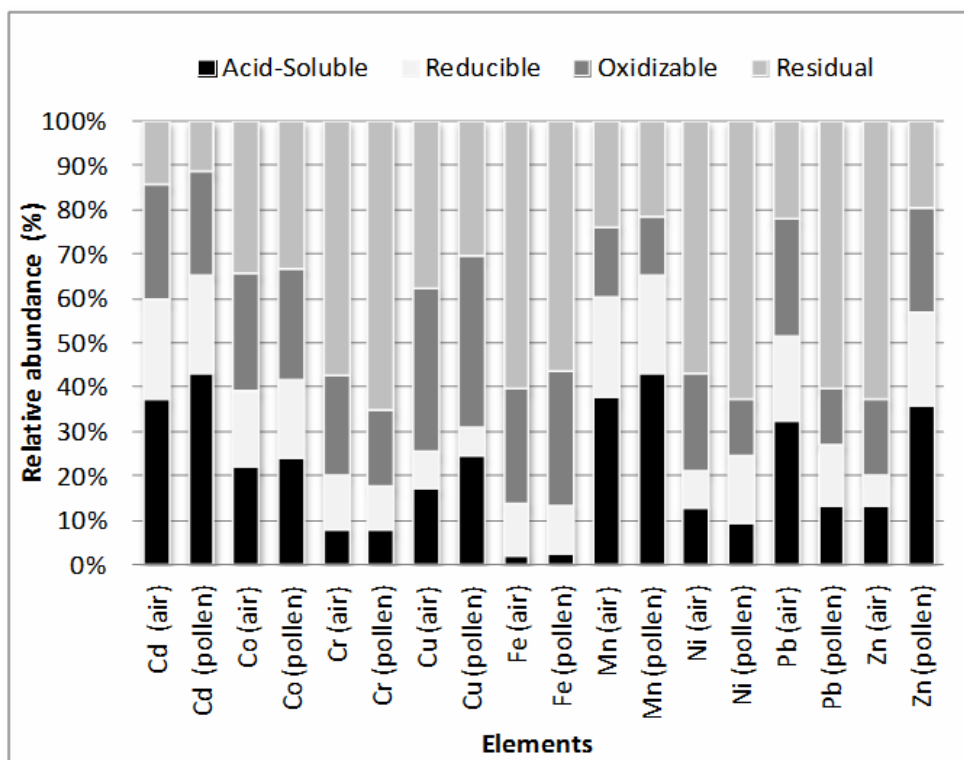


Fig. 1. Relative abundance in each fraction of all elements in the air and pollen filters dust samples

Conclusion

The highest percentages of Cd, Mn and Zn were found in acid soluble fraction indicating that these metals were bound to carbonates and the mean concentrations [$\mu\text{g/g}$] of these elements in dusts from air filters/pollen filters were: cadmium 6.21/7.54; manganese 86.71/91.54; zinc 64.32/71.76. Co (air filters/pollen filters: 34.6%/33.5%), Cr (air filters/pollen filters: 57.7%/65.4%), Fe (air filters/pollen filters: 60.4%/56.7%), Ni (air filters/pollen filters: 57.0%/63.0%) and Pb (air filters/pollen filters: 63.1%/60.4%) were mainly found in residual fraction which associated in the mineral structures. Cu was dominant in oxidizable and residual fractions indicating that bound to sulfide and organic matter. The calculated concentrations [$\mu\text{g/g}$] of Cu in dusts from air filters/pollen filters were: 16.54/14.54 in oxidizable and 17.15/11.54 in residual fractions.

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OCENA ZAWARTOŚCI METALI ŚLADOWYCH W POWIETRZU SAMOCHODOWYM I W ODFILTROWANYCH PYŁACH Z WYKORZYSTANIEM PROCEDURY EKSTRAKЦИИ BCR

Abstrakt: Zbadano zawartość Cd, Co, Cr, Cu, Mn, Ni, Pb, Fe i Zn w pyłach powietrza z pojazdów i w filtrach pyłowych za pomocą sekwencyjnej procedury ekstrakcji BCR (*European Community Bureau of Reference*). Skład ekstraktów zawierających substancje rozpuszczalne w kwasach, redukowalne i utlenialne był badany za pomocą metody indukcyjnie wzbudzonej plazmy sprzężonej z optyczną spektroskopią emisyjną (ICP-OES). Wyniki wskazują, że zarówno powietrze, jak i materiał z filtrów pyłowych zawierały zwiększone stężenia Fe. Do oszacowania dokładności metody wykorzystano standardowy materiał referencyjny BCR 701. Odzysk wszystkich pierwiastków mieścił się w zakresie 95,4-101,3%. Średnie stężenia [µg/g] pierwiastków śladowych w pyłach z filtrów powietrza/filtrów pyłowych wynosiły dla kadmu 16,72/17,56; kobaltu 24,22/23,72; chromu 46,02/55,44; miedzi 44,92/37,67; żelaza 1868,03/1854,92; manganu 231,2/213,64; niklu 38,89/45,27; ołowiu 60,99/67,17; cynku 199,58/201,25. Uzyskane wyniki są zgodne z danymi literaturowymi.

Słowa kluczowe: samochodowy filtr powietrza, samochodowy filtr przeciwpylowy, pył, metale śladowe