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Evaluation of heavy metal content in agricultural soil fertilized with chemical fertilizers in the Tasawa Agricultural Project, Libya

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ABSTRACT

The Tasawa agricultural project was constructed to produce grains in the southern region. The project has employed various chemical fertilizers to enhance agricultural production. However, the use of these fertilizers may lead to the accumulation of heavy metals in the soil. Soil samples taken from the project site showed a mean concentration of Chromium (Cr), Copper (Cu), Cadmium (Cd), Manganese (Mn), Zinc (Zn), Nickel (Ni), and Iron (Fe) at 25.97±11.02, 0.95±0.94, 4.37±3.23, 12.26±7.59, 2.21±2.98, 9.15±10.93, and 267.50±84.96 mg/kg, respectively. The geochemical pollution index (Igeo) shows the non-pollution of Cu, Cd and Zn, while Cr, Mn, Ni, and Fe are unpolluted or slightly polluted. Pearson correlation coefficient indicated a significant positive correlation between Fe-Mn (r=.987**), a significant negative correlation between Mn-Cr (r = -.943**) and a negative correlation between Fe-Cr (r= -.878*).

INTRODUCTION

The Libyan state seeks to achieve food, water and energy security for all its citizens and to distribute job opportunities and economic prosperity fairly among all regions (Fathi, et al., 2007, Elmnifi, et al., 2023, Elnaggar, et al., 2023). This requires the attention of scientists and specialists in all aspects of life. We find there many general studies on the transition towards renewable energies, decarbonization (Fathi, et al., 2017, 2018), and decentralization of the energy and water system (Nassar, et al., 2006, 2007, 2016-a, 2016-b, 2021, 2023, 2024-a, 2024-b).The same applies to water, as studies focused on making the most of waste water for sewage (Alsharif, et al., 2023, Miskeen, et al., 2023, Yasser et al., 2023),and rainwater, adopting several policies to recycle it, constructing dams in valleys, using treated water to irrigate trees, forests, and farms, reducing the waste of groundwater, and rationalizing its consumption in a way that ensures its sustainability (Awad, et al., 2023).To achieve sustainability in agriculture, we need extensive studies that include the entire plant life cycle, the type of plant, the type of soil and fertilizers, and the characteristics of irrigation water in terms of salinity (Kaes, 2024).

