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# EXTRACTABILITY AND AVAILABILITY OF SOME NUTRIENTS IN CONTAMINATED SOILS OF ASSIUT.*

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**Abstract:** Various extraction procedures were employed for measuring extractable concentrations of potential micronutrients and toxic elements in polluted soils which were irrigated by sewage waste waters for a long time (>45 years).

The extractability of Fe, Zn, Mn, Cu, Cd, Ni and Cr in six contaminated soils near Assiut city using eight different extraction procedures, (HCl + H2SO4, DTPA, AB-DTPA, EDTA, EDTA-Aac, EDTA-AAAc, EDTA-Ammonium carbonate and EDTA-Ammonium Caitrate) as well as total element contents were studied. Extractability of metals from soils samples varied depending on types of metals and extractants used.

Our results emphasized that DTPA extractant may not be a good extractant for heavy metals, and its use for extraction of heavy metals from contaminated soils is questionable. EDTA extraction procedures were not specific for all micronutrients and heavy metal extractions. These results lead to the general conclusion that no one extraction procedures is suitable for testing the status of all metals in all soils after all crops. Precautions should be taken when deciding which extraction solution should be used.

**key words**: extractability, availability, nutrients, contaminated soils.

## Introduction

Heavy metals pollution of soils is increasingly becoming a global problem with the development of industry, irrigation of using wastewater and the application of sewage sludge even if it is relatively localized at present. The soil-plant system is the fundamental constructive unit of the geosphere and biosphere. Therefore, heavy metals pollution of soil has an important influence not only on the yield and quality of crops, but also on the quality of atmospheric and aquatic environment, and even on the health of human beings via food chains. Trace metals in soils may exist in different chemical forms or ways of binding. In unpolluted soils or sediments trace metals are mainly bound to silicates and primary minerals forming relatively immobile species, whereas in polluted ones trace metals are generally more mobile and bound to other soil or sediment phases.

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In environmental studies the determination of the different ways of binding gives more information on trace metal mobility, as well as on their availability or toxicity in comparison with the total element content. However, the determination of the different ways of binding is difficult and often impossible. Different approaches are used for soil and sediment analysis. Many of them focused on pollutant desorption from the solid phase, others are focused on the pollutant adsorption from a solution by the solid phase. Among those approaches based on desorption; leaching procedures are the most widely accepted and used.

Extraction procedures by means of a single extractant are widely used in soil science. These procedures are designed to dissolve a phase whose element content is correlated with the availability of the element to the plants. This approach is well established for major elements and nutrients and it is commonly applied in studies of fertility and quality of crops, for predicting the uptake of essential elements, for diagnosis of deficiency or excess of one element in a soil, in studies of the physico-chemical behavior of elements in soils and for survey purposes. To a lesser extent they are applied to elements considered as pollutants such as heavy metals. The application of extraction procedures to polluted or naturally contaminated soils is mainly focused to ascertain the potential availability and mobility of pollutants which is related to soil-plant transfer of pollutants and to study its migration in a soil profile which is usually connected with groundwater problems.

During the last decades several extraction procedures for extracting heavy metals in soils have been developed and modified. In this respect, two groups of tests must be considered: the single reagent extraction test, (one extraction solution and one soil sample), and the sequential extraction procedures, (several extraction solutions are used sequentially to the same sample) although this last type of extraction is still in development for soils. Both types of extraction are applied using not only different extracting schemes but also different laboratory conditions. This lead to the use of a great deal of extraction procedures. The transfer of heavy metals from soils to plants is dependent on three factors: the total amount of potentially available elements (quantity factor), the activity as well as the ionic ratios of elements in the soil solution (intensity factor), and the rate of element transfer from solid to liquid phases and to plant roots (reaction kinetics) (Bruemmer *et al,* 1986). Because correlations were found between the soluble heavy metals concentrations in the soil and the heavy metals concentration in plants, several countries passed legislation establishing quality standards based on soluble heavy metals concentration in the soil. These concentrations must be reduced below maximum threshold levels to avoid reduction in plant growth or nutritional quality. Moreover, the leaching of metals into the ground water must be minimized.

This research was designed to determine the extractability of some micro- and heavy metals in soils of six villages irrigated with sewage waste waters for along period (> 45 years) near Assiut city using different single extraction methods as well as total. In the other part of this study, the metals uptake by corn and wheat plants is investigated and correlated with the nutrient contents in the polluted soils. The obtained data when applying these testes are used for decision makers in topics such as land use of soil or in countermeasures application.

## Materials and Methods

### Study area:

Madabegh county wastewater management system uses aeration, short- term storage and slow rate irrigation. Over 90% of the effluent originates from domestic sources, most of which are nonorganic chemical manufacturing and chemical related. An average of $80\times10^3$ $m^3/d$ of wastewater is conveyed to the system by collection network of gravity sewers and pumping stations. The wastewaters have been applied to the fields, through flood irrigation system, to a big area of 8000 – 10000 feddan (1 Feddan = 1.038 acre) for a long time (40-45 years) without any other pretreatment.

### Soils and Water samples:

Along the irrigation cannel six locations (Madabegh, Mankabad, Ellwan, Bani Hussien, Bani Ghalib, Gahdam) represented the diversity in soil properties of the studied area were chosen. Each location was represented by three different sites from which representative composite samples were collected. Eighteen soil profiles (6 locations, of three sites each) were made and samples from surface (0-25 cm) and subsurface (25-50cm) layers were collected. The collected soil samples were air dried, crushed and passed through a 2mm sieve and kept for analysis. Soil analysis for certain soil properties (particle size distribution, E.C., pH, O.M.%, Ca, Mg, N, P, K and heavy metals were measured in each sample) of both layers and summarized in Tables (1and 2).

**Table(1):** Particle size distribution and texture grade of the studied soils.

| Location (Village) | Sites (Profile) | Depth (cm) | Clay | Silt | Sand | Texture grade@ |
|---|---|---|---|---|---|---|
| | | | % | | | |
| El Madabegh | 1 | 0-25 | 2.65 | 4.34 | 93.00 | S |
| | | 25-50 | 5.70 | 2.40 | 91.90 | S |
| | 2 | 0-25 | 5.87 | 3.80 | 90.33 | S |
| | | 25-50 | 3.07 | 3.00 | 93.93 | S |
| | 3 | 0-25 | 8.85 | 5.00 | 86.14 | S |
| | | 25-50 | 5.85 | 2.25 | 91.90 | S |
| Mankabad | 1 | 0-25 | 6.79 | 1.97 | 91.24 | L. S. |
| | | 25-50 | 9.82 | 13.30 | 76.88 | L. S. |
| | 2 | 0-25 | 14.27 | 15.55 | 70.18 | S. L. |
| | | 25-50 | 8.95 | 20.66 | 70.40 | S. L. |
| | 3 | 0-25 | 14.05 | 10.50 | 75.44 | S. L. |
| | | 25-50 | 11.71 | 14.67 | 73.62 | S. L. |
| Ellwan | 1 | 0-25 | 3.85 | 13.31 | 82.84 | L. S.. |
| | | 25-50 | 13.47 | 26.70 | 59.83 | S. L. |
| | 2 | 0-25 | 12.22 | 15.06 | 72.72 | S. L. |
| | | 25-50 | 8.98 | 21.23 | 69.79 | S. L. |
| | 3 | 0-25 | 7.97 | 18.90 | 73.12 | S. L. |
| | | 25-50 | 14.20 | 15.10 | 70.70 | S. L. |
| Bani Hussien | 1 | 0-25 | 17.94 | 11.08 | 70.98 | S. L. |
| | | 25-50 | 10.48 | 24.12 | 65.40 | S. L. |
| | 2 | 0-25 | 8.01 | 23.84 | 68.15 | S. L. |
| | | 25-50 | 9.47 | 15.72 | 74.82 | S. L |
| | 3 | 0-25 | 5.10 | 13.11 | 81.80 | L. S. |
| | | 25-50 | 18.00 | 5.54 | 76.46 | S. L. |
| Bani Ghalib | 1 | 0-25 | 6.34 | 1.97 | 91.69 | S |
| | | 25-50 | 5.47 | 11.40 | 83.12 | L. S. |
| | 2 | 0-25 | 7.30 | 13.77 | 78.93 | L. S. |
| | | 25-50 | 9.18 | 8.44 | 82.38 | L. S. |
| | 3 | 0-25 | 5.30 | 23.28 | 71.42 | S. L. |
| | | 25-50 | 13.29 | 17.46 | 69.25 | S L. |
| Gahdam | 1 | 0-25 | 9.00 | 4.03 | 86.97 | L. S. |
| | | 25-50 | 16.18 | 25.58 | 58.24 | S. L. |
| | 2 | 0-25 | 7.54 | 5.87 | 86.59 | L. S. |
| | | 25-50 | 4.70 | 7.42 | 87.88 | L. S.. |
| | 3 | 0-25 | 9.20 | 20.94 | 69.86 | S. L. |
| | | 25-50 | 15.61 | 26.44 | 57.95 | S. L. |

@ S, L.S, and S.L. mean Sandy, Loamy sand, and sandy loam, respectively.

**Table(2):** Some chemical properties of the soils of the studied soils

| Village | Depth | EC | CEC | pH | % | | meq / 100 g | | | P |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 1:1 | $Cmol^+/kg$ | 1:1 | O.M | N | $K^+$ | $Ca^{++}$ | $Mg^{++}$ | ppm |
| El Madabeg | 0-25 | 1.0 | 0.82 | 7.10 | 3.00 | 0.07 | 27.50 | 15.73 | 8.80 | 27.33 |
| | 25-50 | 1.8 | 0.46 | 7.60 | 1.40 | 0.08 | 28.02 | 21.73 | 5.73 | 22.67 |
| Mankabad | 0-25 | 1.5 | 2.76 | 7.81 | 2.10 | 0.08 | 25.40 | 36.00 | 4.00 | 25.91 |
| | 25-50 | 3.5 | 2.40 | 8.05 | 1.50 | 0.09 | 28.72 | 36.67 | 8.40 | 20.38 |
| Ellwan | 0-25 | 0.5 | 4.74 | 7.56 | 3.30 | 0.09 | 27.50 | 39.33 | 7.33 | 25.75 |
| | 25-50 | 1.4 | 4.15 | 7.76 | 1.80 | 0.08 | 27.15 | 39.33 | 15.33 | 20.46 |
| Bani Hussien | 0-25 | 1.3 | 4.65 | 7.59 | 2.40 | 0.09 | 27.67 | 39.33 | 10.67 | 24.87 |
| | 25-50 | 2.4 | 5.55 | 7.75 | 1.90 | 0.08 | 28.19 | 33.33 | 16.00 | 20.63 |
| Bani Ghalib | 0-25 | 2.5 | 3.79 | 7.96 | 2.90 | 0.08 | 26.27 | 21.33 | 19.07 | 25.36 |
| | 25-50 | 4.9 | 3.34 | 8.03 | 1.50 | 0.08 | 26.97 | 23.33 | 8.67 | 21.90 |
| Gahdam | 0-25 | 2.0 | 3.03 | 7.71 | 2.80 | 0.08 | 27.15 | 35.33 | 14.67 | 25.01 |
| | 25-50 | 3.3 | 2.80 | 7.86 | 2.30 | 0.08 | 27.93 | 34.13 | 8.53 | 22.35 |

**Heavy metals extractability:**

In the first part of this study, the extractability of heavy metals of different single-extraction procedures was investigated. Nine different single-extraction procedures of variable pH (ranged between 4.5 and 7.5), strength and concentration of the active ingredients, and the operations of the procedure (soil: extractant ratio, shaking and equilibrium time) were selected. The detailed operations were identical to the references in Table (3). In the same time, total concentrations of heavy metals in the soils of the studied area were determined after digestion using the three-acids mixture ($HClO_4$ + $H_2SO_4$ + $HNO_3$) according to Chapman and Pratt (1961).

