

## Effects of Long-Term Cultivation on Soil Physicochemical Properties and Heavy Metal Accumulation

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### ABSTRACT

This study evaluated the effects of long-term cultivation on the physicochemical properties of soil, including pH, organic matter (OM), electrical conductivity (EC), cation exchange capacity (CEC), and concentrations of some heavy metals (HMs) such as Cr, Cu, Cd, Mn, Zn, Ni, Fe, and Pb of Open Farm (OF) soils. A total of 210 soil samples were collected from six depths (0-60 cm). The findings revealed slight alterations in physicochemical properties resulting from cultivation and chemical fertilizers. Heavy metal concentrations in OF soils were below maximum permissible limits, but Cu, Cd, Mn, and Zn accumulated compared to uncultivated soils. The heavy metals pollution index (HPI) indicated low pollution for Fe in OF soils ( $PI \leq 1$ ), with HPI values decreasing with increasing soil depth. The Geoaccumulation index (Igeo) indicated that Open-farm soils were uncontaminated or moderately contaminated with Mn. A strong positive correlation between physicochemical characteristics and heavy metals in OF soils suggests a common pollution source, whereas negative correlations indicate different pollution sources. Principal component analysis (PCA) identified three major components in soil, each associated with specific heavy metals. The study indicated that Cd, Cu, and Zn originated from agricultural activities, while Cr, Ni, Fe, and Mn were derived from natural soil-forming processes.

## تأثيرات الزراعة طويلة الأمد على الخصائص الفيزيائية والكيميائية للتربة وتراكم المعادن الثقيلة

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| المخلص                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | الكلمات المفتاحية                                                                         |
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| قيمت هذه الدراسة تأثيرات الزراعة طويلة الأمد على الخصائص الفيزيائية والكيميائية للتربة في المزارع المفتوحة (OF)، بما في ذلك درجة الحموضة (pH)، والمادة العضوية (OM)، والتوصيل الكهربائي (EC)، وسعة التبادل الكاتيوني (CEC)، وتركيزات بعض المعادن الثقيلة (HMs) مثل الكروم (Cr)، والنحاس (Cu)، والكاديوم (Cd)، والمنغنيز (Mn)، والزنك (Zn)، والنيكل (Ni)، والحديد (Fe)، والرصاص (Pb). جُمعت ما مجموعه 210 عينات تربة من ستة أعماق (0-60 سم). وأظهرت النتائج تغيرات طفيفة في الخصائص الفيزيائية والكيميائية ناجمة عن الزراعة واستخدام الأسمدة الكيميائية. كانت تركيزات المعادن الثقيلة في تربة المزارع المفتوحة أقل من الحدود القصوى المسموح بها، إلا أن عناصر النحاس والكاديوم والمنغنيز والزنك أظهرت تراكمًا مقارنة بالتربة غير المزروعة. وأشار مؤشر تلوث المعادن الثقيلة (HPI) إلى وجود مستوى منخفض من تلوث الحديد في تربة المزارع المفتوحة ( $PI \leq 1$ )، حيث تناقصت قيم المؤشر مع زيادة عمق التربة. كما أظهر مؤشر التراكم الجيولوجي (Igeo) أن تربة المزارع المفتوحة كانت غير ملوثة أو ملوثة بشكل معتدل بالمنغنيز. وتشير العلاقة الإيجابية القوية بين الخصائص الفيزيائية والكيميائية والمعادن الثقيلة في تربة المزارع المفتوحة إلى وجود مصدر مشترك للتلوث، في حين تشير العلاقات السلبية إلى مصادر تلوث مختلفة. وحدد تحليل المكونات الرئيسية (PCA) ثلاثة مكونات رئيسية في التربة، ارتبط كل منها بمعادن ثقيلة محددة؛ حيث أشارت الدراسة إلى أن الكاديوم والنحاس والزنك نشأت عن الأنشطة الزراعية، بينما نتج الكروم والنيكل والحديد والمنغنيز عن العمليات الطبيعية لتكوين التربة. | المزرعة المفتوحة<br>الارتباط<br>المعادن الثقيلة<br>مؤشر التلوث<br>تحليل المكونات الرئيسية |

## Introduction

Air, water, and soil pollutants are harmful chemical or physical substances that degrade environmental quality and threaten human and ecosystem health. There are numerous local studies addressing air pollution resulting from the

energy, industry and transport sectors, as well as the unsanitary burning of waste [1-16]. However, soil pollution has not received sufficient attention from researchers.

Soil is a fundamental resource that supports life on Earth and is among the three most important components of our planet.

