INTRODUCTION

The Gabal El Asfar Old Farm is a government-owned fruit plantation situated on the north-eastern edge of Cairo on land that was originally desert. The farm has been irrigated with sewage for more than 80 years and there are environmental and health concerns about soil, water and crop contamination at the site and also about potentially toxic heavy metals. The extent of soil contamination at the site provides a model of the potential long-term effects of heavy metals on crops for sewage sludge-treated soils in Egypt. This adopts an approach followed by European scientists in assessing potential long-term impacts of recycling sewage sludge on agricultural land using sites with long histories of sludge application and referred to as so-called ‘historic sites’. These are field sites which have been treated with sludge for many years in operational practice and where the concentrations of heavy metals have been significantly raised above background values, representing potentially a worse-case of soil contamination. Investigations using historic site soils provide a valuable adjunct to the classically designed and controlled field trials for assessing the environmental effects of sewage sludge. Their value is due to the long period of time required for repeated sludge application to accumulate heavy metals in soil. If it can be demonstrated that there are minimal long-term risks to the environment under these worse-case conditions, this provides additional assurance and security about the long-term safety and sustainability of controlled recycling sewage sludge on agricultural land.

Several investigators studied the effect of long term irrigation of sewage in Gabal El Asfar farm. [1, 2, 3, 4, 5]. They found that several conditions to irrigation with sewage effluent the higher level of PTEs accumulation in soil. In India, Rattan et al. reported that Sewage irrigation for 20 years resulted to significant build-up of DTPA-extractable Zn (208%), Cu (170%), Fe (170%), Ni (63%) and Pb (29%) in sewage-irrigated soils over adjacent tubewell water-irrigated soils, whereas Mn was depleted by 31%. Soils receiving sewage irrigation for 10 years exhibited significant increase in Zn, Fe, Ni and Pb, while only Fe in soils was positively affected by sewage irrigation for 5 years. Among these metals, only Zn in some samples exceeded the phytotoxicity limit.
Materials & Methods
Two surveys of the Gabal El Asfar Old Farm were undertaken as part of Cairo Sludge Disposal Study to assess the long-term effects of heavy metals in sludge-treated soil on crop quality. In the first survey, the relationships between total and DTPA extractable heavy metals in soil and concentrations in citrus fruit were examined. Concentrations of heavy metals in leaves of citrus were measured in the second survey and related to total and DTPA extractable metals in soil. The results of both surveys are summarized here and an integrated interpretation of the potential long-term implications of heavy metals in sewage sludge recycled on agricultural land in Egypt.

Since the site is only irrigated with partially treated effluent, this represents a potentially severe disease exposure risk to labourers working on the land and also to consumers of the produce. Some of the crops grown would represent a high risk to consumers, such as salads and other crops that may be eaten raw. It was beyond the scope of the Study to investigate these issues (although there is an urgent need to do so), but advantage was taken of the soil samples taken during the site surveys for examination of residual pathogens and parasites since these would provide a useful comparison with soil samples taken from selected field trials carried out by the Study. The samples collected for soil and plants are presented in Table 1.

Table 1: Areas selected for sampling, date of first planting and current cropping in the three main area of Gabal El Asfar Old Farm

| Plantation Date of Gearment | Duration of Sewage Irrigation | Current Planting | Intercropping at Time of Sampling |
|-----------------------------|--------------------------------|------------------|----------------------------------|
| **Southern area**           |                                |                  |                                  |
| 53                          | 1928                           | 69               | Seedless orange                  |
| 52                          | 1954                           | 69               | Seedless orange                  |
| 13                          | 1928                           | 67               | Valencia orange                  |
| 75                          | 1953                           | 64               | Mandarin                         |
| 76                          | 1953                           | 64               | Mandarin                         |
| 67                          | 1954                           | 43               | Orange                           |
| 65                          | 1958                           | 39               | Orange                           |
| 22                          | 1960                           | 37               | Valencia orange                  |
| 23                          | 1960                           | 37               | Orange                           |
| 12                          | 1962                           | 35               | Lemon                            |
| 61                          | 1963                           | 32               | Orange                           |
| 62                          | 1965                           | 32               | Orange                           |
| 20                          | 1972                           | 25               | Orange                           |
| 21                          | 1972                           | 25               | Valencia orange                  |
| 40                          | 1984                           | 13               | Lemon                            |
| 73                          | 1989                           | 8                | Mandarin                         |