**Table(3):** Heavy metals extraction procedures used in the study.

| Extractant | pH | Time | Ratio | References |
|---|---|---|---|---|
| 1) HCl + $H_2SO_4$ (0.05 N HCl + 0.025 N $H_2SO_4$) | 1.58 | 15 min | 1:4 | El-Koumey et al., (1997) Wear and Evans (1968) |
| 2) DTPA (0.1 M TEA+ 0.01 M CaCl2+ 0.005 M DTPA) | 7.3 | 2 h | 1:2 | El-Koumey et al.,(1997) |
| 3) AB-DTPA (Ammonium bicarbonate DTPA, 0.005 M DTPA+ 1 M $NH_4HCO_3$) | 7.6 | 15 min | 1:2 | Zhu and Alva (1993) |
| 4) EDTA (0.05 M EDTA) | 7 | 1h | 1:10 | Feng et al., (2005) |
| 5) EDTA-Ammonium acetate (0.007 M EDTA + 1N ammonium acetate) | 7 | 1 h | 1:10 | Trierweiler and Lindsay (1969) |
| 6) EDTA-AAAc (EDTA- Ammonium acetate with acetic acid), (0.5 M $NH_4OAc$ + 0.5 M HOAc + 0.02 M $Na_2$-EDTA) | 4.65 | 1 h | 1:10 | Abd El-Haleem et al., (2002) |
| 7) EDTA Ammonium carbonate (0.01 M EDTA + 1M $(NH_4)_2CO_3$) | 8.6 | 1 h | 1:10 | Trierweiler and Lindsay (1969) |
| 8) EDTA-Ammonium Citrate (0.01M EDTA+ 1 M $NH_4$- Citrate) | 4.8 | 1h | 1:10 | El-Koumey et al., (1997) |

**Pot experiments:**

Two pot experiments were conducted in the greenhouse at the Faculty of Agriculture, Assiut University at Assiut. Soil samples, 5 Kg each, were packed in plastic pots of 15X17.5 cm, irrigated and left for one weak for drying. Corn (Nifertete 3) seeds (10 seeds/ pot) were sown in each pot and thinned to 4 seedlings per pot after 10 days. Plants were watered to field capacity. When seedlings grow up to the first true- leaf (2 weeks), the plants were supplied with sufficient amounts of N P K at a rates of 0.16 gm N/ pot, 0.33 gm P/ pot and 0.28 gm K/ pot, in the forms of $NH_4NO_3$, $KH_2PO_4$ and $K_2SO_4$. Water loss was compensated for by daily addition of distilled water. Thirteen weeks (75 days) after planting, (aerial portion) were harvested, and soil samples were collected from each pot. To study the effects of the following crop on the extractability of polluted metals wheat plants were grown in the same pots. Ten seeds of wheat were sown in each pot (Giza 168). After 20 days from planting, wheat plants were thinned to 6 plants/pot. Pots were fertilized with N, P and K as mentioned in the first experiment. Plant samples were collected after 90 days and soil samples were collected from each pot. Corn and wheat were freshly weighed, washed with distilled water, oven dried at 70° C for 24 hours to constant weight, dry weighed, ground to pass through a 20 mesh sieve and stored for analysis. Plant dry materials were digested using $H_2SO_4$ + $H_2O_2$ acid mixture as described by Parkinson and Allen, (1975) and prepared for heavy metals and micronutrients determination. Soil samples were air-dried, crashed to pass from 2 mm sieve and stored for metal extractions later on.

**Results and Discussion**

**Part 1:**

**Total metal contents in soils:**

Table (4) and Figures (1-2) show the variation in total amounts of Fe, Mn, Zn, Cu, Cd, Ni, Pb and Cr in soils of the six locations (villages) of the study area irrigated by untreated swage wastewater for 40-45 years. T otal amount of Fe, Zn, Mn, Cu, Cd, Cd, Pb, Ni and Cr in the different locations of the studied area were decreased in surface and subsurface layers in the following order: Fe > Mn > Zn > Cu > Ni > Pb > Cr > Cd. The data point to a decreasing order between the beginnings of the irrigation canal (Madabegh) toward its end at (Gahdam) along a distance of about 20 km. This may be attributed to the probable variations in the settlement processes downward the stream of the irrigation cannel. Most of the solid materials carried out with the sewage wastes present in irrigation water settled at the beginning of the irrigation cannel and gradually decrease with distance toward the end of the irrigation cannel. Similar results were obtained by El Sokkary and Sharaf (1996) and Abdel Salam (2002).

**Table(4):** Total amounts of micronutrients and heavy metals (ppm) in soils of the studied area.

| Locations (Villages) | Site | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
|---|---|---|---|---|---|---|---|---|---|
| | | Surface layer | | | | | | | |
| Madabeg | 1 | 5904.9 | 1485.2 | 382.7 | 551.8 | 2.3 | 75.5 | 46.2 | 15.1 |
| | 2 | 4314.0 | 1649.6 | 501.5 | 213.1 | 1.5 | 39.3 | 40.6 | 6.6 |
| | 3 | 4415.6 | 1251.9 | 719.9 | 389.2 | 1.2 | 67.4 | 34.8 | 6.0 |
| | Mean | 4878.2 | 1462.2 | 534.7 | 384.7 | 1.7 | 60.7 | 40.5 | 9.2 |
| Mankabad | 1 | 1744.2 | 153.4 | 544.3 | 52.6 | 0.7 | 11.9 | 24.9 | 6.6 |
| | 2 | 1938.5 | 1044.4 | 589.0 | 346.7 | 1.2 | 39.7 | 29.9 | 11.5 |
| | 3 | 1479.3 | 629.6 | 699.9 | 382.0 | 1.8 | 24.3 | 30.8 | 7.3 |
| | Mean | 1720.7 | 609.1 | 611.1 | 260.4 | 1.2 | 25.3 | 28.5 | 8.5 |
| Ellwan | 1 | 1523.1 | 251.0 | 713.5 | 355.9 | 1.6 | 22.9 | 43.9 | 15.4 |
| | 2 | 2558.0 | 633.0 | 597.0 | 355.5 | 1.8 | 35.7 | 33.4 | 7.6 |
| | 3 | 1634.0 | 388.5 | 719.6 | 300.6 | 1.0 | 17.8 | 39.1 | 15.8 |
| | Mean | 1905.0 | 424.2 | 676.7 | 337.3 | 1.5 | 25.5 | 38.8 | 12.9 |
| Bani Hussien | 1 | 2008.0 | 120.9 | 598.7 | 207.9 | 0.8 | 17.1 | 47.8 | 13.9 |
| | 2 | 1463.1 | 144.5 | 743.7 | 212.0 | 0.7 | 22.2 | 34.6 | 8.2 |
| | 3 | 1899.9 | 92.7 | 543.3 | 164.9 | 0.5 | 20.0 | 33.2 | 13.9 |
| | Mean | 1790.3 | 119.4 | 628.5 | 194.9 | 0.7 | 19.8 | 38.5 | 12.0 |
| Bani Ghalib | 1 | 1184.4 | 223.2 | 488.9 | 134.1 | 0.8 | 16.1 | 32.9 | 18.0 |
| | 2 | 4596.7 | 133.6 | 457.5 | 134.6 | 0.8 | 12.0 | 34.5 | 9.2 |
| | 3 | 2841.8 | 156.9 | 456.7 | 136.5 | 1.3 | 10.6 | 32.3 | 18.1 |
| | Mean | 2874.3 | 171.2 | 467.7 | 135.1 | 1.0 | 12.9 | 33.2 | 15.1 |
| Gahdam | 1 | 1180.5 | 111.8 | 440.9 | 104.2 | 0.5 | 13.1 | 29.2 | 15.5 |
| | 2 | 1361.0 | 149.1 | 480.6 | 105.7 | 0.4 | 11.7 | 37.4 | 15.1 |
| | 3 | 1007.3 | 164.3 | 464.0 | 156.6 | 0.5 | 16.8 | 36.0 | 8.3 |
| | Mean | 1183.0 | 141.7 | 461.8 | 122.2 | 0.5 | 13.9 | 34.2 | 13.0 |