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Therefore, its quality is characterized by a high degree of spatiotemporal variability in biological, physical, and chemical properties, caused by natural or anthropogenic activities [17,18]. Soil is an important component of our ecosystem. It receives heavy metals from many different sources. It simultaneously acts as a buffer, controlling the movement of these elements to other compartments. Many pollutants can be retained in soil for extended periods [19]. Contamination of soils with heavy metals (HMs) is a major environmental issue due to the intensive use of agrochemicals. In greenhouse soil and the surrounding area, HM pollution is common. The addition of large amounts of fertilizers, biocides, and metal-enriched chemicals, along with amendments of organic matter, may also cause soil and environmental pollution in the greenhouse [20]. In undisturbed soils, heavy metals are inherited mainly from parent materials and tend to remain at stable levels [21,22]. Large quantities of chemical fertilizers, organic fertilizers, and pesticides are added to greenhouse soils than to arable fields to achieve higher yields. These additions result in the accumulation of heavy metals, salinity, and pesticide residues, and cause soil degradation through acidification [21, 23]. Phosphate fertilizers are an important source of heavy metals such as Zn, Cu, and Cd entering agricultural soils. These elements are mainly associated with NPK fertilizers; furthermore, applying high rates of nitrogen fertilizer to agricultural soils increases the accumulation of Cd and Pb in agricultural products [24–26]. Natural and anthropogenic inputs are the primary sources of heavy metals. In recent decades, the natural input of heavy metals into soils from paedogenic processes has been exceeded by human inputs; however, sewage sludge and fertilizers are also important sources of heavy metals [27,28]. Application of huge amounts of fertilizers and pesticides without any appropriate strategies has been adopted in greenhouse vegetable production systems to achieve high crop production, which has led to a series of consequences, in particular, the accumulation of heavy metals in the soil-vegetable system under greenhouse conditions has exposed serious health risk for human consumption [29, 30]. Content of Cd, Cu, Pb, and Zn in greenhouse vegetable soils has risen to twice the background values as a result of highly intensive cultivation and multiple cropping, which also lead to decline of soil quality, such as soil salinization, soil acidification, soil nutrient imbalance, besides accumulation of excessive heavy metals in soil [31–33]. Greenhouse soils are often highly contaminated with heavy metals, especially Cd, derived from pesticides and fertilizers applied in large quantities each year [19,34]. The long-term use of greenhouse agricultural practices can enhance the mobility of heavy metal residues if they are not controlled [35,36]. It has been reported that about 10 million hectares of agricultural soils in China have been contaminated with Cd [30,37]. One of the primary sources of soil quality deterioration and contamination of agricultural products is the accumulation of heavy metals in greenhouse soils and in vegetables [31]. Heavy metals such as Pb, Cu, Cr, Zn, Ni, Cd, and others occur at low concentrations in the soil environment to maintain an optimal ecological equilibrium; their concentrations depend primarily on the composition of the parent material [21]. Heavy metals are introduced into agricultural soils primarily through agrochemicals, biosolids, manures, and compost amendments, resulting in substantially increased soil concentrations of Cd, Cu, and Zn [38,39]. The intensive

greenhouse production system in the Jordan Valley, the Kingdom of Jordan, since 1998, has led to higher electrical conductivity in the top and subsoil layers compared to uncultivated soil, the buildup of salts, and a lower level of total and NO<sub>3</sub><sup>-</sup> nitrogen due to the application of excessive use of organic and mineral fertilizers. A significant increase in Chloride and a decrease in bacterial count were also observed compared to uncultivated soil. [40,41]. Production of vegetables in greenhouses in Turkey has been widespread since the 1970s (tomatoes, cucumbers, peppers, and eggplants), accounting for about 96% of total greenhouse production [42,43]. The high concentrations of heavy metals Cr, Cd, and Pb exceeded WHO standards, as found in greenhouse soil samples in Zilfi province in the Kingdom of Saudi Arabia [44].

The objectives of this study were to (I) evaluate the impact of long-term cultivation on the accumulation of eight heavy metals in soils from open farms and uncultivated areas within the Alshatti agricultural project, and (II) to examine changes in soil physicochemical properties.

## Materials and methods

### Geology and geography of the study area

Libyan soils are typically shallow, sandy, low in organic matter, and have limited water-holding capacity. Aridity is the dominant feature, and most soils are undeveloped or only partially developed. The southern desert region experiences hot summers and cool winters. Aeolian deposits significantly influence the formation of sandy soils in Libya [45]. The Alshatti district in southwestern Libya, between 27°28' N latitude and 12° to 16' E longitude (Fig. 1), is known for producing vegetables, cereals, and forages. Soil samples were collected from an agricultural project comprising 300 farms, irrigated by a sprinkler system powered by artesian wells. The study area is situated in an arid zone, with average temperatures ranging from 0 to 45°C. According to contemporary soil classification systems from Russia and the USDA, Aridisols and Entisols are the predominant soil orders in this region. These soils are characterized by a sandy texture and low organic matter content [46]. Soil samples were collected from open farms subjected to intensive cultivation and systematic chemical fertilization for over 40 years, as well as from an undisturbed, uncultivated area that served as a reference.



**Figure 1:** Map of Libya, elucidating the study area

### Collection and preparation of soil samples

A total of 45 soil samples were collected from the cultivated area, and 18 from undisturbed, uncultivated land nearby were considered reference soil. All samples were collected in triplicate from January to April 2025. The soil studied had been intensively cultivated with vegetables, irrigated with Artesian wells, and fertilized mainly with diammonium phosphate and Urea for over 40 years. Six soil subsamples of 1000g each were taken from each site at depths of 0-10, 10-

20, 20-30, 30-40, 40-50, and 50-60 cm using a stainless-steel soil auger. Samples were collected in clean polyethylene bags, labelled, and transferred to the laboratory for processing. At the laboratory, the soil samples were homogenized and air-dried to a constant weight at room temperature. They were then ground to pass through a 2mm sieve and stored in closed plastic containers [47]. Diammonium phosphate and Urea were also collected in triplicate from farmers, as these are the main fertilizers used, and analyzed to assess their heavy metal content [48,49]

### Chemical analysis

#### Physicochemical analysis

Physicochemical characteristics, including Soil pH, Electrical Conductivity (EC), Cation Exchange Capacity (CEC), Soil texture, and Organic matter (OM), were measured as described by [50]. The soil texture of the samples was determined using the method described by [51]. Briefly, in a 500 mL beaker, 50 mL of Calgon solution (10% sodium hexametaphosphate,  $\text{Na}_6[(\text{PO}_3)_6]$ ) was mixed with 40 g of an oven-dried, 2mm sieved soil sample. To this mixture, 200 mL of distilled water was added, and the mixture was soaked for 10 min. The solution was mixed thoroughly with an electric mixer for 5 min and equilibrated overnight under static conditions. Further, the mixture was filtered through a 50 $\mu\text{m}$  sieve to separate the sand (the separated sand was dried in an oven at 105 °C for 24 hrs). The filtrate was transferred to a 1-L sedimentation cylinder and diluted to 1000 mL with distilled water. The cylinder was vigorously shaken for 3 minutes and then placed on a flat surface. 44 mL of suspension from the top level was transferred to a weighed beaker and dried in the oven at 105° C; this portion represents silt and clay. The leftover solution in the cylinder was kept for an additional 4-6 hours, and another 50 mL from the top of the suspension was transferred to a weighed beaker and dried at 105 °C. This portion represents the percentage of clay. Soil texture was calculated as follows:

Soil texture was calculated as follows.

$$\% \text{Sand} = \text{weight of sand} \times \frac{100}{40} \quad (1)$$

$$\% \text{Silt Clay} = \text{weight of silt} \times \frac{1000}{50} + \frac{100}{40} \quad (2)$$

$$\% \text{ of Clay} = \text{weight clay} \times \frac{1000}{50} + \frac{100}{40} \quad (3)$$

#### Determination of HMs in samples of soils and chemical fertilizers.

Heavy metal concentrations in soil samples and chemical fertilizers were determined according to the protocol described in [52]. For soil analysis, 0.5 g of dried sample was digested with 15 mL of a mixture of  $\text{HNO}_3$ :  $\text{H}_2\text{SO}_4$ :  $\text{HClO}_4$  (5:1:1) at 80°C until a clear solution formed. The resulting solution was filtered through Whatman No. 42 filter paper into a 50 mL volumetric flask and diluted with deionized water. Heavy metal concentrations were measured using an atomic absorption spectrophotometer (Analytik Jena, NOVA-A400, Germany) equipped with an air-acetylene flame. The heavy metal content of chemical fertilizers, specifically diammonium phosphate (DAP) and urea (46%), was assessed using the protocol outlined in [48]. In this procedure, 5 g of

sample was placed in a 150 mL beaker and digested at 80°C with 40 mL of 5% (v/v)  $\text{HNO}_3$  on a hotplate. The digest was filtered through Whatman No. 42 filter paper into a 50 mL volumetric flask and diluted to volume with 5%  $\text{HNO}_3$ . Samples were subsequently analyzed using an atomic absorption spectrophotometer. All safety and quality assurance procedures were followed to ensure reliable results. To prevent cross-contamination, glassware was cleaned with a diluted  $\text{HNO}_3$  solution, and only analytical-grade reagents and deionized water were used. Working standard solutions for each element were prepared in triplicate for calibration and analysis. Reagent blanks corrected instrument readings. Analytical results were validated by analysing soil and fertilizer samples in triplicate, and fresh working standards were prepared before each analysis.

### 3.0. Assessment of soil pollution.

#### Assessment of contamination factor (Cf) and contamination degree (Cdeg)

The contamination factor (CF) for selected metals in soil samples from OF was calculated using equation (4) from [53]. CF represents the concentration of heavy metals in surface soil compared to the uncultivated soil reference value. CF is classified into four categories, as shown in Table 1.

$$CF = \frac{(C_m \text{ Sample})}{(B_m \text{ Background})} \quad (4)$$

Where:  $C_m$  sample = Concentration of HM in the soil sample.  $C_m$  Background = Concentration of the HM in the reference soil sample (uncultivated soil).

The contamination factors for all elements are summarized to represent the contamination degree (Cdeg), which is also divided into four categories [53], as shown in Table 2.

$$Cdeg. = (CF_1 + CF_2 + CF_3 + \dots CF_n) \quad (5)$$

Where, Cdeg = contamination degree, Cf = concentration factor

#### Pollution Load Index (PLI)

The Pollution Load Index (PLI) provides a straightforward method for assessing soil quality and estimating metal contamination. It enables comparison of total metal loads across soil depths and sampling sites. Each element's PLI is classified as low ( $PI \leq 1$ ), medium ( $1 < PI < 3$ ), or high ( $PI > 3$ ). PLI is calculated using Equation 6, as shown in [55].  $HPI = (CF_1 \times CF_2 \times CF_3 \times CF_4 \times \dots CF_n)^{1/n}$  (6). Where CF = Contamination factor and n = number of metals.

#### Geochemical Index (Igeo)

The geochemical index (Igeo) is widely used to estimate the potential risk of metal contamination derived from anthropogenic sources. The level of soil contamination with heavy metals was assessed using the geoaccumulation index (Igeo), as described by [56] in equation 7. In this study, the geoaccumulation index is used to assess heavy metal contamination in soil by comparing current levels in GH and OF soils with those in uncultivated soils (the reference). Igeo was calculated based on the following formula:

$$I_{geo} = \log_2 \left[ \frac{C_n}{(B_n \times 1.5)} \right] \quad (7)$$

Where:  $C_n$  represents the measured concentration of the element within the analyzed samples.

**Table 1:** Categories of contamination factor (CF) and Contamination degree

| Contamination factor | Level of contamination            | Contamination degree ( $C_{deg}$ ). | Contamination degree category        |
|----------------------|-----------------------------------|-------------------------------------|--------------------------------------|
| $CF < 1$             | low contamination factor          | $C_{deg} < 8$                       | Low degree of contamination          |
| $1 \leq CF < 3$      | moderate contamination factor     | $8 \leq C_{deg} < 16$               | Moderate degree of contamination     |
| $3 \leq CF \leq 6$   | considerable contamination factor | $16 \leq C_{deg} < 32$              | Considerable degree of contamination |
| $CF > 6$             | very high contamination factor    | $32 \geq C_{deg}$                   | Very high degree of contamination    |

B<sub>n</sub> denotes the background or pristine value of the element. The constant 1.5 is incorporated to account for lithological variations and to minimize the influence of potential background fluctuations.

This method evaluates the extent of metal pollution across seven enrichment classes, as presented in Table 2, in order of increasing geochemical index values.