| **Northern area**           |                                |                  |                                  |
| 2                           | 1914                           | 83               | Pecan                            |
| 6                           | 1914                           | 83               | Pecan + lemon                     |
| Forest                      | 1956                           | 42               | Orange (balady)                  |
| A                           | 1956                           | 43               | Valencia orange                  |
| B                           | 1956                           | 43               | Valencia orange                  |
| C                           | 1956                           | 43               | Valencia orange                  |
| D                           | 1956                           | 43               | Valencia orange                  |
| 28                          | 1962                           | 35               | Soliman pasha                    |
| 29                          | 1962                           | 35               | Pecan                            |
| 2                           | 1986                           | 11               | Mandarin                         |
| 5                           | 1987                           | 10               | Seedless orange                  |
| 7                           | 1987                           | 10               | Valencia orange                  |
| 10                          | 1991                           | 6                | Khalili orange                   |
| 14                          | 1992                           | 4                | Valencia orange                  |
| 2                           | 1993                           | 4                | Mandarin                         |

| **Reclamation area**        |                                |                  |                                  |

Chemical analyses for soil (0-30cm) depth and plant samples were carried out according to the methods described by [6] and [7] and total potential toxic elements [7]. The data were statistically analyzed using software package (Cohort2).

RESULTS AND DISCUSSION
The heavy metal contents of citrus leaves and fruit (orange - eleven sampling sites; mandarin - four sampling sites), and total and DTPA extractable concentrations in soils were measured in samples collected from different areas of the Farm during two site surveys.

Total and DTPA concentrations of heavy metals in the surveyed soils showed significant enrichment by long-term irrigation with sewage effluent. For example, the maximum total concentrations of Zn and Cu were 530 and 366 mg kg⁻¹, respectively, representing a potential risk to crop yields (Tables 2 and 3). The maximum Cd concentration detected was 9 mg kg⁻¹ and may be a potential risk to the human food chain from uptake into staple crops grown at the farm.

Table 1: Statistical summaries of total and extractable trace elements in soil and concentrations in citrus fruit from Gabal El Asfar Old Farm (Survey 1)

|            | Zn      | Cu      | Ni      | Cd      | Pb      | Cr      |
|------------|---------|---------|---------|---------|---------|---------|
| Minimu     | 180     | 50      | 1       | 1       | 5       | 80      |
| Maximu     | 530     | 117     | 51      | 9       | 70      | 230     |
| Mean       | 325     | 81      | 23      | 3       | 21      | 158     |
| Median     | 310     | 82      | 21      | 2       | 10      | 160     |
| EC         | 300     | 140     | 75      | 3       | 300     | 1       |
| lim³(1)    |         |         |         |         |         |         |

DTPA extractable soil concentration (mg kg⁻¹)

|            | Zn      | Cu      | Ni      | Cd      | Pb      | Cr      |
|------------|---------|---------|---------|---------|---------|---------|
| Minimu     | 16      | 8.0     | 0.56    | 0.10    | 2.0     | 0.2     |
| Maximu     | 56      | 27.0    | 7.40    | 0.30    | 10.0    | 1.2     |
| Mean       | 33      | 13.5    | 3.21    | 0.17    | 4.8     | 0.6     |
| Median     | 32      | 11.4    | 3.30    | 0.18    | 4.0     | 0.6     |

Tissue concentration in citrus fruit (orange and mandarin) (mg kg⁻¹ DM)

|            | Zn      | Cu      | Ni      | Cd      | Pb      | Cr      |
|------------|---------|---------|---------|---------|---------|---------|
| Minimu     | 0.37    | 0.12    | 0.02    | 0.02    | 0.24    | 0.36    |
| Maximu     | 2.60    | 0.48    | 1.20    | 0.10    | 2.10    | 3.60    |
| Mean       | 1.03    | 0.22    | 0.38    | 0.07    | 1.01    | 1.92    |
| Median     | 1.00    | 0.24    | 0.22    | 0.09    | 0.90    | 2.10    |

Note: (1) European maximum soil limit value (CEC Directive 86/278/EEC)

DTPA extractable metals were significantly (P<0.001) correlated with the total contents of Zn (r=0.91**), Cu (r=0.83**), Ni (r=0.62**) and Pb (r=0.55**) in soil when data from both surveys were pooled for statistical evaluation (Table 4 and Figure 1). There was also evidence of a weak relationship between DTPA extractable Cd and the total soil concentration, although this only just achieved significance at P=0.05. However, when bioavailability was assessed on the basis of data from the chemical analysis of citrus fruits and leaves, there were no significant relationships apparent between the concentrations of heavy metals in plant tissues and the corresponding amounts of total or DTPA extractable metals in soil.
These data confirm international experience with the sludge use in agriculture, which demonstrates that both Cu and Ni are unlikely to be phytotoxic when normal sludges produced at WWTPs are applied to farmland at agronomic rates in practice.[8,9] This is because Cu is relatively tightly bound to soil organic matter limiting its mobility in sludge-treated soil and plants are also able to control the accumulation of Cu in their tissues much more so than for Zn or Ni. Nickel poses little actual risk to crops in practice because concentrations in sludge are small relative to its phytotoxic threshold content in sludge-amended soil. The results presented here were consistent with earlier surveys of citrus at the Old Farm, which were optimistic about the positive, nutritional benefits of sewage irrigation for crop production.