Heavy metals are natural components of the Earth's crust; some are biologically important, exist in deficient concentrations, and play an essential role in human health (Juvanovic. et al., 1995). The two primary sources of heavy metals in soil are the background natural chemistry of the Earth and pollution resulting from various human activities, which can occur through manure, sewage sludge, deposition of atmospheric particles, and fertilization with animal waste (Abdolhossien et al., 2012). Soil is the main sink for metals released into the environment from various anthropogenic sources (Niragu, 1991). The accumulation of heavy metals in agricultural soil is a growing concern due to its relationship to food safety or its arrival through leaching into groundwater and surface water represented by nearby rivers and water bodies, which may lead to potential health risks. Hazardous heavy metals may also come from the rocks that make up the soil or anthropogenic sources such as solid or liquid sediments, agricultural activities, or industrial and urban emissions (Wilson and Pyatt, 2007). Soil pollution with heavy metals results from various human activities such as various means of transportation, agricultural practices, industrial activities, and waste disposal methods. The total concentration of heavy metals in the soil may indicate the total metals present. However, it does not limit us to any information about their chemical nature or bioavailability (Jin. et al., 2005). The composition of the soil solution reflects the density and distribution of trace elements in the aqueous phase of the soil. It represents integrating multiple chemical, physical and biological processes within the soil. These heavy elements exist in the soil in various chemical forms, such as water-soluble and exchangeable, but bound to specific sites of organic and inorganic components and in the structure of primary and secondary minerals (Lake et al., 1984). In agricultural soils, the concentration of heavy metals increases as the soil develops. During the soil formation process, their movement can be altered by environmental conditions such as land use, agricultural inputs, climate change or saturation beyond the soil's buffering capacity. Heavy metals exist in different chemical forms in soil, and these forms affect their reactive mobility and bioavailability (Zhang and Gong, 1996). Agricultural soil is polluted with heavy metals by adding chemical fertilizers, pesticides, and untreated or partially treated wastewater, which are considered among the most important factors of soil pollution worldwide. The transport and fate of heavy metals in soil depend mainly on their chemical form and their isolation from minerals. They are absorbed into the soil by an initial rapid reaction in minutes or hours, followed by slow adsorption reactions that take days or even years. They are redistributed in different chemical forms through their varying bioavailability, transport and toxicity (Smith, 1997). The ingress of toxic heavy metals into the soil is increasing due to the reuse of agricultural chemicals, municipal wastes, sewage sludge disposal, and wastewater used for irrigation. Its concentration in soil depends on the geochemical properties of the soil. In contrast, soil quality mainly depends on the degree of temporal and spatial variation of its physical, chemical and biological properties caused by natural and anthropogenic interactions (Schindelbeck et al., 2008; Maltas et al., 2013). Heavy elements are absorbed and accumulated in different plant species and varieties. They can accumulate in some plants in large quantities without showing symptoms of toxicity, while lead enters the plant tissues very quickly and accumulates in the green organs (Lehoczky et al., 2002). Heavy metals can accumulate in the soil of open vegetable fields and greenhouses due to excessive use of chemical and organic fertilizers for a long time. Using animal manure, pesticides, irrigation with wastewater, petrochemical spills, and atmospheric sedimentation also contribute to soil pollution (Fytianos et al., 2001; Huang and Jin, 2008). It is believed that the activities of microbes and enzymes are appropriate indicators of soil quality and can be affected by several factors, such as irrigation and fertilization. In most soil environments, sorption is the dominant diversification process. Therefore, the bulk of the heavy elements in soil are associated with the solid phase of that soil. Thus, environmental pollution arises when These elements are mobilized in the soil solution, absorbed by plants and transferred to the surface or groundwater (Ben Achiba et al., 2009). Soil pollution with heavy metals such as Cr, Cd, Ni, Cu, Zn, and Fe is one of the most severe environmental problems worldwide. The continuous elimination of the accumulation of hazardous materials in the soil, the disposal of municipal and industrial waste, natural agricultural practices, and the use of agricultural fertilizers and pesticides have contributed to the accumulation of many heavy elements in the soil (Huda et al., 2024). Inorganic fertilizers are commonly used, including high impurities such as phosphate, nitrates, and potassium salts. The distribution of heavy metals in soil is controlled by several interactions, including mineral precipitation and dissolution, ion exchange absorption and adsorption, hydrological cross-linking and complexation, biological movement and immobilization, and plant uptake (Kumperman and Crreiro 1997; Nassar et al., 2023). The long-term use of fertilizers and pesticides for agricultural purposes has contributed to the continued accumulation of high amounts of heavy elements in the soil, in addition to phosphate fertilizers and superphosphate, which are one of the primary sources of heavy metal inputs in agricultural activities as they contain many impurities such as Pb, Hg, Cd (Cyedele et al., 2006). The concentration of heavy metals in the soil solution plays a vital role in controlling the bioavailability of metals in plants. Irrigating wastewater contaminated with heavy metals for an extended period also increases its content in the soil, which leads to increased absorption by plants. This process depends on the type of soil, the types of plants, and their stages of growth (Nazir et al., 2015). The use of phosphate fertilizers in agricultural soil also increased the levels of some elements such as Cd, Cr, and Pb. The addition of phosphate fertilizers significantly reduced the pH of the soil, which led to the absorption of minerals from the soil tissue. The most important sources of lead contamination of agricultural soil are sludge, organic fertilizer, and sediments from the atmosphere (Nicholson et al., 2003; Ahmed et al., 2023). Organic matter also plays an influential role in controlling the movement of elements in the soil, as it may reduce the concentrations available in the soil through sedimentation, adsorption and cross-linking processes (Bernal et al., 2007). Fertilizing agricultural soil with nitrogen fertilizers reduced its pH and changed its other properties (Brady and Weil, 2002). Phosphate fertilizers can contain some heavy metals as impurities, such as Cd, Pb, and As, and the use of traditional inorganic phosphate fertilizers has led to contamination of agricultural soils with P, Cd, Cr, Cu, Zn, Ni, Pb in Argentina (Giuffrede Lopez Carnle et al., 1997). In England and Wales, phosphate fertilizers were an essential source of heavy metals, especially Zn, Cu, and Cd, which entered agricultural soil. The prolonged addition of excessive amounts of phosphate and other chemical fertilizers that contain relatively high contents of heavy metals caused the unintended addition of metals to Soil (Nicholson et al., 2003). Organic fertilizer may also contain many heavy metals, as adding enormous amounts of organic fertilizer annually to agricultural soil in China for vegetable production was an essential source of heavy metals in the soil (Liu et al., 2005). Long-term fertilization with mineral and nitrogen fertilizers can increase the concentration of micronutrients in the soil solution. The concentration of Zn and Cu increases when the soil is fertilized with nitrogen. At the same time, permethrin significantly reduced these elements, and fertilization with phosphorus and potassium did not affect the concentration of