**Table 2:** Index of Geoaccumulation ( $I_{geo}$ ) for contaminated levels in the soil [55]

| $I_{geo}$ class | $I_{geo}$ value   | Contamination level                       |
|-----------------|-------------------|-------------------------------------------|
| 0               | $I_{geo} \leq 0$  | Uncontaminated                            |
| 1               | $0 < I_{geo} < 1$ | Uncontaminated to moderately contaminated |
| 2               | $1 < I_{geo} < 2$ | Moderately contaminated                   |
| 3               | $2 < I_{geo} < 3$ | Moderately to strongly contaminated       |
| 4               | $3 < I_{geo} < 4$ | Strongly contaminated                     |
| 5               | $4 < I_{geo} < 5$ | Strongly to extremely contaminated        |

### Multivariate Statistical Analysis

#### Correlation analysis

Correlation analysis is a standard statistical method for evaluating relationships in paired data and is widely applied in trace-metal database analysis [19]. In this study, Pearson correlation significance was calculated among various physicochemical parameters and heavy metal concentrations in soil at the 0.95 and 0.01 confidence levels ( $P < 0.05$  and

$P < 0.01$ ).

#### Principal component analysis (PCA)

Principal component analysis (PCA) with varimax rotation was conducted in SPSS 26 to identify accumulation patterns and support data interpretation [57]. In the open-farm soils, PCA revealed that heavy metal accumulation patterns often stem from common sources. Assigning factor loadings to each heavy metal helps determine its likely source of accumulation and highlights potential relationships among metals and accumulation patterns.

### Results and Discussion

#### Soil properties in soil depths

Table 3 summarizes the physicochemical properties measured at different soil depths in open-farm samples. Mean pH values increased with depth in open-farm soils but decreased in uncultivated soils. These results are consistent with [58], which found lower pH in open-farm soils compared to uncultivated soils. The decrease in pH in open-farm soils is linked to fertilizer use, which promotes nitrification and acidification. Ongoing fertilizer application also increases  $H^+$  release by roots [40,59,60]. Organic matter and electrical conductivity, which indicates soluble salt concentration, both declined with depth in all soils. Cation exchange capacity (CEC), reflecting the soil's ability to retain exchangeable cations, decreased with depth in open-farm soils. In uncultivated soils, CEC decreased to 20-30 cm, then increased

**Table 3:** Physicochemical properties of Open-Farm and uncultivated soil samples at different depths.

| Table 1. Physicochemical properties of Open Farm and Uncultivated soil samples at different depths. |         |                 |           |            |           |           |           |              |       |       |           |
|-----------------------------------------------------------------------------------------------------|---------|-----------------|-----------|------------|-----------|-----------|-----------|--------------|-------|-------|-----------|
| Parameter                                                                                           |         | Soil depth (cm) |           |            |           |           |           | Soil texture |       |       | Soil type |
|                                                                                                     |         | 0-10            | 10-20     | 20-30      | 30-40     | 40-50     | 50-60     | Sand%        | Silt% | Clay% |           |
| pH                                                                                                  | OF      | 6.93±0.12       | 6.78±0.13 | 6.78±0.09  | 6.95±0.08 | 7.14±0.17 | 7.24±0.07 | 98.34        | 0.86  | 0.96  | Sandy     |
|                                                                                                     | Range   | 6.72-7.23       | 6.52-7.12 | 6.56-6.99  | 6.76-7.14 | 6.81-7.60 | 7.11-7.42 |              |       |       |           |
|                                                                                                     | Unculti | 8.22            | 8.20      | 8.22       | 8.20      | 8.00      | 8.01      |              |       |       |           |
| OM (%)                                                                                              | OF      | 0.75±0.04       | 0.73±0.02 | 0.49±0.01  | 0.37±0.03 | 0.31±0.04 | 0.30±0.07 |              |       |       |           |
|                                                                                                     | Range   | 0.65-0.85       | 0.70-0.77 | 0.47-0.50  | 0.30-0.43 | 0.23-0.40 | 0.20-0.50 |              |       |       |           |
|                                                                                                     | Unculti | 1.80            | 0.80      | 0.80       | 0.80      | 0.80      | 0.60      |              |       |       |           |
| EC (µS/cm)                                                                                          | OF      | 0.21±0.03       | 0.20±0.03 | 0.19±0.03  | 0.16±0.03 | 0.17±0.02 | 0.18±0.03 |              |       |       |           |
|                                                                                                     | Range   | 0.16-0.31       | 0.14-0.28 | 0.14- 0.27 | 0.12-0.24 | 0.13-0.23 | 0.14-0.26 |              |       |       |           |
|                                                                                                     | Unculti | 17.97           | 0.83      | 0.83       | 0.83      | 1.81      | 2.04      |              |       |       |           |
| CEC (Cmol/kg)                                                                                       | OF      | 4.22±0.30       | 4.14±0.42 | 3.54±0.36  | 3.09±0.29 | 2.68±0.28 | 2.80±0.48 |              |       |       |           |
|                                                                                                     | Range   | 3.60-5.03       | 3.50-5.38 | 3.00-4.60  | 2.53-3.77 | 2.10-3.17 | 1.83-3.97 |              |       |       |           |
|                                                                                                     | Unculti | 5.80            | 2.70      | 3.60       | 6.30      | 6.80      | 5.80      |              |       |       |           |

OF= Open Farm; Unculti. = Uncultivated. SD= Standard Deviation

#### Concentrations of Heavy metals in soil samples at different depths of GH, OF and uncultivated soils

Table 5 shows the mean concentrations of heavy metals (Cr, Cu, Cd, Mn, Zn, Ni, Fe, and Pb) in OF and uncultivated soils at depths from 0 to 60 cm. In OF, concentrations range from 25.94±5.59 to 26.62±5.91 mg kg<sup>-1</sup>. In uncultivated soils, mean Cr concentration decreases from 29.43 mg kg<sup>-1</sup> at 0-10 cm to 25.56 mg kg<sup>-1</sup> at 50-60 cm. Cr concentrations do not vary significantly with depth at any site. These results align with those reported by [61].