**Table 4: continue.**

| Vilage | Sites | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
|---|---|---|---|---|---|---|---|---|---|
| | | Subsurface Layer | | | | | | | |
| Madabeg | 1 | 3861.2 | 1085.7 | 462.7 | 396.7 | 1.2 | 40.5 | 45.1 | 41.5 |
| | 2 | 2019.2 | 574.7 | 394.1 | 150.8 | 0.8 | 13.7 | 34.5 | 18.4 |
| | 3 | 2701.1 | 458.5 | 436.5 | 171.1 | 0.9 | 28.7 | 39.5 | 20.2 |
| | Mean | 2860.5 | 706.3 | 431.1 | 239.5 | 0.9 | 27.6 | 39.7 | 26.7 |
| Mankabad | 1 | 1318.6 | 118.6 | 457.7 | 140.5 | 1.1 | 9.0 | 30.1 | 17.8 |
| | 2 | 1818.6 | 218.4 | 589.1 | 293.7 | 0.9 | 15.7 | 32.4 | 19.6 |
| | 3 | 1437.9 | 195.3 | 616.5 | 249.5 | 1.4 | 12.4 | 30.6 | 18.1 |
| | Mean | 1525.1 | 177.4 | 554.4 | 227.9 | 1.1 | 12.3 | 31.0 | 18.5 |
| Ellwan | 1 | 1716.3 | 139.5 | 691.0 | 238.0 | 1.1 | 13.1 | 34.5 | 13.4 |
| | 2 | 2286.6 | 251.6 | 582.4 | 277.6 | 1.2 | 16.2 | 47.3 | 16.7 |
| | 3 | 1549.0 | 177.3 | 554.7 | 273.3 | 0.9 | 9.6 | 39.1 | 24.5 |
| | Mean | 1850.6 | 189.5 | 609.4 | 263.0 | 1.1 | 13.0 | 40.3 | 18.2 |
| Bani Hussien | 1 | 1902.0 | 173.4 | 608.3 | 205.4 | 1.0 | 22.7 | 44.4 | 16.6 |
| | 2 | 1375.2 | 86.6 | 469.1 | 184.9 | 0.8 | 17.8 | 41.8 | 40.1 |
| | 3 | 2165.7 | 105.7 | 600.6 | 140.0 | 0.7 | 15.2 | 38.5 | 20.0 |
| | Mean | 1814.3 | 121.9 | 559.3 | 176.8 | 0.8 | 18.6 | 41.6 | 25.6 |
| Bani Ghalib | 1 | 1141.1 | 148.7 | 591.1 | 115.7 | 0.7 | 13.4 | 42.3 | 13.5 |
| | 2 | 1139.9 | 102.8 | 480.9 | 95.2 | 0.6 | 9.6 | 34.5 | 29.8 |
| | 3 | 1252.1 | 137.7 | 428.4 | 115.3 | 0.9 | 17.0 | 24.8 | 14.8 |
| | Mean | 1177.7 | 129.7 | 500.1 | 108.7 | 0.7 | 13.3 | 33.9 | 19.4 |
| Gahdam | 1 | 1407.1 | 113.9 | 471.4 | 97.1 | 0.7 | 16.1 | 40.1 | 37.4 |
| | 2 | 1624.4 | 112.9 | 461.1 | 86.6 | 0.5 | 13.2 | 29.2 | 15.5 |
| | 3 | 1150.5 | 143.6 | 514.9 | 156.6 | 0.7 | 14.6 | 41.0 | 13.6 |
| | Mean | 1394.0 | 123.4 | 482.5 | 113.4 | 0.6 | 14.6 | 36.8 | 22.2 |

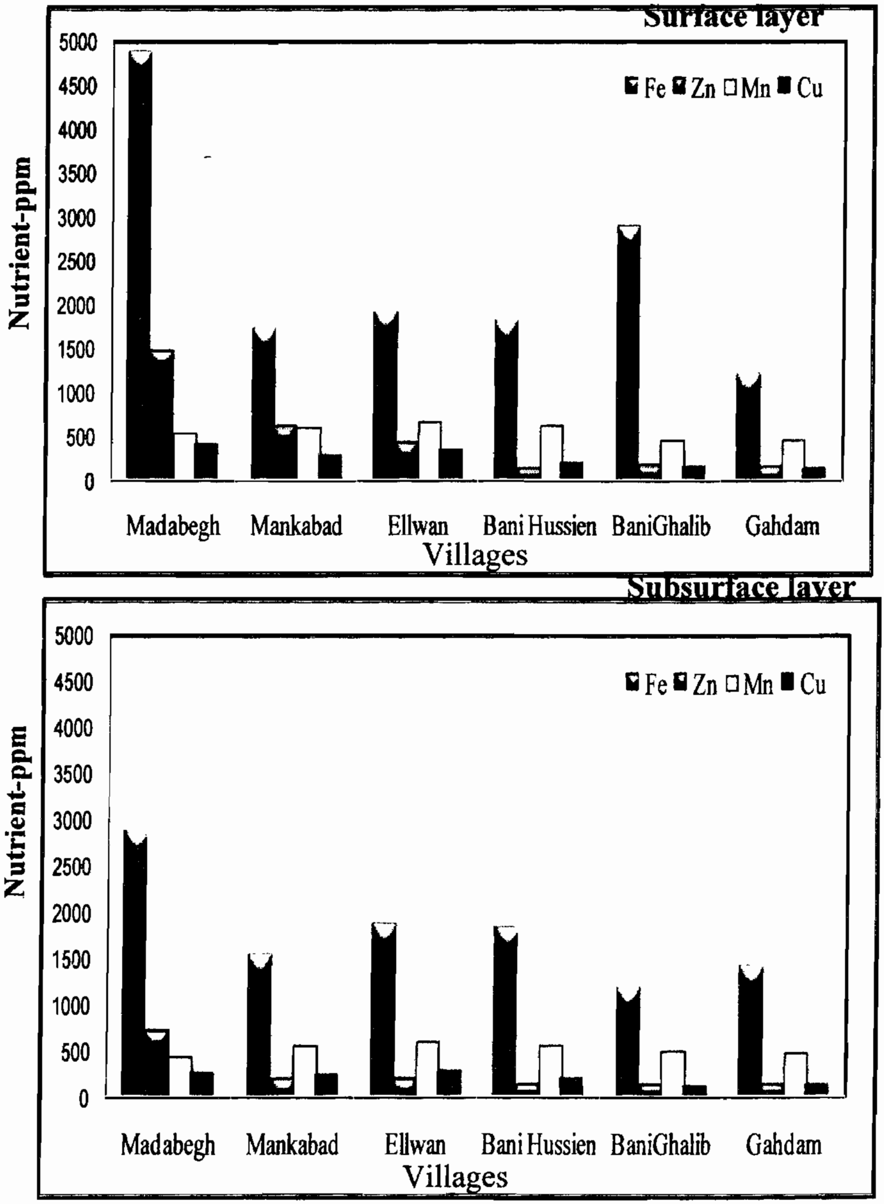



**Fig.(1):** Variation in total contents of micronutrients - ppm (Fe, Zn, Mn, and Cu) in the soils of the studied six locations irrigated with sewage waste water.

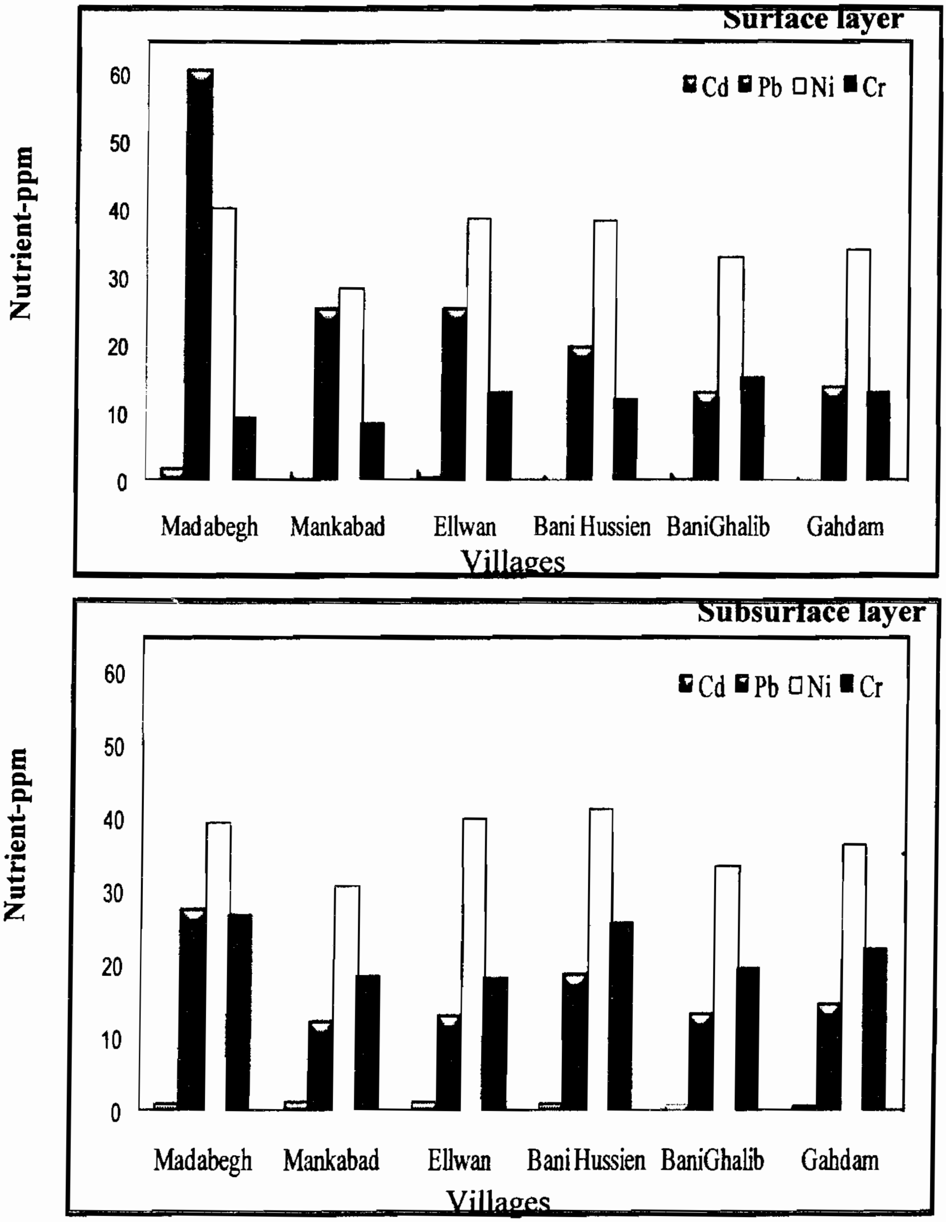



**Fig.(2):** Variation in total contents of micronutrients - ppm (Cd, Pb, Ni, and Cr) in the different layers of the studied six locations irrigated with sewage waste water.

**Interrelationships among total element contents` and soil properties:**

The relationships between the total concentrations of metals in different locations of the polluted area and soil properties are summarized in Table (5). Generally, significant negative correlation coefficients were obtained between total of each Fe, Zn, Cu, Cd, Pb and Cr in soil and soil pH. The correlation coefficients between soil pH and total amount of Fe, Zn, Cu, Cd, Pb and Cr were -0.681**, -0.675**, -0.525*, -0.560*, -0.644** and -0.632**, respectively. The CEC was significantly positive correlated with total amounts of Fe and Mn (The r values were 0.626** and 0.661**, respectively), and negatively correlated with total Zn (r= -0.514**).