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the mentioned elements in the solution soil (Rutkowska et al., 2009). The continuous addition of chemical fertilizers to agricultural soil also increased the concentration of heavy elements in it, and the long-term use of municipal and industrial wastewater in irrigation had a significant contribution to increasing the content of trace elements such as Cd, Cu, Zn, Pb, Ni, Mn, Cr in the soil surface and increasing the NPK compound fertilizer in a large amount led to an increase in the concentration of Cd and Ni (Mapanda et al., 2005). In contrast, the concentration of Hg in agricultural soil decreased. Adding large quantities of chemical and animal fertilizers and poultry manure also increases some elements such as Cd, Cu, and Zn in the soil and plants (He et al., 2005). Heavy metals may negatively affect the soil environment, agricultural production or product quality, and water quality, ultimately harming the organism's health through the food chain. These effects are closely linked to bioavailability, which in turn is controlled by the types of metal ions in the soil, so determining The free metal ion concentration in the soil solution is essential, and the free metal ion concentration depends not only on the total mineral content but also on the species of minerals present in the soil and some other environmental conditions such as pH, concentration of complexing ligands in the solution and soil colloid (Ene et al., 2009). Limestone and superphosphate fertilizers contain essential trace elements for plant nutrients as primary elements and some other metal impurities, such as cadmium, which is considered a severe pollutant (Taylor and Percival, 2001). Heavily fertilized soils contained lower levels of available and total metals than unfertilized soils, suggesting that long-term fertilization does not increase soil metal concentrations (Jones et al., 2002). Trace metal and heavy metal concentrations in common commercial fertilizers are generally low, and soil contamination with heavy metals depends mainly on soil organic matter, location and source of fertilizers. Moreover, using agricultural fertilizers for decades has not significantly increased metal concentrations in soil (McBride and Spiers, 2001). Pollution with heavy metals is a serious environmental problem because they are not biodegradable; some show bioaccumulation, and most have toxic effects on living organisms when they exceed a certain level in the soil. Some of them are stationary due to their natural movement to some extent. At the same time, some are mobile; therefore, there is a potential possibility of their transmission through soil layers to the aquifer or through absorption by plant roots (Sherene, 2010). Heavy metals are known to cause serious health problems, such as tumors caused by carcinogens and other diseases. Therefore, awareness of the health risks associated with environmental chemicals has led to a significant shift in international interest in preventing the accumulation of heavy metals in soil, food, and other ecosystems. They are of concern due to their non-biodegradable nature and long-term biological half-life (Mapanda et al., 2005; Ahmed et al., 2009).

This study aims to assess the extent to which fertilization affects the concentration of some heavy metals, such as Cd, Cr, Cu, Fe, Mn, Cr, and Ni, in the soil of the Tasawa agriculture project.

METHOD

Site description

This research was carried out on several farms in the Tasawa Agricultural Project, located in southwestern Libya at coordinates 26°12'7"N 13°23'23"E, with an altitude of approximately 400 meters above sea level. The project was established to produce improved seeds, and the area experiences an average temperature ranging from 10°C during winter to over 45°C during summer. The farms are irrigated using the sprinkling method with water from artesian wells, and the soil is sandy (Al-danasuri, 1967).