Table 4 shows that in open-farm soil samples, Cu concentrations ranged from 0.67±0.30 to 1.11±0.60 mg/kg, with the highest levels at a depth of 50–60 cm. The mean concentration of Cd ranged from 1.03±0.83 to 5.35±3.41 mg/kg at the respective soil depths. Cu and Cd were not detected in uncultivated soils, likely because they are absent from chemical fertilizers. Chemical fertilizers such as diammonium phosphate and NPK are known to contain trace-element impurities; significant amounts of these impurities have been found in phosphate fertilizers used in Jordan [62-

64]. Manganese concentrations in the soil samples were low, which aligns with previous findings [65]. In OF soils, the mean Mn concentration was highest at 0-10 cm (23.75±3.25 mg kg<sup>-1</sup>) and lowest at 50-60 cm (6.47±4.42 mg kg<sup>-1</sup>). Uncultivated soils exhibited a similar trend, with a maximum mean of 17.90 mg kg<sup>-1</sup> in the 0-10 cm layer and a minimum of 12.88 mg kg<sup>-1</sup> in the 50-60 cm layer. Overall, OF soils exhibited higher mean Mn concentrations compared to uncultivated soils. Table 4 indicates that the mean Zn concentration in Open-Farm soils ranged from 0.72 ± 0.49 to 4.32 ± 3.16 mg kg<sup>-1</sup>, whereas Zn was not detected in uncultivated soils. These results are consistent with previous studies. The presence of Zn in OF soils is attributed to its inclusion in diammonium phosphate fertilizer. Zn concentrations in all samples remained below permissible limits. Additionally, Zn levels decreased with increasing soil depth, consistent with findings reported in [61]. Ni concentrations in OF soil samples ranged from 10.13 ± 4.85 to 11.05 ± 4.52 mg.kg<sup>-1</sup>, while uncultivated soils ranged from 6.35 to 8.05 mg.kg<sup>-1</sup>. Ni levels were consistent



**Table 4:** Concentration of heavy metals (mg.kg-1) in different soil depths (cm) of Open Farm and uncultivated

|    |          | Depths (cm)   |               |               |                |               |                | Pl*     |
|----|----------|---------------|---------------|---------------|----------------|---------------|----------------|---------|
|    |          | 0-10          | 10-20         | 20-30         | 30-40          | 40-50         | 50-60          |         |
| Cr | OF       | 25.94 ± 5.59  | 25.49 ± 5.43  | 25.69 ± 5.53  | 26.62 ± 5.91   | 26.02 ± 5.29  | 26.05 ± 5.36   | 70-100  |
|    | Range    | 13.22-38.35   | 12.89-37.02   | 13.11-37.20   | 14.15 - 40.62  | 14.92 - 37.52 | 14.23 - 37.57  |         |
|    | Unculti. | 29.43         | 26.82         | 28.27         | 27.58          | 26.87         | 25.56          |         |
| Cu | OF       | 0.95 ± 0.38   | 0.67 ± 0.30   | 0.8 ± 0.54    | 1.08 ± 0.65    | 1.01 ± 0.41   | 1.11 ± 0.60    | 20-30   |
|    | Range    | ND -1.87      | ND -1.40      | ND - 2.43     | 0.15 - 3.00    | 0.11 - 2.07   | ND - 2.83      |         |
|    | Unculti. | ND            | ND            | ND            | ND             | ND            | ND             |         |
| Cd | OF       | 3.91 ± 2.24   | 3.22 ± 2.11   | 3.88 ± 2.84   | 1.03 ± 0.83    | 2.30 ± 0.83   | 5.35 ± 3.41    | 0.2-1.0 |
|    | Range    | ND - 9.33     | ND - 9.17     | ND - 12.02    | ND - 3.47      | ND - 3.53     | ND - 15.00     |         |
|    | Unculti. | ND            | ND            | ND            | ND             | ND            | ND             |         |
| Mn | OF       | 23.75 ± 3.25  | 16.67 ± 4.50  | 10.32 ± 4.22  | 9.4 ± 4.48     | 6.83 ± 4.43   | 6.47 ± 4.42    | -       |
|    | Range    | 17.83 - 29.81 | 6.90 - 28.14  | 3.2 - 20.95   | 2.52 - 21.39   | ND - 18.59    | ND -18.81      |         |
|    | Unculti. | 17.90         | 15.50         | 11.20         | 10.66          | 12.94         | 12.88          |         |
| Zn | OF       | 4.32 ± 3.16   | 2.46 ± 1.37   | 0.72 ± 0.49   | 2.05 ± 1.31    | 2.29 ± 1.99   | 1.42 ± 1.06    | 50      |
|    | Range    | ND - 13.58    | ND - 5.89     | ND - 2.08     | ND - 5.67      | ND - 8.23     | ND - 4.55      |         |
|    | Unculti. | ND            | ND            | ND            | ND             | ND            | ND             |         |
| Ni | GH       | 9.21 ± 1.80   | 8.84 ± 1.63   | 8.32 ± 1.59   | 8.79 ± 2.14    | 9.12 ± 2.37   | 8.96 ± 2.38    | 50      |
|    | Range    | 6.74 - 14.44  | 6.27 - 13.46  | 5.92 - 12.93  | 5.70 - 15.01   | 5.72 - 16.07  | 5.67-16.02     |         |
|    | OF       | 10.59 ± 4.59  | 11.05 ± 4.52  | 10.36 ± 4.94  | 10.81 ± 5.12   | 10.47 ± 5.04  | 10.13 ± 4.85   |         |
| Fe | Range    | 2.81 - 23.58  | 4.31 - 24.06  | 4.68 - 25.15  | 3.83 - 25.94   | 3.33 - 25.40  | 3.71 - 24.59   | 50,000  |
|    | Unculti. | 7.15          | 8.05          | 6.70          | 6.84           | 6.35          | 6.45           |         |
|    | GH       | 725.39 ± 31.2 | 694.39 ± 50.4 | 561.04 ± 35.2 | 486.02 ± 41.52 | 464.17 ± 46.0 | 478.96 ± 46.10 |         |
|    | Range    | 640.83-788.3  | 556.30-779.8  | 488.10-656.2  | 423.43-604.0   | 411.50-601.3  | 396.09-606.7   |         |
|    | OF       | 373.86 ± 72.1 | 330.20 ± 33.3 | 273.63 ± 41.0 | 223.71 ± 35.4  | 205.97 ± 37.6 | 197.64 ± 62.51 |         |
|    | Range    | 173.24-512.2  | 281.04-428    | 176.84-373.5  | 118.40- 270.7  | 104.20 - 268  | 26.12-325.75   |         |
|    | Unculti. | 886.00        | 758.00        | 635.00        | 516.00         | 520.00        | 592.00         |         |