**Table(5):**Correlation coefficients between total amounts of micronutrients and heavy metals, and soil properties of the soils of the studied area.

| Total | Depth | EC | pH | Clay | Silt | Sand | CEC | O.M. |
|---|---|---|---|---|---|---|---|---|
| Fe | 0-25 | -0.101 | -0.467 | -0.312 | -0.242 | 0.341 | -0.626** | 0.017 |
| | 25-50 | -0.412 | -0.681** | -0.336 | -0.462 | 0.467 | -0.339 | -0.207 |
| Zn | 0-25 | -0.243 | -0.675** | -0.068 | -0.339 | 0.313 | -0.447 | 0.038 |
| | 25-50 | -0.346 | -0.601** | -0.502* | -0.521* | 0.573* | -0.514* | -0.372 |
| Mn | 0-25 | -0.440 | 0.146 | 0.320 | 0.303 | -0.396 | 0.347 | 0.138 |
| | 25-50 | 0.035 | 0.247 | 0.389 | 0.493* | -0.510* | 0.661** | 0.187 |
| Cu | 0-25 | -0.378 | -0.525* | 0.060 | 0.014 | -0.039 | -0.005 | -0.053 |
| | 25-50 | -0.430 | -0.554* | -0.087 | 0.049 | -0.003 | 0.039 | -0.320 |
| Cd | 0-25 | -0.343 | -0.560* | -0.073 | -0.065 | 0.087 | -0.150 | -0.032 |
| | 25-50 | -0.234 | -0.174 | 0.052 | 0.170 | -0.145 | -0.002 | -0.202 |
| Pb | 0-25 | -0.212 | -0.644** | -0.107 | -0.278 | 0.280 | -0.346 | -0.102 |
| | 25-50 | -0.149 | -0.661** | -0.294 | -0.302 | 0.333 | -0.215 | -0.240 |
| Ni | 0-25 | -0.334 | -0.445 | -0.073 | 0.017 | 0.019 | 0.035 | 0.267 |
| | 25-50 | -0.232 | -0.365 | -0.024 | 0.093 | -0.059 | 0.426 | 0.334 |
| Cr | 0-25 | 0.058 | -0.019 | -0.277 | 0.034 | 0.097 | 0.327 | -0.048 |
| | 25-50 | -0.475* | -0.632** | -0.008 | -0.197 | 0.148 | -0.199 | 0.017 |

*,** = $p < 0.05$ and 0.01, respectively.

**Extractability of different extraction procedures:**

The objective of this part is to explore the extractability of some extraction procedures that widely used in determining the amount of mobile forms of heavy metals in polluted soils irrigated with untreated sewage wastewaters.

Comparing the extraction procedures for their ability to extract the potential of each heavy metal to enter the mobile pools and becoming environmentally hazard is undertaken.

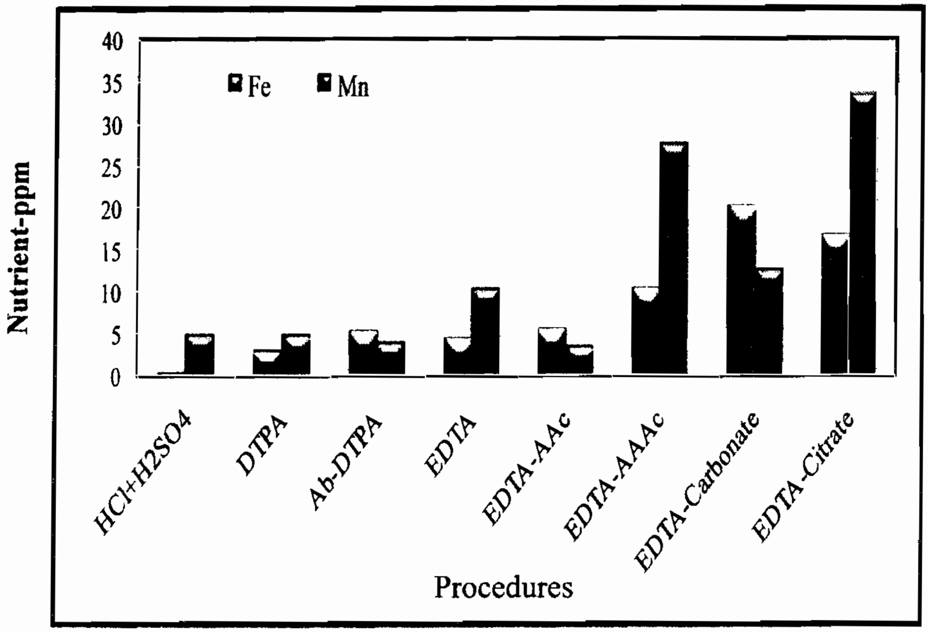
The amounts of extractable Fe, Mn, Zn, Cu, Cd, Ni, Pb and Cr in the 18 soils via the tested extractants varied depending on metals and extractants used. Among the extractants, EDTA-AAAc extracted the largest proportion of Fe, Zn, Mn, Cu, Cd and Pb followed by EDTA-Ammonium Citrate with Fe, Zn, Mn, Cu and Pb, while HCl + $H_2SO_4$ were the lowest for the entire eight studied elements. Regardless of soils and extractants, relative extractability was higher for Ni and Cr as compared to other two heavy metals (Table 6 and Figure 3).

Table (7) showed the correlation coefficients for soil pH and extracted Zn, and Pb. These relations showed that extracted Zn, with HCl+$H_2SO_4$, EDTA, EDTA-AAAc and EDTA-Ammonium Carbonate extractants correlated highly significantly and negatively with soil pH. At the same time the extracted Zn by HCl+$H_2SO_4$, EDTA and EDTA- AAAc were correlated significantly and negatively with soil CEC. However, extracted Pb with AB-DTPA and EDTA-AAc correlated significantly and negatively with soil CEC; the r values were -0.52* and -0.489*, receptively. The relations presented in Table (7) showed that the concentrations of DTPA, and HCl + $H_2S0_4$ Mn were negatively and significantly correlated with soil pH. Also, Mn extracted with AB-DTPA and EDTA were positively and significantly with soil CEC.

In the present study, extracted Cu with DTPA, EDTA, EDTA-AAc, EDTA-AAAc, EDTA-Ammonium Carbonate and EDTA-Ammonium Citrate at each site of the studied area were strongly related to soil pH (r = -0.511*, -0.479*, -0.492*, -0.543*, -0.654** and -0.556*, respectively). It was found that soil pH of studied area was highly significant and negatively correlated with EDTA-AAAc and HCl+$H_2SO_4$ extracted Cd and Ni, Table (7). Cation exchangeable capacity of the tested soils was correlated significantly and positively with EDTA-AAc – Cd, while CEC was correlated significantly and negatively with EDTA – Cd. Correlation coefficients between CEC of the polluted soils and extracted Ni with EDTA, EDTA-AAAc, EDTA-Ammonium Citrate and HCl+$H_2SO_4$ are presented in Table (7). The data of these extractants showed a good and significant correlation with soil CEC.

**Table(6):** Minimum, maximum, and averages of extracted amounts of micronutrients and heavy metals (ppm) using different extraction procedures.

| Extractant | | Depth | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
|---|---|---|---|---|---|---|---|---|---|---|
| $HCl + H_2SO_4$ | Min | 0-25 | 0.00 | 0.00 | 0.00 | 0.20 | 0.00 | 0.00 | 0.00 | 0.00 |
| | | 25-50 | 0.00 | 0.04 | 0.04 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 |
| | Max | 0-25 | 2.44 | 0.80 | 9.86 | 0.96 | 0.26 | 1.23 | 1.41 | 0.40 |
| | | 25-50 | 0.52 | 1.96 | 8.60 | 0.68 | 0.07 | 0.97 | 0.77 | 0.20 |
| | Aver. | 0-25 | 0.24 | 0.24 | 4.72 | 0.38 | 0.04 | 0.21 | 0.22 | 0.02 |
| | | 25-50 | 0.09 | 0.53 | 3.70 | 0.31 | 0.03 | 0.18 | 0.12 | 0.02 |
| DTPA | Min | 0-25 | 0.45 | 0.97 | 2.16 | 0.15 | 0.00 | 0.00 | 0.33 | 0.00 |
| | | 25-50 | 0.42 | 0.90 | 1.16 | 0.12 | 0.00 | 0.00 | 0.49 | 0.00 |
| | Max | 0-25 | 5.95 | 9.92 | 7.58 | 3.59 | 1.13 | 3.00 | 0.74 | 0.71 |
| | | 25-50 | 2.78 | 6.39 | 6.40 | 2.25 | 0.11 | 1.96 | 0.91 | 0.69 |
| | Aver. | 0-25 | 2.87 | 2.78 | 4.60 | 1.62 | 0.36 | 0.93 | 0.59 | 0.26 |
| | | 25-50 | 1.03 | 1.94 | 2.98 | 1.00 | 0.03 | 0.58 | 0.65 | 0.30 |
| DTPA-Amm.Bicarbonate | Min | 0-25 | 1.75 | 0.65 | 0.94 | 0.29 | 0.00 | 0.19 | 0.00 | 0.00 |
| | | 25-50 | 1.92 | 0.61 | 0.00 | 0.47 | 0.00 | 0.19 | 0.00 | 0.03 |
| | Max | 0-25 | 19.11 | 3.35 | 7.59 | 3.59 | 0.03 | 5.70 | 0.21 | 0.20 |
| | | 25-50 | 9.38 | 8.58 | 8.48 | 4.02 | 0.04 | 3.13 | 0.22 | 0.17 |
| | Aver. | 0-25 | 5.06 | 1.34 | 3.83 | 2.12 | 0.01 | 1.67 | 0.09 | 0.09 |
| | | 25-50 | 3.92 | 1.94 | 4.48 | 2.46 | 0.01 | 0.89 | 0.09 | 0.11 |
| EDTA | Min | 0-25 | 0.00 | 1.98 | 1.43 | 1.50 | 0.06 | 2.86 | 0.45 | 0.00 |
| | | 25-50 | 0.00 | 0.00 | 1.39 | 1.94 | 0.01 | 2.02 | 0.90 | 0.00 |
| | Max | 0-25 | 5.86 | 7.97 | 25.00 | 10.05 | 0.97 | 9.92 | 3.31 | 0.44 |
| | | 25-50 | 6.42 | 9.92 | 17.57 | 13.56 | 1.09 | 17.66 | 2.80 | 1.69 |
| | Aver. | 0-25 | 4.31 | 4.20 | 10.29 | 6.95 | 0.54 | 5.85 | 1.99 | 0.02 |
| | | 25-50 | 3.25 | 3.90 | 8.96 | 6.63 | 0.38 | 7.34 | 2.13 | 0.09 |
| EDTA- AAc | Min | 0-25 | 1.68 | 1.50 | 0.00 | 0.08 | 0.21 | 0.37 | 0.39 | 0.00 |
| | | 25-50 | 4.41 | 0.87 | 0.69 | 1.00 | 0.00 | 0.02 | 0.16 | 0.00 |
| | Max | 0-25 | 9.21 | 14.24 | 7.70 | 24.67 | 0.95 | 29.19 | 1.58 | 1.85 |
| | | 25-50 | 20.90 | 12.71 | 6.50 | 17.52 | 0.80 | 8.19 | 2.24 | 3.32 |
| | Aver. | 0-25 | 5.34 | 6.34 | 3.26 | 8.55 | 0.55 | 6.81 | 0.91 | 0.31 |
| | | 25-50 | 8.91 | 4.68 | 3.04 | 6.55 | 0.41 | 3.75 | 0.97 | 0.69 |
| EDTA- AAAc | Min | 0-25 | 2.69 | 1.25 | 11.25 | 0.80 | 0.00 | 0.46 | 0.17 | 0.30 |
| | | 25-50 | 1.28 | 1.22 | 11.50 | 0.49 | 0.00 | 0.40 | 0.22 | 0.42 |
| | Max | 0-25 | 25.94 | 101.15 | 49.60 | 15.47 | 0.32 | 27.38 | 1.35 | 0.78 |
| | | 25-50 | 14.50 | 33.80 | 42.10 | 8.59 | 0.15 | 8.94 | 1.12 | 2.98 |
| | Aver. | 0-25 | 10.16 | 20.37 | 27.64 | 7.18 | 0.06 | 7.47 | 0.44 | 0.41 |
| | | 25-50 | 8.38 | 5.71 | 28.27 | 4.75 | 0.04 | 3.71 | 0.44 | 0.98 |
| EDTA-Amm. Carbonate | Min | 0-25 | 2.93 | 0.80 | 6.57 | 1.21 | 0.00 | 0.00 | 0.00 | 0.00 |
| | | 25-50 | 2.99 | 0.43 | 7.24 | 0.65 | 0.00 | 0.00 | 0.00 | 0.00 |
| | Max | 0-25 | 85.24 | 19.49 | 24.88 | 11.96 | 0.90 | 8.90 | 1.13 | 0.94 |
| | | 25-50 | 34.41 | 5.41 | 14.20 | 9.49 | 0.13 | 3.14 | 0.96 | 1.66 |
| | Aver. | 0-25 | 20.03 | 5.50 | 12.54 | 4.77 | 0.11 | 2.18 | 0.25 | 0.39 |
| | | 25-50 | 8.61 | 2.02 | 10.45 | 3.55 | 0.03 | 1.53 | 0.31 | 0.42 |
| EDTA-Amm. Citrate | Min | 0-25 | 5.03 | 1.93 | 13.35 | 1.03 | 0.03 | 1.07 | 0.12 | 0.35 |
| | | 25-50 | 6.97 | 1.18 | 13.45 | 0.22 | 0.03 | 1.04 | 0.15 | 0.35 |
| | Max | 0-25 | 42.94 | 56.80 | 47.15 | 18.86 | 0.59 | 23.82 | 0.38 | 0.42 |
| | | 25-50 | 23.07 | 55.60 | 41.45 | 9.98 | 0.15 | 15.73 | 0.60 | 2.00 |
| | Aver. | 0-25 | 16.58 | 14.83 | 33.40 | 5.96 | 0.31 | 7.50 | 0.20 | 0.38 |
| | | 25-50 | 13.03 | 6.88 | 31.07 | 4.82 | 0.07 | 4.11 | 0.26 | 0.87 |