Zinc is the element associated principally with the risk of phytotoxicity in sludge-treated agricultural land. The Zn concentrations detected in soil sampled in the second survey of Gabal El Asfar Old Farm were modest and below the considered phytotoxic concentration. Nevertheless, the Zn content of soil was raised by almost 300 % above background, but there was no evidence of increased Cu content in citrus leaves or fruit (Table 5; Figures 2 and 3). These data indicate that Zn phytotoxicity is very unlikely from recycling sewage sludge to citrus crops. Cadmium is the only element of concern in terms of the risk to human health from uptake into food crops grown on sludge-treated soil. The total Cd concentration in soil at Gabal El Asfar Old Farm was raised to a value 3 times the maximum EU limit for this element in sludge-treated agricultural soil. Despite the marked increase in soil Cd content, there was no detectable transfer into citrus leaves or fruit (Figure 4). The absence of Cd uptake into citrus fruit is to be expected because fruits are amongst the least sensitive plant parts to Cd accumulation. These data emphasise the minimal risk to the human diet from Cd in fruit crops grown on sludge-treated soil.

Leaf tissues of a number of inter-crops at Gabal El Asfar were also sampled in the survey including maize (n = 9) and one sample each of egg plant, pepper and potato. In all cases, leaf tissue concentrations were low and in some cases Cu status was below the deficiency threshold. The Cd content in leaves was small and generally <0.02 mg kg\(^{-1}\). DM.

Lead was readily extracted from soil by the DTPA reagent (Figure 1), but is strongly bound to the soil matrix and is not transferred to crop tissues. The chemical analysis of citrus leaves and fruit confirmed the Pb accumulation in plant parts to be negligible (Table 4). Uptake of Cr into plant tissues is also restricted because of the capacity of soil to bind this element strongly in unavailable forms. In this case, however, the DTPA extractable fraction of this element was small and reflected the low bioactivity of Cr in sludge-treated soil (Figure 1 and Table 4). DTPA is widely used in nutrient diagnosis as a tool for assessing potential soil deficiencies. However, these results show that the degree of extractability of a particular element does not necessarily provide a measure of its bioavailability to crops for assessing toxicological risks of heavy metals to crops or human health in sludge-treated desert soil. Soil extraction with DTPA was not a reliable indicator of bioavailability to citrus in reclaimed desert soil.

Similar results were reported by [1] who indicted that after 85 years sewage farming at El-Gabal Al-Asfar, PTEs like Fe, Zn, Mn, Cu, and Pb accumulated in the upper soil, [2,3] found that the longer the period of irrigation with sewage effluent the higher the level of PTEs accumulation in soil and concluded that the progressive increase of PTEs in the soil represents serious risk to the cultivated plants. [4] found that soil irrigated for 75 years with sewage effluent showed increment in the total content of PTEs compared to control. The obtained values were 316.9, 276.4, 9.31, 43.81 and 213.3 ug / g soil for Zn, Cu, Cd, Ni, and Pb, respectively. They added that all values

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**Table 3: Statistical summaries of total and extractable heavy metals in soil and concentrations in citrus leaves from Gabal El Asfar Old Farm (Survey 2)**

| Element | Total soil concentration (mg kg\(^{-1}\)) | DTPA extractable soil concentration (mg kg\(^{-1}\)) | Tissue concentration in citrus leaves (mg kg\(^{-1}\) DM) |
|---------|------------------------------------------|---------------------------------------------------|------------------------------------------------------|
| Zn      | Min 32, Max 143, Mean 95, Median 103     | Min 4.0, Max 18.8, Mean 15.2, Median 16.6         | Min 1.2, Max 150, Mean 24.8, Median 20.9            |
| Cu      | Min 7, Max 92, Mean 45, Median 38        | Min 9.8, Max 42.5, Mean 15.8, Median 13.6         | Min 0.8, Max 9.4, Mean 3.8, Median 3.5             |
| Ni      | Min 0.2, Max 4.6, Mean 1.6, Median 7.0  | Min 0.02, Max 0.8, Mean 0.21, Median 0.14         | Min 0.0, Max 0.4, Mean 0.1, Median 0.1             |
| Cd      | Min 16, Max 920, Mean 160, Median 367   | Min 0.12, Max 0.24, Mean 0.05, Median 0.04        | Min 0.2, Max 0.2, Mean 0.2, Median 0.1             |
| Pb      | Min 89, Max 366, Mean 15.8, Median 24.8 | Min 0.09, Max 0.24, Mean 0.14, Median 0.01        | Min 0.01, Max 0.05, Mean 0.05, Median 0.03        |

