Assessment of ecological pollution of heavy metals in surface soils of different sites within northwest of Iraq

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Abstract
Heavy metals pollution in surface soils of different urban areas in northwest Iraq were investigated. Correlation between screened metals' spatial distribution (33 elements) and point and non-point pollution sources were performed. Obtained results showed that the levels of 15 elements (Hg, Ag, Cd, Sb, Sr, Ti, Mo, Rb, Sc, Zn, Ni, Cr, Cs, Te and Ca) exceeded the reference values of urban soil constituents. Higher observed levels of these metals, indicating that the urban soils of the studied areas are enriched with these contaminants. Further, higher concentrations of some "unexpected" toxic metals were detected in the studied locations. Estimated correlation coefficients of metal concentrations suggested three potential sources for soil pollution by heavy metals in the surveyed region. Different industrial activities, dominated by petroleum and petrochemical industries, are likely to be responsible for the releases of many heavy metals, e.g. Ni, Cr and Cu. Military activities were considered an important source for some toxic metals, e.g. Zr, Rb, V, and As, since these metals were observed at high levels in the studied sites. Trace metals such as Cu, Pb, Zn are found to be correlated with agricultural practices. However, the higher observed concentration of Ca in the surveyed locations can be attributed to natural processes' impact. Overall, this research's outcomes can be useful for the policymaking efforts dedicated to solving complex environmental problems related to soil pollution and being a guide for soil quality in northwest Iraq.

Keywords: Heavy metals pollution; Surface soil; spatial distribution; Northwest of Iraq

1. Introduction
Urban soil plays a potential role in the food chain and hence is crucial for human health since it is a very important pathway for different pollutants. In many developing countries, rapid urbanization is strongly related to socio-economic growth and affects the living environment's biogeochemical composition [1]. Soil compaction, loss of organic matter, pesticides and fertilizers, atmospheric deposition, structural changes and infection of pathogenic microorganisms are some of the several competed and interacted adverse processes that influence soils' functionality in urban areas [2][3]. Urban soil pollution may result from the accumulation of anthropogenic contaminants [4]. However, spatial distribution patterns of soil pollution around local emitters are much more heterogeneous [5][6]. For instance, severe heavy metal contamination in urban soils by anthropogenic sources was reported from areas surrounding industries [7][8]. An inverse relationship between the increase in pollutant concentrations and the source's distance is because contaminant levels in these areas' surface soils are significantly higher than the reference concentrations [9][10]. However, environmental risk assessment of pollution depends not only on the soil's total metal content but also on their mobility and bioavailability controlled by soil texture and other physicochemical properties [11][12].

Different anthropogenic sources for soil pollution exist, and therefore, the synergistic effects of emitted contaminants may deteriorate the situation. For example, areas of armed conflict in Northwest Iraq have exposed to sequenced military activities as part of the was against terrorism after 2003.
Consequently, a wide range of military wastes was emitted/deposited into these areas, alongside other industrial emissions, which resulted in unprecedented damages for the ecological systems and human health [13, 14]. Heavy metals are one of these contaminants accumulated in soils and increased the risk of causing a range of diseases (e.g., cancers and affected infants' birth for inhabitants living around) [15].

Salah Al-Deen province is a densely populated city located in Iraq Northwest (see Figure 1). It includes one of the largest petroleum complexes constructed in the Baji District of the City at the end of the 1970s. The petroleum complex comprises petroleum refinery and other oil facilities where different chemical agents, including heavy metals-based chemicals, are used in refining processes. Further, larger parts of Salah Al-Deen province have witnessed an intensive use of military weapons containing chemical agents, e.g., trace elements (Arsenic, Copper lead and zinc), explosives or radioactive elements, during military activities after 2003. Therefore, national concerns over adverse environmental impacts and health effects for ecosystems and residents have increased in recent years. However, there is limited information on urban soil contamination by heavy metals in this area at present. This research aims to assess the degree of heavy metal contamination in surface soil in different sites within Salah Al-Deen province and investigate the spatial distribution of multielement and possible sources.

Figure 1. Soil sampling points in the study area

2. Materials and method

2.1 Study area
Province Salah Al-Deen is a part of the desert environment of western Iraq of which comprises surface areas of 24,363 km² (see Figure 2). The city's geographic location represents an extension of the vast Arabian desert dominated in the Arabian Peninsula's central plain. The climate and geography of Salah Al-Deen Province have always been dominated by semi-arid and arid (with more than 50% arid area) weather conditions. The province is typically characterized by large industrial investments, urban land use and some rural areas. Geomorphologically, the survey region is characterized by two distinct zones. The first zone is the Beji district, characterized by hilly terrain, where the largest petroleum complex in Iraq is located. Beji district is a populated area (a total population of approximately 173,000 inhabitants [16]. Large industrial activities, including petroleum and petrochemicals, vegetable oils industry,
fertilizers production, and power generation plants, are predominant in the Baji district. The second zone is the Al-Oja district, where the urban centre is located and which is mostly flat. Al-Oja district is located immediately west of the Tigers river passing through Salah Al-Deen province, with 3800 inhabitants [16]. Both districts witnessed several dramatic military activities after 2003. Consequently, loads of military wastes containing different heavy metals were emitted and deposited, leaving a legacy on the province's landscape.