Sample collection and analysis of physio-chemical parameters

Soil samples were collected during the year 2022. A soil auger sampler was used to collect samples. 1.0 kg of soil was collected in polyethylene bags from each site. The required data were recorded on each bag and transported to the laboratory. The soil was air-dried after being homogenized to a constant weight, sieved through a sieve with a diameter of 2 mm, and kept in a dry place to conduct the required analyses (Makhzom et al., 2023).

Assessment of heavy metals in soil

For assessing heavy metals in soil samples, about 1.0 g of dried soil sample was digested in about 15 mL of aqua-regia (HCl: HNO3) (3:1) for approximately 4-5 hours as described by (Inoti et al., 2012). Using an electric heater, at a temperature of about 110°C. The digestion product was cooled and filtered through filter paper (Whatman No. 42) into a 50 ml volumetric flask and brought to the mark with deionized water, the concentration of heavy metals in the samples, was measured by an Atomic Absorption Spectrophotometer (NOVA-A400).

Determination of geochemical index (Igeo)

The geochemical index (Igeo) was calculated as mentioned in (Muller 1969). This method is widely used to evaluate the degree of contamination by comparing the heavy metal levels obtained with the background levels

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originally used with bottom sediments. It can also be applied to soil contamination assessment. As shown in equation (1):

$$I_{geo} = \log_2(C_n/1.5B_n) \qquad (1)$$

Where:

Cn is the concentration of the element in the enriched samples, Bn is the concentration of the element in the background soil or the initial value of the element, and the constant is 1.5 to reduce the effect of possible differences in the background values that can be attributed to the variation of rock differences (Salem et al., 2022). This method evaluates the degree of metal pollution in terms of seven pollution levels based on increasing values of the index, Table 1.

Table 1. Geochemical accumulation factor, its value and degree

Igeo value	Igeo degree	Pollution level
>5	6	**Highly polluted**
4-5	5	**Highly polluted or severely polluted**
3-4	4	**High pollution**
2-3	3	**Somewhat polluted or highly polluted**
1-2	2	**Somewhat polluted**
0-1	1	**Unpolluted or somewhat polluted**
0	0	**Uncontaminated**

Statistical analysis

All experiments were conducted by taking three replicates. Statistical analysis of the data was performed using the SPSS 16 software package (Nassar et al., 2021), and the significance of the Pearson correlation between various heavy metal concentration was calculated at confidence levels of 0.95 and 0.01 (P>0.05 and P > 0.01).

RESULTS AND DISCUSSION

Concentration of heavy metals in soil samples

Table 2 presents the mean ± SD of heavy metals in soil samples. The concentration of Cr in cultivated soil samples ranged between 13.75 - 38.05 mg/kg with a mean of 25.97±11.02, whereas its concentration in uncultivated soil ranged between 25.56 and 29.43 with a mean of 27.42±0.54 mg/kg. However, the concentration of Cr was below the permissible limits set by the World Health Organization/FAO 2001 (Salem et al., 1995; Hipkin et al., 1999; WHO/FAO 2001; Salem et al., 2005; Bhatti et al., 2016; Al-Ethawi et al., 2019; Salem et al., 2020; Salem et al., 2022). High concentrations of Cr, reaching 58.3–62.3 mg/kg, have been recorded in agricultural lands irrigated with wastewater (Hipkin et al., 2004; Saltali et al., 2005; Heekmah 2006; Salem and Abuhadara 2010; Khan et al., 2013;Banson et al., 2014). In addition, high concentrations of Cr, ranging between 47.76 and 89.23 mg/kg, were also recorded in agricultural soils that had been fertilized with chemical fertilizers for an extended period (Li et al., 2011). The current results are consistent with what was recorded in the Punjab region of India, where the concentration of Cr was close to what was recorded in this study (Table 4), where its value ranged between 8.86 and 35.58 mg/kg (Bhatti et al., 2016). A high concentration of Cr, reaching 28.3 mg/kg, was also recorded in Spanish agricultural soil due to climate change (Naveedullah et al., 2013). The Cu content in soil samples from cultivated soils ranged between 0.04 and 2.27, with a mean of 0.95±0.94 mg/kg. However, Cu was not detected in uncultivated soil samples. The low Cu content in the soil is due to the non-industrial study area, so there is no apparent source of contamination with this element. Pollution with Cu might be attributed to various human activities, such as excessive chemical fertilizers and low-quality irrigation water (Bhatti et al., 2016; Markovic et al., 2010; Hang and Jin, 2008). Cd is one of the most environmentally toxic heavy metals, as it adversely affects soil suitability, plant metabolism, biological activity, and the health of animals and humans (Kabata-Pendias, 2000). Table 4 shows the concentration of Cd ranged between 0.69 - 6.72 with a mean of 4.37±3.23 mg/kg which is below the permissible limits (WHO/FAO 2001). However, Cd was not detected in uncultivated soil samples. The present results are consistent with previous results reported in [60]. High levels of Cd have been recorded in soils irrigated with wastewater, in areas with high vehicle traffic, excessive use of agrochemicals, or located near industrial areas [Lehoczky et al., 2004; Bhatti et al., 2016; Ebong and Ekong, 2015). The mean concentration of Mn in the cultivated soil was 12.96±7.59 and ranged between 6.36 - 22.80 mg/kg. Whereas in the uncultivated soil's concentration ranged between 10.66 -17.90 with a mean of 13.51±1.22, the Mn concentration in the cultivated and uncultivated soil was less than the permissible limits (WHO/FAO 2001). Low Mn, Cr, Cu, Ni, Cd, and Zn concentrations were recorded in soil samples irrigated with river water in Swat Province, Pakistan (Salem and