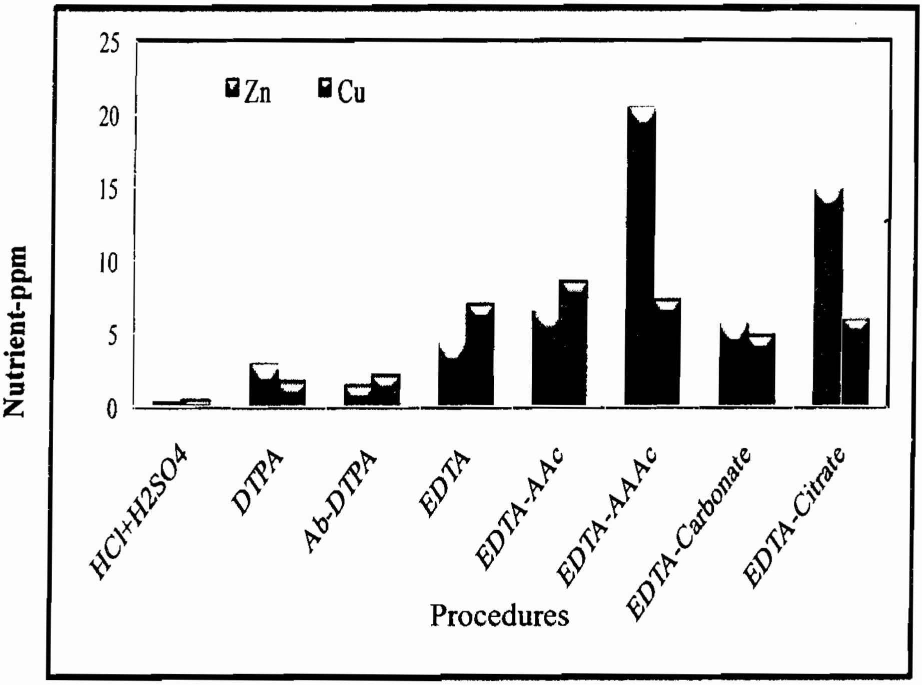
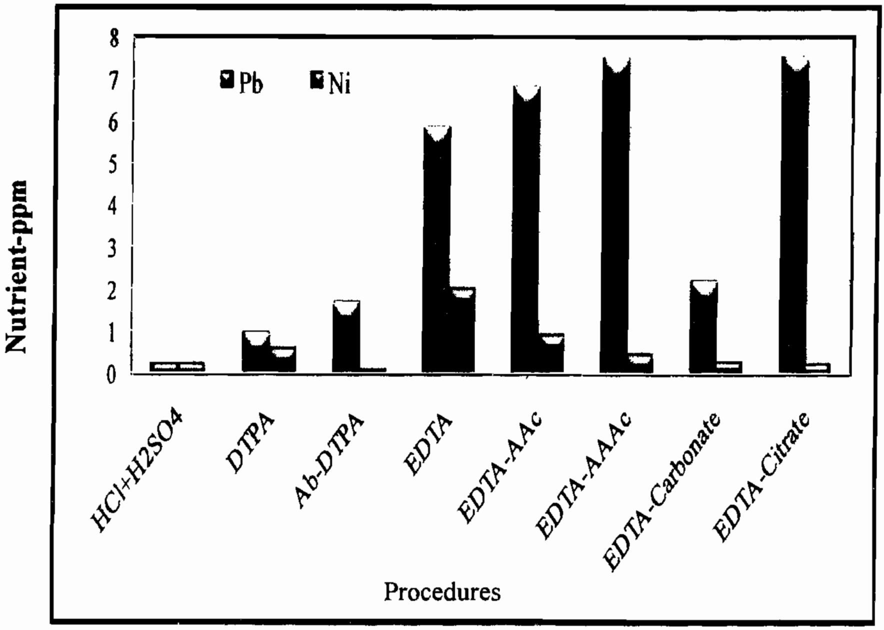




**Fig. (3):** The concentrations of extractable heavy metals (ppm) in 18 soils by the eight tested extractions.



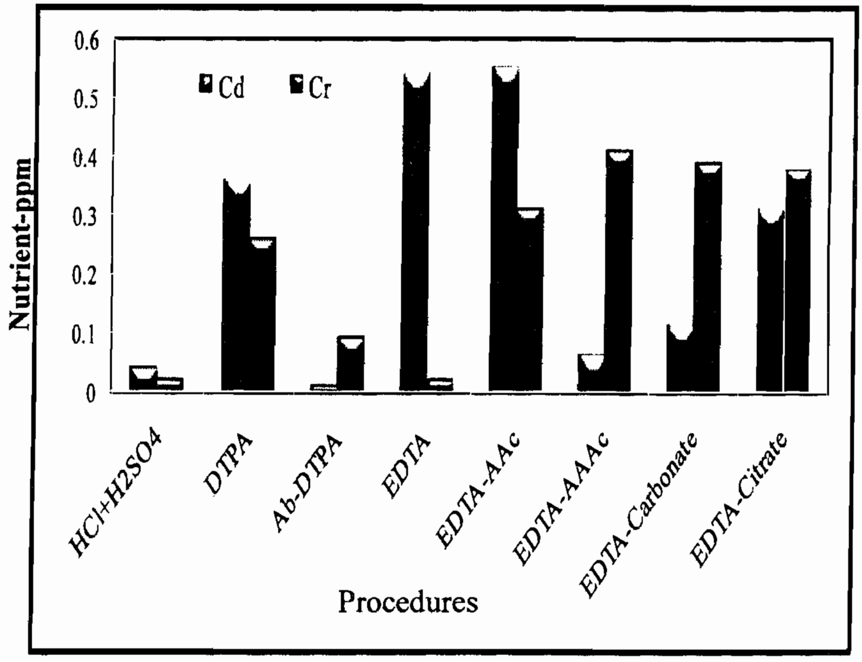




**Fig. (3):** Continue

**Table(7):** Correlation Coefficients between extracted heavy metals in the studied area and soil (pH and CEC) in the surface soils layer:

| pH | Fe | Zn | Mn | Cu | Cd | Pb | Ni | |
|---|---|---|---|---|---|---|---|---|
| Soil pH | | | | | | | | |
| HCl+$H_2SO_4$ | 0.359 | 0.418 | -0.631$^{**}$ | -0.556$^{*}$ | -0.628$^{**}$ | 0.120 | -0.632$^{**}$ | 0.014 |
| DTPA | -0.563$^{*}$ | -0.752$^{**}$ | -0.621$^{**}$ | -0.511$^{*}$ | -0.464 | -0.699$^{**}$ | -0.030 | -0.240 |
| Ab-DTPA | -0.538$^{*}$ | -0.719$^{**}$ | 0.340 | -0.284 | -0.585$^{*}$ | -0.580$^{*}$ | -0.379 | 0.247 |
| EDTA | -0.496$^{*}$ | -0.187 | -0.108 | -0.041 | -0.053 | 0.307 | -0.015 | -0.007 |
| EDTA-Aac | 0.443 | -0.276 | -0.025 | -0.492$^{*}$ | -0.090 | -0.515$^{*}$ | 0.309 | 0.234 |
| EDTA-AAAc | -0.718$^{**}$ | -0.708$^{**}$ | 0.103 | -0.479$^{*}$ | -0.535$^{*}$ | -0.653$^{**}$ | -0.659$^{**}$ | -0.036 |
| EDTA-Amm. Carbonate | -0.124 | -0.664$^{**}$ | -0.173 | -0.543$^{*}$ | -0.082 | -0.710$^{**}$ | 0.048 | -0.359 |
| EDTA-Amm. Citrate | -0.742$^{**}$ | -0.600$^{**}$ | 0.678$^{**}$ | -0.654$^{**}$ | -0.295 | -0.536$^{*}$ | -0.068 | -0.330 |
| Soil CEC | | | | | | | | |
| HCl+$H_2SO_4$ | -0.218 | 0.287 | -0.008 | -0.120 | -0.353 | 0.191 | -0.564$^{*}$ | 0.283 |
| DTPA | 0.117 | -0.446 | -0.356 | -0.342 | -0.048 | -0.398 | 0.247 | 0.238 |
| Ab-DTPA | -0.452 | -0.504$^{*}$ | 0.641$^{**}$ | 0.028 | -0.443 | -0.402 | -0.381 | -0.317 |
| EDTA | -0.044 | 0.049 | 0.599$^{**}$ | 0.307 | -0.101 | 0.380 | 0.638$^{**}$ | 0.263 |
| EDTA-Aac | -0.006 | -0.013 | 0.408 | -0.075 | -0.242 | -0.381 | 0.236 | 0.212 |
| EDTA-AAAc | -0.316 | -0.471$^{*}$ | -0.102 | -0.080 | 0.027 | -0.385 | -0.560$^{*}$ | -0.241 |
| EDTA-Amm. Carbonate | -0.659$^{**}$ | -0.518$^{*}$ | -0.004 | -0.221 | -0.158 | -0.327 | -0.265 | 0.133 |
| EDTA-Amm. Citrate | -0.268 | -0.420 | 0.316 | -0.132 | -0.147 | -0.429 | -0.552$^{*}$ | -0.416 |

*, **= significant at: $p < 0.05$, $p < 0.01$ and $p < 0.001$ probability levels. All other $p > 0.05$.

Generally, and depending on the previous results, it could be stated that the soil pH and CEC of the soils are the main soil factors which affected on most of the heavy metal contents of the soil which extracted with various extractants.

**Part 2**

This part of the research work devoted to study the suitability of extraction procedures for predicting the levels of available micronutrients and heavy metals in polluted soils.

In all soil after planting the extractable Zn, Cu and Pb from soil samples were decreased, but the extractable amounts of other metals (Fe, Mn, Cd, Ni and Cr) were remarkably increased after corn harvesting. However, after wheat harvesting, in all soil locations, the amount of all extractable metals, but not Zn, Cd, Ni and Cr, were decreased comparing either to the initial amounts before planting or to the amounts after corn harvesting. The mean values of extractable Fe, Zn, Mn, Cu, Cd, and Pb were decreased. It should be noticed that the changes in extractable amounts of metals after corn harvesting are little higher than that after wheat harvesting. These changes reflect the effect of plant type on the status of micronutrients and heavy metals in root zone. The changes in the amounts of extractable metals could be attributed either to the variations in crop requirements of micronutrients and heavy metals, or to the variations in the ability of corn and wheat root system and root exudates to release the metals from the exchange sites, or dissolve the metals from their bearing compounds. Root system and root exudates both exert great influences on solubility and release of nutrients into root zone, thus increase the amounts of extractable metals. Even thought both corn and wheat crops are C4 plants, their root systems are markedly differed, and exert different effects on the status of metals in the surrounding root zone. Therefore, good care should be taken, when deciding which element will be determinate. Soil type and the type of crop plants should be considered when deciding which extraction procedure should use.

Data summarized in Table 8 show the amounts of extractable micronutrients and heavy metals extracted by eight different extraction procedures from polluted soils before planting, and at the end of growth seasons of corn and wheat. The ability of the extraction procedures were varied with the type of extractant, the type of polluted metals, and the type of previous planted crop. For instance, with respecting to the extractable amount of Fe, before planting EDTA-AAAc gave the highest amount, while after corn and wheat EDTA gave the highest amount. In general, the amounts of metals extracted with HCL+$H_2SO_4$ were the lowest, either before planting or after growing of corn and wheat crop. The EDTA extraction procedures, on the other hand, were the most efficient procedures for extracting the micronutrients and heavy metals from the soils irrigated with sewage wastewater.

**Table(8):** The effects of extraction procedures on the amount of extracted metals (ppm) during the growth seasons.

| Extraction Procedures | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
|---|---|---|---|---|---|---|---|---|
| Before Planting | | | | | | | | |
| HCl+H2SO4 | 0.24 | 0.24 | 4.72 | 0.38 | 0.04 | 0.21 | 0.22 | 0.02 |
| DTPA | 2.87 | 2.78 | 4.60 | 1.62 | 0.36 | 0.93 | 0.59 | 0.26 |
| Ab-DTPA | 5.06 | 1.34 | 3.83 | 2.12 | 0.01 | 1.67 | 0.09 | 0.09 |
| EDTA | 4.31 | 4.20 | 10.29 | 6.95 | 0.54 | 5.85 | 1.99 | 0.02 |
| EDTA-Aac | 5.34 | 6.34 | 3.26 | 8.55 | 0.55 | 6.81 | 0.91 | 0.31 |
| EDTA-AAAc | 10.16 | 20.37 | 27.64 | 7.18 | 0.06 | 7.47 | 0.44 | 0.41 |
| EDTA-Carbonate | 20.03 | 5.50 | 12.54 | 4.77 | 0.11 | 2.18 | 0.25 | 0.39 |
| EDTA-Amm.Citrate | 16.58 | 14.83 | 33.40 | 5.96 | 0.31 | 7.50 | 0.20 | 0.38 |
| Mean | 8.07 | 6.95 | 12.53 | 4.69 | 0.25 | 4.08 | 0.59 | 0.24 |
| $LSD_{0.05}$ | 4.66** | 4.23** | 3.61** | 1.86** | 0.16** | 1.99** | 0.16* | 0.17** |
| After Corn | | | | | | | | |
| HCL+H2SO4 | 0.11 | 0.26 | 7.38 | 0.36 | 2.25 | 1.46 | 0.18 | 0.00 |
| DTPA | 16.31 | 5.40 | 15.13 | 11.25 | 4.71 | 5.02 | 2.32 | 0.00 |
| Ab-DTPA | 32.21 | 2.15 | 6.81 | 4.65 | 1.53 | 2.73 | 1.20 | 0.35 |
| EDTA | 54.63 | 4.40 | 13.34 | 2.83 | 4.11 | 4.29 | 1.85 | 1.39 |
| EDTA-AAc | 22.13 | 8.92 | 4.73 | 1.11 | 7.41 | 2.19 | 1.14 | 4.36 |
| EDTA-AAAc | 13.78 | 3.00 | 13.66 | 1.64 | 1.66 | 3.25 | 2.24 | 0.00 |
| EDTA-Carbobate | 2.91 | 3.82 | 19.17 | 1.17 | 1.23 | 1.82 | 0.41 | 0.12 |
| EDTA-Amm.Citrate | 8.04 | 3.22 | 39.16 | 1.20 | 1.36 | 1.53 | 3.06 | 1.86 |
| Mean | 18.77 | 3.90 | 14.92 | 3.03 | 3.07 | 2.79 | 1.55 | 1.01 |
| $LSD_{0.05}$ | 6.30** | 1.12** | 2.66** | 0.79** | 0.52** | 0.69** | 1.25** | 2.16** |
| After Wheat | | | | | | | | |
| HCL+H2SO4 | 1.12 | 0.21 | 0.96 | 0.08 | 0.38 | 0.07 | 0.38 | 0.24 |
| DTPA | 4.59 | 2.04 | 4.87 | 3.42 | 2.79 | 1.94 | 0.20 | 0.58 |
| Ab-DTPA | 9.11 | 2.64 | 8.18 | 4.04 | 3.91 | 6.64 | 0.16 | 0.48 |
| EDTA | 19.91 | 5.47 | 18.55 | 2.85 | 4.97 | 5.00 | 4.16 | 0.74 |
| EDTA-AAc | 8.45 | 12.88 | 20.30 | 0.63 | 1.03 | 0.95 | 0.54 | 0.27 |
| EDTA-AAAc | 9.06 | 4.38 | 10.42 | 1.37 | 1.57 | 2.68 | 1.80 | 1.32 |
| EDTA-Carbobate | 2.39 | 14.52 | 20.02 | 1.18 | 1.33 | 1.79 | 1.45 | 0.79 |
| EDTA-Amm.Citrate | 5.72 | 5.02 | 10.40 | 4.23 | 9.55 | 1.92 | 0.50 | 0.00 |
| Mean | 7.54 | 5.89 | 11.71 | 2.22 | 3.19 | 2.62 | 1.15 | 0.55 |
| $LSD_{0.05}$ | 2.49** | 4.93** | 2.64** | 0.56** | 0.97** | 0.60** | 1.09** | 0.46** |

* and ** significant at probability level <0.05 and 0.01, respectively n.s not significant

Extraction procedures were also greatly varied in predicting the changes in the status of metals in root zone associated with changing the type of crop plants. The EDTA- procedures show the highest variations in the extractable amounts of most metals, especially Fe, Mn, Cd and Ni. For instance, the amounts of extracted Fe by EDTA were changed from 4.31 ppm before planting to 54.63 and 19.91 ppm after corn and wheat harvesting, respectively. However, in the case of Zn the extracted amounts were changing from 4.20 ppm before planting to only 4.40 and 5.47 ppm after corn and wheat, respectively. Using acetate ions or acetic acids with EDTA improve its ability to predict the variations in the amounts of available metals.

Nyamangara, and Mzezewa (1999) reported that EDTA extraction procedure is used to extract the bioavailable fractions of metal in sewage sludge contaminated area. The release of metal ions electrostatically weakly bound to exchange surfaces is promoted by ion exchange with cations such as ammonium ($NH_4^+$) (Ure, 1996). Moreover, including the acetate ions and/or acetic acids and ammonium ions with EDTA enhances its ability to extract labile (exchangeable and soluble) form of metals, and improves its ability to predict the variations in the amounts of available metals. This is quite obvious in the case of Fe (Table 8) .EDTA-Ammonium forms strong complexes with numerous heavy metals and release their exchangeable forms (Kabata-Pendias and Pendias, 1984), and organically complexes "pools" (Podlesakova *et al.*, 2001).

The presence of acetate ions with EDTA also promotes the ability of EDTA to extract metals from polluted soils. The data presented in Table 8 show that in all studied growth seasons the highest values of all extractable metals, but Cu, Cd, and Pb, were obtained by EDTA-AAAc (pH 4.65), while the lowest were found with $HCL+H_2SO_4$. The superiority of EDTA-AAAc as compared to other extractants may be duo to its low pH (4.65) and the chelating effects of acetate anions, each of the two characters may enhanced its metals extractability (Hegazy, 1980; Hegazy *et al.*, 1991; and Abd El-Haleem *et al.*, 2002).