2.2 Soil Sampling and Analysis

Two urban districts of Salah-Aldeen province were surveyed for soil contamination by heavy metals, Beji (BG) and Tikrit (T). A total of 132 soil samples were collected from 44 locations in Salah-Aldeen province. Seventy-five samples were collected from 25 sites around the petroleum complex in the Baji district. Sampling sites were selected randomly around petroleum installation to reflect different distances from the point source pollution. Fifty-seven soil samples were collected from 19 sites from different land-use types in the Tikrit district to qualify for heavy metals contamination. Composite surface soil samples (at a depth of 0-20 cm) were collected by mixing three sub-samples from each sampling point. On-site, all samples were sieved to remove unwanted materials and stored in polyethylene bags. For analytical analysis, soil samples were dried, grounded, homogenized and screened for 33 macro and micro-elements using X-ray fluorescence spectrometry (Niton XL3t 950 XRF Analyzer) (Mo, Zr, Sr, U, Rb, Th, Pb, Au, Se, As, Hg, Zn, W, Cu, Ni, Co, Fe, Mn, Cr, V, Ti, Sc, Cu, K, S, Ba, Cs, Te, Sb, Sn, Cd, Ag and Pd). For quality control and quality assurance, precision and accuracy were checked using referenced soil samples and triplicated samples.

2.3 Data Analysis

The frequency distribution of the heavy metals in topsoil samples was analyzed using descriptive statistics. Correlation coefficients were employed, use simple correlation analysis to explore the relationships among screened heavy metals. Therefore, a link between the spatial distribution of heavy metals and their potential sources were developed. A significance level of p < 0.05 was considered to reflect significant differences between examined variables. All Statistical analysis was performed Microsoft Excel 2016 and R for Windows (RStudio v. 1.1.453. Inc., Boston, USA).

3. Results and discussion

Mean concentrations and background levels of heavy metals in the urban soils in Salah Aldeen province are shown in Table 1. Multielement concentrations in topsoil are categorized into six groups based on their concentration in order to simplify results description and interpretation. Group A consisting of eight trace metals (U, Th, Au, Se, Hg, W, Co and Pd) was observed at the lowest concentrations. Heavy metal concentrations of group B (Mo, Rb, Pb, As, Cu and Sc) and group C (Ag, Cd, Sn, Sb and V) were notably higher than group A species. Though soil content of multi-elemental forms belonged to group D (Zr, Zn, Ni, Cr, Cs and Te) was relatively higher than group C parameters. However, these metals' concentration was considerably lower than those from group E (Sr, Mn, Ti and Ba). Total soil content of Fe, Ca, K, and S (group F) appeared at the highest level than other heavy metals.

The soil content of heavy metals was anticipated to vary from one sampling point to another depending on the distance between surveyed locations and pollution sources [17]. Therefore, due to heterogeneity of variances, high standard error of mean and coefficient of Variation of heavy metal concentrations accounted for such variability (Table 1).

Obtained results indicated that most elements concentrations presented in group A were observed within element background values of natural soil, except for the mercury that appeared at a relatively high level (0.22 mg kg\(^{-1}\)) in soil samples [18]. The mean concentrations of Mo, Rb, and Sc in group B were in sites surveyed. However, it was difficult to assess Rb and Sc data against regulatory limits due to a lack of information. However, the soil content of Pb, As, and Cu complied with the environmental quality standard for soils. Average Ag, Cd and Sb levels in group C exceeded reference concentrations, but Sn and V levels were within reference values. Except for the Zr level, all heavy metals in group D,
including Zn, Ni, Cr, Cs, were above standard urban soils. The results showed that both Sr and Ti concentration were markedly higher than standard values in group E metals. Still, manganese and barium concentration have not indicated significant differences with the standard limits. Calcium’s total soil content was higher than benchmark concentration compared to Fe, K and S concentration presented in group E.