**Table 4: Linear regression models (y = a + bx) of the relationships between the DTPA extractable and total concentrations of heavy metals in soil for pooled data from Survey 1 and 2**

| Element | Intercept (a) | Slope (b) | Correlation coefficient (r) | Significance (P) |
|---------|---------------|-----------|-----------------------------|------------------|
| Zn      | 7.4           | 0.08      | 0.91                        | <0.001***        |
| Cu      | 6.0           | 0.13      | 0.83                        | <0.001***        |
| Ni      | 1.3           | 0.05      | 0.64                        | <0.001***        |
| Cd      | 0.14          | 0.03      | 0.29                        | 0.03*            |
| Pb      | 2.8           | 0.08      | 0.65                        | <0.001***        |
| Cr      | 0.09          | 0.001     | 0.38                        | 0.004**          |

**Table 2: Correlation coefficients (r) of relationships between heavy metal concentrations in soil and citrus at Gabal El Asfar Old Farm**

| Element | Fruit (n = 15) | Leaves (n = 41) |
|---------|----------------|-----------------|
|        | Total          | DTPA            | Total          | DTPA            |
| Zn\(^{(1)}\) | -0.11ns        | -0.03ns         | -0.12ns        | -0.05ns         |
| Cu      | 0.27ns         | -0.03ns         | 0.36ns         | 0.24ns          |
| Ni      | 0.06ns         | -0.35ns         | 0.38ns         | 0.18ns          |
| Cd      | 0.39ns         | 0.35ns          | 0.35ns         | 0.47ns          |
| Pb      | 0.24ns         | -0.24ns         | -0.10ns        | 0.04ns          |
| Cr      | 0.09ns         | 0.01ns          | 0.01ns         | -0.05ns         |

Note: ns, not significant at P=0.05

\(^{(1)}\) Outlier value of 150 mg Zn kg\(^{-1}\) in leaves omitted from the correlation analysis on leaf tissue content
were remarkably over the safe values of these PTEs that should be found in soils. In Germany [5] in a study aimed to determine whether >110 years of sewage application has led to recognizable changes in the metal chemistry of soils from former sewage farms came to similar conclusion.

Fig. 1: DTPA extractable concentrations of heavy metals in relation to the total heavy metal content in soil from Gabal El Asfar Old Farm (■, Survey 1; ◆, Survey 2).

Fig. 2: Concentrations of (a) Zn and (b) Cu in leaves of citrus in relation to the total content in soil from Gabal El Asfar Old Farm (Survey 2).

Fig. 3: Concentrations of (a) Zn and (b) Cu in leaves of citrus in relation to the DTPA extractable contents in soil from Gabal El Asfar Old Farm (Survey 2).

Fig. 4: Cadmium concentrations in citrus fruit in relation to (a) total and (b) DTPA extractable Cd content in soil at Gabal El Asfar Old Farm (Survey 1).
CONCLUSION

The study of long-term contamination of soil with heavy metals has not demonstrated a potential risk to crop quality and yield or human health from the slow accumulation of heavy metals in sludge-treated agricultural soil. Heavy metal concentrations in plant tissues remained low and within normal ranges despite significant increases in soil content after long-term irrigation with sewage effluent. Concentrations of heavy metals in plant tissues were not related to total or DTPA extractable metals in contaminated soil. DTPA may not be a sufficiently reliable indicator of actual phytoavailability of trace elements in sludge-treated soil, although it is accepted that DTPA is widely used in nutrient diagnosis assessment.

This study of long-term contamination of reclaimed desert soil has demonstrated, and provides assurance of, the minimal risk to crop quality, yield and human health from heavy metals applied to soil in sewage sludge for desert reclamation and fruit production. A more detailed analysis of dietary exposure to Cd under Egyptian conditions is recommended, following the approaches adopted in the UK and US for setting Cd soil limits or loading rates for this element. Such information would be valuable in formulating the long-term management strategy of the site and for sludge and effluent reuse generally in Egypt.

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