The DTPA extraction procedures, on the other hand, extract low amounts of heavy metals and micronutrients compared to EDTA extraction procedures (Table 8). For instance, before planting, the amounts of Fe, Zn, Mn, and Cu extracted by DTPA were 2.87, 2.78, 4.6, and 1.62 ppm, respectively, compared to 4.31, 4.20, 10.20, and 6.95 ppm extracted by EDTA, respectively. The same thing is quite true for heavy metals, especially Cd, Cr and Pb. This may be due to what have been mentioned by Bermond *et al.* (1998) that the EDTA procedures are non-specific extraction, and they can extract labile and non-labile fractions of micronutrients and heavy metals. Nomeda *et al.* (2004) reported that extractant's pH has a considerable influence on extractable heavy metals.

Inspecting our results emphasized that choosing the extraction procedures for studying the status of heavy metals pollutants in soils irrigated for a long time with sewage wastewater requires taking into account the aim of the investigation, the soil type, the source of the contaminants, as well as the agricultural practices and the nature of the previous crops. These findings are in agreement with those reported by Brazauskine *et al.* (2004), and Gupta and Sinha (2006).

**Metals concentrations in corn and wheat plants grown on soils irrigated with sewage wastewater for long time:**

Concentrations of heavy metals and micronutrients in corn and wheat plants grown on soils irrigated with sewage wastewater for long time are considerably varied with the variations in the amounts of available metals among the studied soils in different locations. The concentrations of Fe, Pb, Ni, and Cr in corn plants were decreased with increasing the distance from the source-point of pollution (Madabegh). For instance, the concentrations of Ni in corn plants grown on soils of (Madabegh and Mankabad) are noticeably higher than those of Bani-Ghalib and Gahdam. Also corn plants grown on soils of Bani-Ghalib, which is far away from the source point of pollution, contained the lowest amount of Cr ( 3.40 ppm) as compared with those grown on the soils of Madabegh, Mankabad, Ellwan, Bani Hussein, and Gahdam (9.42, 23.71, 20.64, 16.78, and 10.23 ppm, respectively). The variations in the concentrations of the other metals among locations did not show obvious trends (Table 9). The concentrations of all studied heavy metals in corn plants, but not Pb in some locations, are higher than the (accepted ranges). The concentrations of Pb in corn plants grown on the soils of Ellwan, Bani Hussein, and Gahdam were lower than the normal level of 4 mg $kg^{-1}$ dry matter as reported by Kabata Pendias and Pendias (1992).

To some degree, the variations in metals concentrations in corn plants are in agreement with the variations in the available amounts of metals in soils. The disagreements, that might be noticed, between the concentrations of heavy metals in corn plants and the amounts of extracted metals from soils of the studied location could be due to the variability in the potentials of the different extraction procedures used in this study.

**Table(9):** Dry matter accumulation (gm/pot$^{-1}$) and concentrations of micronutrients and heavy metals in corn plants grown on polluted soils of the studied locations[@].

| Locations (Villages) | DM gm pot$^{-1}$ | Nutrient - ppm | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
| Madabegh | 4.34 | 879.07 | 34.82 | 54.79 | 18.29 | 7.84 | 21.53 | 18.81 | 9.42 |
| Mankabad | 6.53 | 800.49 | 28.30 | 55.53 | 16.67 | 12.43 | 4.70 | 17.93 | 23.71 |
| Ellwan | 4.58 | 470.23 | 36.66 | 36.66 | 20.19 | 30.13 | 1.54 | 14.86 | 20.64 |
| Bani Hussien | 6.26 | 643.51 | 29.42 | 55.52 | 19.88 | 6.93 | 1.68 | 12.33 | 16.78 |
| Bani Ghalib | 4.40 | 585.59 | 22.82 | 64.02 | 18.64 | 8.92 | 10.51 | 2.53 | 3.40 |
| Gahdam | 3.54 | 609.99 | 25.49 | 57.77 | 20.11 | 16.90 | 0.99 | 4.20 | 10.23 |
| Mean | | 664.81 | 29.58 | 54.05 | 18.96 | 13.86 | 8.49 | 10.11 | 14.03 |
| $LSD_{0.05}$ | -- | 110.76** | 4.50** | 8.21** | n.s. | 15.76* | 5.36** | 4.70** | 6.02** |

* and ** significant at probability level ≤ 0.05 and 0.01, respectively
n.s not significant [@] Each value represents a mean of three sites

The concentrations of Fe, Mn, Cu, Cd, and Pb, in wheat straw, show significant differences between locations (Table 10). However, the variations in concentrations of all studied metals, but not Cu, in wheat seeds were significant. Straw and seeds of wheat plants grown on locations near to the source point of sewage-waste water (Madabegh and Mankabad) show high concentrations of micronutrients and heavy metals compared to those far away from the source point of pollution. Lead concentrations, for example, in straw of wheat plants grown on soils of Bani-Ghalib and Gahdam were 8.67 and 7.03 ppm, respectively, compared to 15.6 and 13.8 ppm in straw of wheat plants grown on the soils of Madabegh and Mankabad, respectively. In general view, the concentrations of heavy metals and micronutrients in wheat plants either significantly decreased or show no significant variations with increasing the distance from the source point of pollutants. This is, in general, in agreements with the variations in extractable heavy metals and micronutrients between soils of different locations. The concentrations of heavy metals in most plants grown on the studied locations were decreased with increasing the distance from Madabegh and Mankabad. As mentioned before, this is a direct result of decreasing the amounts of heavy metals pollutant in the down-stream of the irrigation cannel.

**Table(10):** Dry matter accumulation ($gm/pot^{-1}$) and concentrations of micronutrients and heavy metals in straw and seeds of wheat plants grown on polluted soils of the studied locations[@].

| Locations (Villages) | DM gm $pot^{-1}$ | Nutrient - ppm | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
| | Wheat straw | | | | | | | | |
| Madabegh | 3.38 | 730.81 | 43.57 | 56.04 | 16.34 | 13.80 | 15.60 | 14.90 | 20.12 |
| Mankabad | 3.66 | 720.92 | 34.52 | 54.44 | 21.22 | 6.64 | 13.80 | 10.08 | 18.86 |
| Ellwan | 3.09 | 586.62 | 30.48 | 49.53 | 27.39 | 9.75 | 8.40 | 14.06 | 19.34 |
| Bani Hussien | 3.71 | 666.80 | 30.55 | 43.92 | 25.39 | 11.23 | 7.13 | 10.41 | 20.59 |
| Bani Ghalib | 3.58 | 662.68 | 29.17 | 42.35 | 28.33 | 12.19 | 8.67 | 12.84 | 18.39 |
| Gahdam | 3.58 | 511.79 | 33.54 | 75.88 | 23.38 | 15.19 | 7.03 | 11.76 | 20.29 |
| Mean | 3.50 | 646.60 | 33.64 | 53.69 | 23.68 | 11.47 | 10.10 | 12.34 | 19.60 |
| $LSD_{0.05}$ | -- | 115.69** | 4.84** | n.s. | 3.78** | 3.44** | 4.96** | n.s. | n.s. |
| Wheat seed | | | | | | | | | |
| Madabegh | 3.39 | 287.10 | 28.08 | 24.65 | 12.52 | 7.27 | 7.27 | 2.52 | 5.44 |
| Mankabad | 3.29 | 435.46 | 34.35 | 24.66 | 13.57 | 5.58 | 5.58 | 8.95 | 8.05 |
| Ellwan | 2.70 | 325.18 | 36.98 | 19.64 | 13.32 | 4.96 | 4.96 | 1.60 | 10.62 |
| Bani Hussien | 3.81 | 377.96 | 30.54 | 26.55 | 14.19 | 7.76 | 7.76 | 11.83 | 2.67 |
| Bani Ghalib | 3.34 | 295.47 | 35.60 | 35.53 | 11.84 | 6.83 | 6.83 | 2.51 | 2.23 |
| Gahdam | 3.60 | 317.17 | 40.38 | 29.44 | 11.26 | 6.13 | 6.13 | 2.83 | 7.84 |
| Mean | 3.35 | 339.72 | 34.32 | 26.74 | 12.78 | 6.42 | 6.42 | 5.04 | 6.14 |
| $LSD_{0.05}$ | -- | 90.69* | 4.67** | 5.78** | n.s. | 1.51** | 1.51** | 3.60** | 4.00** |

* and ** significant at probability level ≤ 0.05 and 0.01, respectively n.s not significant. [@] Each value represents a mean of three sites

The concentrations of metals in reproductive organs, where plants tend to store the metabolic products, may be more sensitive for the variations in the concentrations of metals in root zone. Therefore, regarding to study the effects of heavy metal pollutants on plants, the concentrations of these pollutants in reproductive plant parts, especially seeds should be considered well. For instance, as presented in Table 10, the concentrations of Cd in seeds of wheat plants grown on the soils of all studied locations ranged between 4.96 and 7.76 with an average of 6.42 ppm, which is higher than the normal range of Cd concentrations (0.9 – 2.6 mg $kg^{-1}$ DM) as reported by Kabata-Pendias and Pendias (1992). In general, it may be stated that concentrations of Zn and Mn in seeds of wheat plants grown on the soils of the studied region were in the normal range. Contrary to that, the concentrations of Fe, Cu, Cd, Pb, Ni, and Cr in seeds of wheat plants grown on these soils were higher than the normal ranges reported by Kabata Pendias and Pendias (1992).

**Judging the extraction procedures suitable for micronutrients and heavy metals:**

Melsted and Peck. (1973) defined the available form of nutrient by saying "By available nutrient one usually means the chemical form or forms of an essential plant nutrient in soil whose variation in amount is reflecting in variations in plant growth and plant yield". In polluted soils there are many forms of metals with different degree of solubility's that may be enable them to enter the available pool at any time. Therefore, the extraction procedures used for studying the presence of metals pollutants in soils at any time should have a good association with one of the growth responses whose variations are associated with the variations in the size of the available pool. Nutrient uptake by plants is considered here as one of those crop responses that could be used to judge the suitability of the extraction procedure. The studied procedures for extracting the available amounts of micronutrients and heavy metals from polluted soils were judged using the correlations coefficients between the amounts of extracted metals and metal uptake by corn and wheat plants grown on these soils. Table 11 and 12 represent the correlation coefficients between extractable metals by each of the studied extraction procedures and the metal uptake in corn and wheat plants grown on the soils of the studied area.