Table 1. Statistical metrics of heavy metals concentrations in surface soils of Salah Al-Deen province (mg kg⁻¹).

|        | Mean | SEMᵃ | BCᵇ | LODᶜ | CVᵈ | Mean | SEMᵃ | BCᵇ | LODᶜ | CVᵈ |
|--------|------|------|-----|------|-----|------|------|-----|------|-----|
| Group A |      |      |     |      |     |      |      |     |      |     |
| U      | 0.0  | 0.0  | 5.0 | 0.01 | 0.0 | Mo   | 4.46 | 2.65 | 2.0  | 0.42 | 3.36 |
| Th     | 0.78 | 0.32 | 1.0 | 0.01 | 2.33| Rb   | 32.73| 1.41 | *    | 0.05 | 0.24 |
| Au     | 0.0  | 0.0  | *   | 0.01 | 0.0 | Pb   | 10.37| 2.24 | 19.02| 0.02 | 1.22 |
| Se     | 0.0  | 0.0  | 0.46| 0.02 | 0.0 | As   | 2.72 | 0.65 | 6.75 | 0.01 | 1.36 |
| Hg     | 0.22 | 0.25 | 0.07| 0.01 | 6.63| Cu   | 22.27| 3.82 | 22.22| 0.27 | 0.97 |
| W      | 1.02 | 1.19 | *   | 0.01 | 6.63| Sc   | 35.27| 10.32| *    | 0.02 | 1.65 |
| Co     | 0.0  | 0.0  | 8.96| 0.05 | 0.0 |       |      |      |      |      |      |
| Pd     | 1.10 | 0.73 | *   | 0.01 | 3.78|       |      |      |      |      |      |
| Group B |      |      |     |      |     |      |      |     |      |     |
| Ag     | 5.18 | 1.34 | 0.71| 0.002| 1.46| Zr   | 114.31| 7.40 | 130  | 0.05 | 0.36 |
| Cd     | 7.29 | 1.87 | 0.38| 0.01 | 1.45| Zn   | 82.91| 12.90| 58.1 | 0.01 | 0.88 |
| Sn     | 20.47| 2.21 | 0.50| 0.01 | 0.61| Ni   | 89.48| 9.12 | 18.47| 0.01 | 0.57 |
| Sb     | 32.42| 3.63 | 1.12| 0.02 | 0.63| Cr   | 158.9| 14.22| 48.93| 0.02 | 0.50 |
| V      | 50.09| 4.45 | 73.36| 0.05 | 0.50| Cs   | 64.79| 4.55 | 3.0  | 0.02 | 0.39 |
|       |      |      |     |      |     | Te   | 98.65| 9.54 | 0.001| 0.05 | 0.54 |
| Group C |      |      |     |      |     |      |      |     |      |     |
| Ag     | 5.18 | 1.34 | 0.71| 0.002| 1.46| Zr   | 114.31| 7.40 | 130  | 0.05 | 0.36 |
| Cd     | 7.29 | 1.87 | 0.38| 0.01 | 1.45| Zn   | 82.91| 12.90| 58.1 | 0.01 | 0.88 |
| Sn     | 20.47| 2.21 | 0.50| 0.01 | 0.61| Ni   | 89.48| 9.12 | 18.47| 0.01 | 0.57 |
| Sb     | 32.42| 3.63 | 1.12| 0.02 | 0.63| Cr   | 158.9| 14.22| 48.93| 0.02 | 0.50 |
| V      | 50.09| 4.45 | 73.36| 0.05 | 0.50| Cs   | 64.79| 4.55 | 3.0  | 0.02 | 0.39 |
|       |      |      |     |      |     | Te   | 98.65| 9.54 | 0.001| 0.05 | 0.54 |
| Group D |      |      |     |      |     |      |      |     |      |     |
| Sr     | 338.48| 36.93| 200 | 0.05 | 0.61| Fe   | 18813.4| 1259.1| 24088| 0.04 | 0.37 |
| Mn     | 454.88| 23.60| 494.36| 0.01 | 0.29| Ca   | 77932| 4487.1| 14954| 0.01 | 0.32 |
| Ti     | 1688.48| 120.65| 18.47| 0.01 | 0.40| K    | 8870.7| 583.05 | *    | 0.04 | 0.37 |
| Ba     | 472.03| 62.48| 480.8 | 0.05 | 0.74| S    | 12787.1| 4218.2 | *    | 0.01 | 1.86 |

ᵃStandard Error of Mean, ᵇReference Concentrations, ᶜLower Limit of Detection (mg kg⁻¹), ᵈCoefficient of Variation (%).

Correlation analysis was employed as an interpretive tool to develop understandings of the connection between the spatial distribution of heavy metal concentrations and possible sources for pollution in the studied areas (see Figure 2).

Estimated correlation coefficients have indicated significant positive correlations (p < 0.05) among different heavy metal concentrations in topsoils for the surveyed regions. As shown in Figure 2, there were strong positive correlations among Zr, Ni, Fe, Mn, Cr, Rb, K, V and Ti concentrations in topsoils. Another significant positive correlation was observed among a concentration of Cadmium, Ag, Cs, Te, Sb and Sn (P < 0.05). Also, strontium, Sc, Ca and S were closely associated with each other. Both copper and arsenic have shown a slight correlation with Zr, Ni, Fe, Mn, Cr, Rb, K, V and Ti concentration. Also, copper concentration is related to soil content of Pb, Ba and Zn from one side and with Mo from another side. Zinc is slightly correlated with Zr, Fe and Cr concentration.