OF= Open Farm (n=96); Unculti. = Uncultivated (n=18); SD= Standard Deviation. Pl\* = permissible limit of metals content in agriculture soil [56].

across most depths. As shown in Table 5, the mean Ni concentration remained below permissible limits. Our results align with those reported by [47,61,66]. Iron was the most abundant essential metal measured in the soil samples, with concentrations higher than those of other heavy metals. In OF and uncultivated soils, iron levels ranged from 197.64 ± 62.51 to 373.86 ± 72.06 mg.kg-1 and from 516.00 to 886.00 mg.kg-1, respectively. Several studies have also reported high iron content, including 15,274 mg.kg-1 in the Segura River valley, Alicante, Spain [66], 16,916 mg.kg-1 in the European Mediterranean region, and 5692 mg.kg-1 in agricultural soils of Amritsar, Punjab, India [67]. High iron concentrations may result from long-term irrigation with wastewater and continuous use of inorganic fertilizers in agricultural fields [68]. Pb is a highly hazardous environmental pollutant. Its concentration in agricultural soils varies with parent rock types, soil-forming processes, and human activities in the area [68]. Table 5 shows that Pb was not detected in the soil samples, likely due to the lack of nearby contamination sources such as factories or heavy traffic. Previous studies have reported unsystematic variation in soil physicochemical properties and heavy metal content at different depths [69].

#### Determination of HMS in chemical fertilizer samples

To determine the source of soil contamination, heavy metals (HMs) were also analyzed in the chemical fertilizers used to increase crop yields. Table 5 indicates that the measured heavy metal concentrations were significantly lower than the Canadian Standards limits [70,71].

#### Assessment of soil contamination

Contamination factor (CF), Contamination Degree (Cdeg), Heavy Metal Pollution Index (HPI), and Geochemical Index (Igeo) are the most widely used parameters for quantifying soil pollution; therefore, these indices were used to assess the contamination of Open Farm soils with the studied heavy metals.

**Table 5:** Heavy metals concentration in DAP and Urea (mg.kg<sup>-1</sup>)

| Element | Sample | Min.  | Max.  | Mean ± S.E     | PL*  |
|---------|--------|-------|-------|----------------|------|
| Cr      | DAP    | 3.30  | 3.87  | 3.493 ± 0.188  | -    |
|         | Urea   | 0.13  | 0.32  | 0.237 ± 0.057  | -    |
| Cu      | DAP    | 0.74  | 1.06  | 0.873 ± 0.096  | -    |
|         | Urea   | ND    | ND    | ND             | -    |
| Zn      | DAP    | 30.94 | 37.84 | 33.860 ± 2.061 | 1850 |
|         | Urea   | 0.23  | 0.41  | 0.335 ± 0.055  | -    |
| Cd      | DAP    | 1.16  | 1.23  | 1.194 ± 0.021  | 20   |
|         | Urea   | ND    | ND    | ND             | -    |
| Ni      | DAP    | 2.57  | 2.73  | 2.643 ± 0.047  | 180  |
|         | Urea   | 0.23  | 0.31  | 0.272 ± 0.024  | -    |
| Fe      | DAP    | 4.98  | 7.68  | 6.490 ± 0.796  | -    |
|         | Urea   | 1.96  | 2.00  | 1.983 ± 0.012  | -    |
| Mn      | DAP    | ND    | ND    | ND             | -    |
|         | Urea   | ND    | ND    | ND             | -    |
| Pb      | DAP    | ND    | ND    | ND             | 500  |
|         | Urea   | ND    | ND    | ND             | -    |

\* Canadian standards permissible limit; (-) = not mentioned; (ND) = Not Detected, n=9 for each fertilizer

#### Contamination factor (CF)

Table 6 presents the calculated contamination factors for various heavy metals in Open Farm soils at different depths. According to the CF categories in Table 1, no soil samples show contamination with Cu, Cd, Zn, or Pb at any depth. Both soils exhibit low CF (CF < 1) for Cr and Fe at all depths, and moderate CF (1 ≤ CF < 3) for Mn (0.502 - 3.435) and Ni (1.098 - 1.649). These results align with those reported by [72].

#### Contamination degree (Cdeg)

The Cdeg of all studied heavy metals at soil depths in Open Farm soils is presented in Table 6. The results indicate that the Cdeg of Open Farm soils ranged from 0.784 to 3.86. The degree of contamination for the studied heavy metals was still within the low contamination category, with Cdeg ranging from 3.426 to 6.501. These results also reflected contamination levels ranging from none to moderate at all depths. High Cdeg was reported in the mining area of the

Slovak Republic in 1997, whereas after the end of mining activities, Cdeg decreased by about 41% in 2015 [73].

### Pollution Load Index

The Pollution Load Index (PLI), calculated as the sum of CF values, enables effective comparison of pollution levels across locations. Table 6 shows PLI results for the seven metals. The mean PLI for the open farm was 0.848, with a range of 0.720 to 0.976, indicating a low-pollution site. PLI values decreased with increasing soil depth. Because the soil

surface is the primary point of contact for all activities, higher metal concentrations are expected there. Surface soils are therefore better indicators of metallic burdens, as metal levels decrease with depth at both locations [74]. The extensive use of fertilizers, pesticides, and sewage irrigation, along with farming practices, can lead to the accumulation of heavy metals in agricultural soil. These findings are consistent with those reported by [75,76].