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Alwalayed 2019; Khan et al., 2016). The concentration of Zn in cultivated soil samples ranged between 0 - 6.54 mg/kg with a mean of 2.21±2.98 mg/kg which is below permissible limits (WHO/FAO 2001). however, Zn was not detected in uncultivated soil samples. The recorded concentrations indicating a zinc deficiency in the soil, which could be attributed to the sandy nature of the soil, and the accumulation of zinc ions in the cultivated soil may have been due to the addition of chemical fertilizers. Previous reports also indicated low Zn concentration in soil samples in Accra, Ghana (Fosu-Mensah et al., 2017). High concentrations of Zn were recorded in soil samples collected from electronic waste and other landfill sites (Li et al., 2011; Salem and Noralldien, 2018). The concentration of Ni ranged between 0 -24.79 mg/kg, with a mean of 9.15±10.93 in cultivated soil samples, while its concentration in uncultivated soil samples ranged between 6.35 - 8.05 with a mean of 6.92 ± 0.25 mg/kg (Table 4). These concentrations are below the permissible limits for agricultural soil (WHO/FAO 2001). The presence of Ni in soil samples can be attributed to its presence as an impurity in chemical fertilizers and the sandy nature of the soil. Ni can also be found in considerable quantities in nature. It is essential for living organisms in deficient concentrations and can be toxic when it exceeds the maximum permissible limit (Salem and Chergawi, 2013). High concentrations of Ni reaching 15.1 mg/kg were recorded in cultivated and uncultivated soils in Turkey (Aydinalp, 2009; Jia et al., 2010; Salem et al., 2014; MicoLlopis et al., 2006). High concentrations of Ni, ranging from 21.16-41.18 mg/kg, have also been reported in agricultural soils due to long-term agricultural activities (Jia et al., 2010). In the Punjab region of India, Ni concentrations amounting to 9.67-24.32 mg/kg were found in soil samples collected from four different sites (Vanita et al., 2014). Iron is one of the dominant metals among the essential elements in agricultural soil, showed an average concentration in the studied soil samples ranging between 149.23- 346.53 mg/kg with a mean of 267.50±84.96 mg/kg, while its concentration in uncultivated soil ranged between 516.00 - 886.00 with a mean of 651.17±59.38 mg/kg. This high concentration of Fe, ranging from 10,98 -19,81 mg/kg, reported in agricultural soil samples in the Segura River Valley in Spain (MicoLlopis et al., 2006) further underscores its significance. Similarly, high concentrations of iron were also recorded in soil samples taken from the Amritsar region in the Punjab province, India, where its concentration ranged between 2809-5304 mg/kg in agricultural soil, and it is believed that this is due to prolonged irrigation with wastewater and the continuous use of fertilizers. Inorganic (Vanita et al., 2014).