On the other hand, some negative relationships between elements concentration in surface soils were observed. Lead was negatively correlated with Cs, Te, Sb and Sn concentration. Similarly, sulfur’s soil content was associated with Zr, Ni, Fe, Mn, Cr, Rb, K, V and Ti concentrations. Strong positive correlations could be attributed to the association between metal and environmental processes [5].

Figure 2.
Figure 2. Relationships of heavy metal concentrations in the topsoil of Salah Al-Deen province.

Significant correlations among multielement studies could also be employed as valid evidence for metals derivation from the same pollution sources in Salah Al-Deen province [10, 19]. Three correlated clusters of heavy metals can be determined based on correlation analysis, as shown in Figure 2. The First group consisted of Zr, Ni, Fe, Mn, Cr, Rb, K, V, Ti, Cu and As. The second group includes Cu, Pb, Zn, and Ba. The third group is included of Cd, Ag, Cs, Te, Sb and Sn, Sr, Sc, Ca, and S. Strong elemental association in these clusters could be implicitly indicative for common pollution sources of heavy metals in the surveyed region. Therefore, it can propose three-pointed and non-pointed contamination sources for heavy metal pollution in Salah Al-Deen province's surface soil.

As reported in the introduction, military activities could be one of the most potent sources of heavy metal pollution in Salah Al-Deen province. Multi-elemental analysis performed using X-ray fluorescence spectrometry revealed high concentrations of toxic heavy metals such as Ni, Cr, Mn, Ti, and Cu associated with an "unexpected" presence trace element, e.g. Zr, Rb, V, and As. These elements are usually associated with human-made activities indicating point source contamination and probably related to military activities [20]. Industrial activity, mainly petroleum and petrochemical industries in the beji district in Salah Al-Deen province, and their pollutant emissions and discharges could be among the most important point sources for trace metals. Intensive use of rare substances such as heavy metals, chemical weapons, explosives, and radioactive elements is common practice during military activities and operations [21]. Findings presented in this research confirm the significant increase of trace metals detected in the surveyed region compared with environmental quality standards for soils by US EPA. Moreover, the average values of these metals in Salah Al-Deen province's surface soil found to be great differences quantitatively and qualitatively compared with pristine, domestic, agricultural and industrial territories in Iraq. Therefore, it is very likely to attribute the elevated concentrations of the over-mentioned elements to military activities.

The second source of heavy metal pollution is very likely to be related to the everyday agricultural activities in the Salah Al-Deen province, which was thought to be responsible for increasing some types of elements rather than others. Obtained results indicated high levels of Cu, Pb and Zn in topsoil within study sites. During agricultural practices, uncontrolled use of fertilizers containing different trace elements as additives, e.g. Cu, Pb, Zn and Cd, are widely regarded as important sources for soil
contamination by heavy metals in rural areas [22]. Further, intensive use of rich-organic fertilizers like manures and sewage sludge besides pesticides to optimize yield efficiency are also considered potential sources for soil pollution by Cu, Pb, Zn, and Cd [23]. Moreover, it is well known that organic matter's soil content can play a potential role in preserving multi-elemental composition in soil due to the high affinity between these metals and organic matters [5].

The highest levels of the major elements (e.g. Ca and S) in the surveyed locations are likely related to natural origin. However, the results showed that these metals were positively correlated with residuals of artificial-based elements such as Cd and Sr that can be found in the territories of military grounds or "armed conflict areas". Soil parent materials and different geochemical processes such as pedogenesis have been documented as the main natural sources for maco-elements in soil [24][25]. Though both major elements and trace elements are commonly adsorbed onto smaller-sized soil particles (e.g. clay particles), some maco-elements such as Ca be associated with large-sized soil particles like carbonated materials [26].

4. Conclusions

Obtained results have shown that most heavy metals examined were higher than Environmental quality standards for soils. In particular, elevated levels of some toxic metals alongside the presence of "unexpected" trace metals at relatively high concentrations in topsoils (e.g. Zr, Rb, V, and As) could be caused by human activity sources, possibly associated with military activity. These results may highlight military activities as a potential anthropogenic source for toxic heavy metals besides classical pollution sources presented and discussed in the literature reviews. Findings presented in this research provide environmental assessment for a wide range of heavy metals in surface soil in Salah AlDeen province in Iraq. They could also be used as guidance by stakeholders and decision-makers in designing strategic plans for soil restoration processes.

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