**Table 6:** Concentration factor, contamination degree, and pollution load index for open farm soil samples

| Depth(cm) | CF    |    |    |       |    |       |       | C deg. | PLI   |
|-----------|-------|----|----|-------|----|-------|-------|--------|-------|
|           | Cr    | Cu | Cd | Mn    | Zn | Ni    | Fe    |        |       |
| 0-10      | 0.881 | ND | ND | 1.327 | ND | 1.481 | 0.422 | 0.784  | 0.976 |
| 10-20     | 0.850 | ND | ND | 1.075 | ND | 1.373 | 0.436 | 3.734  | 0.889 |
| 20-30     | 0.909 | ND | ND | 0.921 | ND | 1.546 | 0.431 | 3.807  | 0.864 |
| 30-40     | 0.965 | ND | ND | 0.882 | ND | 1.580 | 0.434 | 3.861  | 0.874 |
| 40-50     | 0.968 | ND | ND | 0.528 | ND | 1.649 | 0.396 | 3.541  | 0.760 |
| 50-60     | 1.019 | ND | ND | 0.502 | ND | 1.571 | 0.334 | 3.426  | 0.720 |

ND = Not Detected, HM = Heavy Metal, CF = Contamination factor C<sub>deg.</sub> = contamination degree

### Geo-chemical Index

Table 7 presents Igeo values and contamination levels for various metals in Open Farm soil samples. The analysis shows that these soils are either uncontaminated or moderately contaminated with Ni (Igeo = 0.017), with no contamination detected for the other measured heavy metals. These findings are consistent with previous studies [49,75,77-79].

**Table 7:** Geoaccumulation index (Igeo) values of Open Farm soils

| HMs | Igeo Value | Contamination Level                      |
|-----|------------|------------------------------------------|
| Cr  | -0.664     | Uncontaminated                           |
| Cu  | 0.0        | Uncontaminated                           |
| Cd  | 0.0        | Uncontaminated                           |
| Mn  | -0.726     | Uncontaminated                           |
| Zn  | 0.0        | Uncontaminated                           |
| Ni  | 0.017      | Uncontaminated / moderately contaminated |
| Fe  | -1.868     | Uncontaminated                           |

### Statistical analysis

#### Correlation analysis

Table 8 presents Pearson's correlation coefficients for heavy metals in soil samples, summarizing the relationships between soil physicochemical properties and heavy metal concentrations. Correlations at the 0.05 and 0.01 significance levels ( $p < 0.01$  and  $p < 0.05$ ) were determined using ANOVA. In Open Farm soils, strong positive correlations at the 0.01 level were identified between CEC-OM ( $r=0.674$ ), Cu-EC ( $r=0.790$ ), Cd-EC ( $r=0.623$ ), Mn-OM ( $r=0.601$ ), Mn-CEC ( $r=0.677$ ), Fe-OM ( $r=0.573$ ), Zn-EC ( $r=0.797$ ), Zn-Cu ( $r=0.620$ ), Ni-Cr ( $r=0.672$ ), and Fe-OM ( $r=0.573$ ). At the 0.05 level, positive correlations were observed between Fe-Cr

( $r=0.436$ ) and Fe-Ni ( $r=0.484$ ). Strong negative correlations at the 0.01 level were observed between Cr-EC ( $r=-0.528$ ), Mn-Cu ( $r=-0.538$ ), and Fe-pH ( $r=-0.696$ ). Additional negative correlations were found between OM-pH ( $r=-0.419$ ), Cr-pH ( $r=-0.437$ ), Cu-Cr ( $r=-0.424$ ), Zn-Cr ( $r=-0.431$ ), and Ni-Mn ( $r=-0.442$ ). The significant positive correlations among heavy metals suggest a common or identical origin [18]. Previous studies have demonstrated that Zn, Cd, and Pb are common anthropogenic elements in urban environments, and their significant correlations indicate a likely shared source [79]. These results are consistent with earlier research reporting both positive and negative correlations between soil properties and heavy metals [75].

#### Principal Component Analysis/Factor Analysis

Several authors have used principal component analysis (PCA) to identify the sources of heavy metals in agricultural soils [49,75,78]. In this study, PCA was applied to identify factors influencing metal concentrations and to apportion sources in Open Farm soils. The results, shown in Tables 9a and 9b, indicate that three principal components account for 85.29% of the total variance. The first component explains 38.24% of the variance, the second 30.64%, and the third 16.42%. Based on matrix rotation, heavy metals were grouped using loading coefficients greater than  $\pm 0.5$ . The first component (PC1) links Zn, Cu, and Cd, which are positively correlated [79]. Long-term use of agrochemicals and pesticides is reported to increase Zn and Cu concentrations as farms in the study area use artesian well water and lack significant traffic or industrial activity,

**Table 8:** Correlation matrix between soil properties and heavy metals in OF soils

|     | pH      | OM     | EC      | CEC    | Cr     | Cu      | Cd     | Mn     | Zn   | Ni    | Fe |
|-----|---------|--------|---------|--------|--------|---------|--------|--------|------|-------|----|
| pH  | 1       |        |         |        |        |         |        |        |      |       |    |
| OM  | -.419*  | 1      |         |        |        |         |        |        |      |       |    |
| EC  | -.161-  | .232   | 1       |        |        |         |        |        |      |       |    |
| CEC | -.374-  | .674** | -.093-  | 1      |        |         |        |        |      |       |    |
| Cr  | -.437*  | -.066- | -.528** | .101   | 1      |         |        |        |      |       |    |
| Cu  | -.136-  | -.094- | .790**  | -.399- | -.424* | 1       |        |        |      |       |    |
| Cd  | -.175-  | -.021- | .623**  | -.368- | -.063- | .621**  | 1      |        |      |       |    |
| Mn  | .158    | .601** | -.179-  | .677** | -.193- | -.538** | -.390- | 1      |      |       |    |
| Zn  | -.028-  | .177   | .797**  | -.172- | -.431* | .620**  | .368   | -.040- | 1    |       |    |
| Ni  | -.384-  | -.123- | .033    | -.325- | .672** | .024    | .383   | -.442* | .121 | 1     |    |
| Fe  | -.696** | .573** | .342    | .301   | .436*  | .203    | .348   | .027   | .316 | .484* | 1  |