First of all, $HCl+H_2SO_4$ extraction procedure did not gave any significant correlation with amounts of micronutrients and heavy metals, but Cd, taken up by either corn and wheat plants, the results which indicated that this procedures is not a suitable one for extracting metals from polluted soils. This extraction procedure gave the lowest amounts of micronutrients and heavy metals, and did not predict the availability of the studied metals well. Therefore, the correlation coefficients between the extracted metals and metals uptake by corn and wheat plants were small and not significant (at p levels $\leq 0.05$). Moreover, the results of this procedure were not persistent and show wide variations between soils and after the harvesting of different crops, therefore the $HCl+H_2SO_4$ extraction procedure could not be used successfully for predicting the status of metal pollutants in soils. Cox and Kamprath (1972) stated that one of the criteria upon which the extraction procedures is selected is that "the extracted amount should be correlated with the growth and response of each crop to that nutrient under various conditions".

DTPA extraction procedure gave significant correlations with Fe, Mn, Zn, Cu, and Pb uptake in corn stover (Table 11). In the case of wheat plants the procedure did not give any significant correlations with any of the studied metals taken up in wheat straw, however, the procedure gave significant correlation with Fe, Mn, Zn, Cu, and Cd in wheat seeds, and with total uptake of Fe, Zn, and Cu (Table 12). On the other hand, the DTPA extraction procedure did not give any significant correlations with the amounts of any one of studied heavy metals either in corn or in wheat plants.

The DTPA micronutrient extraction method, developed by Lindsay and Norvell (1978), is a nonequilibrium extraction for estimating the potential soil bioavailability of Zn, Cu, Mn, and Fe for neutral and calcareous soils. It is, in general, considered by many researchers as a specialized procedure. Vose and Randall (1962) stated that the three important metals pools in supplying micronutrients for plant during the growing season are (i) $H_2O$-soluble; (ii) exchangeable; and (iii) adsorbed, chelated or complexed. The extraction procedure for micronutrients, therefore, should extract a portion or all of these three pools. The DTPA procedure predicts well the overall effects of these three pools on the availability of Fe, Mn, Zn, and Cu in normal soils. Therefore, using the DTPA to predict the amounts of other heavy metals in polluted soils may not be correct, the obtained results revealed that.

**Table(11):** Correlation coefficients between extractable metals by different extraction procedures and metals uptake by corn plants.

| Extractants | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
|---|---|---|---|---|---|---|---|---|
| | | | | Corn stover | | | | |
| $HCl+H_2SO_4$ | -0.23 | -0.15 | -0.09 | -0.09 | -0.02 | -0.10 | -0.09 | . |
| DTPA | 0.44** | 0.72** | 0.43** | 0.27* | 0.11 | 0.26* | 0.18 | . |
| Ab-DTPA | 0.47** | 0.63** | -0.25* | 0.03 | -0.20 | -0.22 | 0.07 | -0.24* |
| EDTA | 0.02 | 0.34** | 0.10 | 0.56** | 0.62** | 0.26* | 0.17 | -0.16 |
| EDTA-AAc | -0.08 | 0.21 | -0.09 | 0.11 | 0.30** | -0.04 | 0.29* | 0.26* |
| EDTA-AAAc | 0.32** | 0.44** | 0.30** | 0.32** | 0.18 | 0.34** | 0.06 | . |
| EDTA-Carbonate | 0 | 0.04 | -0.02 | 0.10 | 0.04 | 0.16 | 0.03 | -0.10 |
| EDTA-Citrate | -0.33** | 0.28* | -0.30* | 0.13 | -0.07 | 0.19 | -0.03 | 0.07 |

*, ** = significant at: $p \leq 0.05$ and 0.01 probability levels, respectively.

The performance of EDTA extraction procedures in predicting the bioavailability levels of metals in polluted soil showed wide variations with the type of extracted metals, the type of plants, and the components of the extraction solutions.

**Table(12):** Correlation Coefficients between extractable heavy metals by different extraction procedures after wheat planting and wheat straw, seeds and total plant uptake:

| Extractants | Fe | Zn | Mn | Cu | Cd | Pb | Ni | Cr |
|---|---|---|---|---|---|---|---|---|
| Wheat straw | | | | | | | | |
| $HCl+H_2SO_4$ | -0.03 | -0.19 | 0.03 | 0.00 | 0.242* | -0.06 | 0.12 | 0.09 |
| DTPA | 0.11 | 0.06 | 0.04 | 0.02 | -0.05 | 0.02 | 0.06 | -0.16 |
| Ab-DTPA | -0.05 | -0.16 | -0.02 | -0.13 | 0.15 | 0.22 | 0.13 | -0.03 |
| EDTA | -0.03 | 0.24* | -0.05 | -0.33** | 0.13 | 0.23 | -0.13 | 0.20 |
| EDTA-AAc | 0.18 | 0.49** | 0.04 | -0.14 | -0.01 | 0.34** | -0.21 | -0.04 |
| EDTA-AAAc | 0.09 | 0.08 | 0.244* | -0.26* | 0.02 | 0.26* | -0.04 | -0.11 |
| EDTA-Carbonate | 0.14 | 0.55** | -0.17 | -0.23* | 0.08 | 0.31** | -0.04 | -0.01 |
| EDTA-Citrate | 0.05 | -0.08 | 0.25* | -0.23 | 0.12 | -0.23 | -0.12 | . |
| Wheat seed | | | | | | | | |
| $HCl+H_2SO_4$ | -0.09 | 0.09 | 0.11 | 0.02 | 0.28* | 0.19 | -0.05 | 0.11 |
| DTPA | 0.49** | 0.63** | 0.54** | 0.53** | 0.43** | 0.12 | -0.16 | 0.05 |
| Ab-DTPA | 0.34** | 0.42** | -0.13 | -0.06 | -0.15 | -0.03 | 0.01 | 0.15 |
| EDTA | -0.17 | -0.21 | 0.06 | 0.18 | -0.14 | 0.11 | 0.28* | 0.13 |
| EDTA-AAc | -0.07 | -0.21 | -0.05 | 0.07 | 0.11 | -0.13 | 0.10 | -0.06 |
| EDTA-AAAc | 0.10 | -0.12 | -0.04 | 0.08 | -0.01 | 0.03 | 0.30** | 0.34** |
| EDTA-Carbonate | 0.06 | -0.15 | -0.18 | -0.05 | -0.01 | 0.14 | -0.04 | 0.04 |
| EDTA-Citrate | -0.09 | -0.07 | -0.04 | -0.14 | 0.05 | -0.04 | -0.13 | . |
| Whole wheat plant | | | | | | | | |
| $HCl+H_2SO_4$ | -0.07 | -0.09 | 0.06 | 0.01 | 0.33** | 0.08 | 0.04 | 0.14 |
| DTPA | 0.35** | 0.41** | 0.19 | 0.24* | 0.16 | 0.09 | -0.09 | -0.09 |
| Ab-DTPA | 0.15 | 0.13 | -0.05 | -0.13 | 0.06 | 0.15 | 0.09 | 0.08 |
| EDTA | -0.11 | 0.05 | -0.03 | -0.19 | 0.04 | 0.24* | 0.14 | 0.24* |
| EDTA-AAc | 0.09 | 0.22 | 0.02 | -0.09 | 0.05 | 0.17 | -0.06 | -0.07 |
| EDTA-AAAc | 0.12 | -0.01 | 0.22 | -0.18 | 0.01 | 0.21 | 0.22 | 0.14 |
| EDTA-Carbonate | 0.13 | 0.30** | -0.21 | -0.21 | 0.06 | 0.32** | -0.05 | 0.02 |
| EDTA-Citrate | -0.01 | -0.10 | 0.22 | -0.25* | 0.12 | -0.20 | -0.18 | . |

*, ** = significant at: $p$ / 0.05 and 0.01 probability levels, respectively.

When the extraction solution contained EDTA only the extracted amounts of Mn, Cu, Cd, and Pb were significantly ($p \leq 0.05$) correlated with the amounts of these metals taken up by corn plants (Table 11). In the case of wheat plants, the significant correlations were found only for Zn and Cu in straw and Ni in wheat seeds (Table 12). The presence of acetate anions with EDTA enhanced its power for extracting the available metals. The amounts of available Cd, Ni, and Cr extracted with EDTA-AAc extraction solution significantly ($p \leq 0.05$) correlated with the amounts of metals taken up by corn plants, and the amounts of available Fe, Mn, Zn, Cu, and Pb extracted with EDTA-AAAc extraction solution significantly ($p \leq 0.05$ ) correlated with the amounts of metals taken up by corn plants. With wheat plants, significant ($p \leq 0.05$) correlations were obtained only for Zn and Pb in straw using EDTA-AAc, and Mn, Cu, and Pb in straw using EDTA-AAAc (Table 12). The extractable metals identified by EDTA methods exhibited relatively poor or no correlation with the other metal content of wheat plants. This discrepancies in the results emphasized that the EDTA extraction procedures were not specific for all micronutrients and heavy metal extractions. The presence of acetate anions in the extraction solutions enhanced the potential extracting power of the solution, and resulted in extracting high amounts of metals from different pools, including the occluded and poorly crystalline forms. Thus, the amounts of metals extracted by these procedures were either weekly correlated or did not correlated at all with the amounts of metals uptake by plants. These results lead to the general conclusion that no one extraction procedures is suitable for testing the status of all metals in all soils after all crops. Precautions should be taken when deciding which extraction solution should be used.

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# قابلية الاستخلاص و التيسر لبعض العناصر المعدنية في الأراضي الملوثة بأسيوط.*

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تم إجراء هذه الدراسة بهدف تحديد انسب المحاليل التي يمكن استخدامها لاستخلاص بعض العناصر الصغرى و العناصر الثقيلة في الأراضي الملوثة بمياه المجارى.

و قد تم أخذ 18 قطاع تمثل 6 قرى تروى بمياه المجارى الملوثة من أكثر من 45 عام بمحافظة أسيوط حيث أخذت عينات سطحية و تحت سطحية. كما زرعت هذه الأراضي بنباتي الذرة و القمح لربط المحتوى العنصري لهذه النباتات بمحتوى التربة من نفس العناصر ( الحديد – المنجنيز – الزنك – النحاس – الكادميوم –الرصاص – النيكل – الكروم ).

و من هذه الدراسة أمكن التوصل إلى كثير من النتائج الهامة مع تحديد أنسب المستخلصات التي يمكن استخدامها في مثل هذه النوعية من الأراضي ( الأراضي الملوثة فقط ) .

كما توصى هذه الدراسة إلى التوسع في هذه النوعية من الأبحاث لتشمل بقية الأراضي الغير ملوثة بأنواعها المختلفة.

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