Table 2. Concentration of heavy metals in soil samples

Soil sample		**Heavy metals (mg/kg. dwt)**						
		Cr	Cu	Cd	Mn	Zn	Ni	Fe
Cultivated soil		20.15	0.04	ND	22.80	0.26	3.78	149.23
		31.93	0.70	0.69	12.78	ND	5.69	305.16
		13.75	2.27	6.72	7.09	6.45	8.02	269.08
		38.05	0.78	5.71	6.35	2.14	24.79	346.53
	Min.	13.75	0.04	0.69	6.35	N. D	N. D	149.23
	Max.	38.05	2.27	6.72	22.80	6.45	24.79	346.53
	Mean ± STD	25.97±11.0	0.95±0.9	4.37±3.2	12.26±7.6	2.21±3.0	9.15±10.9	267.50±85.0
Uncultivated soil	Min.	25.56	ND	ND	10.66	ND	6.35	516.00
	Max.	29.43	ND	ND	17.90	ND	8.05	886.00
	Mean ± STD	27.42±0.5	ND	ND	13.51±1.1	ND	6.92±0.2	651.17±59.4

Geochemical index

Table 3 displays the average contamination levels and geoaccumulation values of various metals found in soil samples. The results indicate that the area of soil under study is not considerably polluted with Cu, Cd and Zn due to the absence of any human activities, heavy traffic, or usage of polluted water for irrigation. However, the remaining elements, Cr, Mn, Ni and Fe, are somewhat polluted. The reason for this pollution could be the long-term usage of chemical fertilizers that contain impurities of these elements. These findings are in line with the results reported in previous studies (Rahman et al., 2012; Fosu-Mensah et al., 2017).

Table 3. mean of Igeo values and pollution of heavy metals

Elements	**Igeo value**	**Pollution level**
Zn	0.0	Uncontaminated
Cu	0.0	Uncontaminated
Cd	0.0	Uncontaminated
Cr	0.188	Unpolluted or slightly polluted
Mn	0.230	Unpolluted or slightly polluted

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Ni	0.326	Unpolluted or slightly polluted
Fe	0.081	Unpolluted or slightly polluted

Statistical analysis

The Pearson correlation coefficient study with the 2-tailed test indicated at the 0.01 level (P>0.01) Table 4. Between heavy metals, a significant positive correlation was found between Fe-Mn (r=.987**). In contrast, a significant negative correlation was observed between Mn-Cr (r = -.943**) and a negative correlation at the (p<0.05) was observed only between Fe-Cr (r= -.878*). These results are consistent with the results reported by (Salem and Al-Ethawi 2013; Al-Ethawi and Salem, 2013).

Table 4. Correlation between heavy elements in soil samples

	Cr	Cu	Cd	Mn	Zn	Ni	Fe
Cr	1						
Cu	-.143-	1					
Cd	.502	.422	1				
Mn	-.943**	.172	-.344-	1			
Zn	-.714-	.688	-.154-	.777	1		
Ni	.468	-.529-	-.073-	-.264-	-.341-	1	
Fe	-.878*	.163	-.245-	.987**	.760	-.156-	1

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

CONCLUSION

From the results, we can conclude that the concentration of all the heavy metals studied was less than the maximum permissible limit in agricultural soil according to the standards of the World Health Organization/Food and Agriculture Organization 2001, while the accumulation index showed that the soil samples were not contaminated with Cu, Cd, and Zn, while the rest of the elements showed that The Cr, Ni, Fe, and Mn were uncontaminated or slightly contaminated. The Pearson correlation coefficient showed the presence of a strong positive correlation between (Fe-Mn) and a strong negative correlation between (chromium - manganese) and only a negative correlation between (iron and chromium). These results indicate that chemical fertilizers will not lead to the accumulation of heavy metals in the soil above permissible limits. Using these fertilizers is not expected to cause a harmful effect in terms of heavy metal pollution, but a slight annual increase can be expected. This increase and other potential inputs of heavy metals into agricultural soils should be monitored, changes that could occur in the soil should be monitored, and strategies should be developed to reduce and control pollution.

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