\*. Correlation is significant at the 0.05 level (2-tailed). \*\*. Correlation is significant at the 0.01 level (2-tailed);

Chemical fertilizers and pesticides are the primary sources of Cu, Zn, and Cd. The second component (PC2), explaining 30.64% of the variance, shows a strong positive correlation among Ni, Cr, and Fe, suggesting geogenic and paedogenic

origins for Ni and Cr. The third component (PC3), accounting for 16.42% of the variance, includes only Mn and has no clear source identified. These findings are consistent with previous studies [47,61,65,77,80].

**Table 9:** Total variance for PCA and FA of heavy metals soil samples of open farm

| Component                                              | Initial Eigenvalues |               |              | Extraction Sums of Squared Loadings |               |              | Rotation Sums of Squared Loadings        |                              |              |
|--------------------------------------------------------|---------------------|---------------|--------------|-------------------------------------|---------------|--------------|------------------------------------------|------------------------------|--------------|
|                                                        | Total               | % of Variance | Cumulative % | Total                               | % of Variance | Cumulative % | Total                                    | % of Variance                | Cumulative % |
| Cr                                                     | 2.677               | 38.236        | 38.236       | 2.677                               | 38.236        | 38.236       | 2.277                                    | 32.535                       | 32.535       |
| Cu                                                     | 2.144               | 30.635        | 68.871       | 2.144                               | 30.635        | 68.871       | 2.134                                    | 30.481                       | 63.016       |
| Cd                                                     | 1.149               | 16.417        | 85.288       | 1.149                               | 16.417        | 85.288       | 1.559                                    | 22.272                       | 85.288       |
| Mn                                                     | 0.500               | 7.143         | 92.431       |                                     |               |              |                                          |                              |              |
| Zn                                                     | 0.401               | 5.729         | 98.160       |                                     |               |              |                                          |                              |              |
| Ni                                                     | 0.076               | 1.084         | 99.245       |                                     |               |              |                                          |                              |              |
| Fe                                                     | 0.053               | 0.755         | 100.000      |                                     |               |              |                                          |                              |              |
| <b>Extraction Method: Principal Component Analysis</b> |                     |               |              |                                     |               |              | <b>Rotation Sums of Squared Loadings</b> |                              |              |
| Eigen value                                            |                     |               |              |                                     |               |              | <b>Elements</b>                          | <b>Component (open farm)</b> |              |
| % Total variance                                       |                     |               |              |                                     |               |              |                                          | <b>PC1</b>                   | <b>PC2</b>   |
| Cumulative. %                                          |                     |               |              |                                     |               |              |                                          |                              | <b>PC3</b>   |
| Extraction Method: Principal Component Analysis.       |                     |               |              |                                     |               |              | <b>Zn</b>                                | .890                         | .007         |
| Rotation Method: Varimax with Kaiser Normalization.    |                     |               |              |                                     |               |              | <b>Cu</b>                                | .804                         | -.145        |
| a. Rotation converged in 5 iterations.                 |                     |               |              |                                     |               |              | <b>Cd</b>                                | .631                         | .264         |
|                                                        |                     |               |              |                                     |               |              | <b>Ni</b>                                | .043                         | .836         |
|                                                        |                     |               |              |                                     |               |              | <b>Cr</b>                                | -.491                        | .834         |
|                                                        |                     |               |              |                                     |               |              | <b>Fe</b>                                | .436                         | .795         |
|                                                        |                     |               |              |                                     |               |              | <b>Mn</b>                                | -.085                        | -.128        |

## Conclusion

This study evaluated soil properties and heavy metal concentrations in open-farm soils after more than 40 years of cultivation in Alshatti province, Libya, using uncultivated soils as a reference. Open-farm soils were slightly more acidic and had lower electrical conductivity than uncultivated soils. Long-term use of chemical fertilizers containing heavy metal impurities has resulted in higher concentrations of Cu, Cd, Zn, and Mn in open-farm soils. We recommend adopting a rational fertilization program and reducing chemical fertilizer use to maintain safe heavy metal levels in these soils. Overall, heavy metal concentrations in all samples remained within the permissible limits set by [69]. There was a positive and significant correlation between organic matter and EC, CEC, Mn, Zn, and Fe. Correlation and principal component analyses of seven heavy metals identified three principal components that explain variability in open-farm soils under vegetable cultivation. PC1 accounted for 32.535% of the variance and was associated with Zn, Cu, and Cd, indicating these elements originate from agricultural activities, including the use of pesticides and fertilizers with metal impurities. PC2 accounted for 30.481% of the variance and was associated with Ni, Cr, and Fe, while PC3 accounted for 20.272% and was associated with Mn, likely reflecting the influence of parent rock material. Although open-farm soils were not contaminated based on the parameters studied, they contained higher heavy metal concentrations than uncultivated soils, suggesting gradual accumulation. Ongoing monitoring and careful management of agricultural practices are necessary to prevent future soil pollution in the area.

**Author contributions:** Mansour: Supervision; Mansour and Samera: Conceptualization; Samera, Aisha and Masouda: Investigation, formal analysis, validation, writing-original draft preparation; Mansour and Yasser: Writing-review and editing.

## Data availability statement

The datasets generated during the current study are not publicly available as the study is ongoing; however, they are available from the corresponding author upon reasonable request.

## Declaration of conflict of interest

The authors confirm that they have no conflicts of interest to declare.

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