An-Najah National University
Faculty of Graduate Studies

THE EFFECT OF SEWAGE SLUDGE FERTILIZATION AND TREATED WASTEWATER IRRIGATION ON THE ACCUMULATION OF HEAVY METALS IN CALCAREOUS SOIL, VERTISOLS SOIL AND BARLEY PLANT (*HORDEUM VULGARE*)

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Dedication

I dedicate my thesis to my parents, my wife, my children,

my brother and sisters

With all respect

Mohammed

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Declaration

I, the undersigned, declare that I submitted the thesis which is titled:

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I declare that the work provided in this thesis, unless otherwise referenced, is the researchers own work, and has not been submitted elsewhere for any other degree or qualification.

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Signature: 

Date: 20/08/2023

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Abstract

This experiment was applied to study the effect of sludge fertilization and irrigation with treated wastewater produced from the Jenin treatment plant on the accumulation of heavy metals in vertisols soil, calcareous soil, and barley plants. The experiment was conducted in a greenhouse under controlled conditions at the National Center for Agricultural Research in Qabatia, Jenin. This study used the soil column experiment with two types of soil: vertisol soil and calcareous soil. A section of each type of soil was taken at a depth of 1 m, and then it was represented in PVC tubes (6 columns for each type of soil) with a length of 1 m and a diameter of 72 mm. Every three soil columns were arranged into lines for the experiment, with two lines for each type of soil. These lines were then connected to an irrigation network. Then 200 ml of sludge was applied to a line of each soil type as fertilizer. After that, during the following growing season of 2021–2022, three barley seeds were sown in each column. After three months of irrigation with treated wastewater, the plants were harvested, the soil columns were dried, and they were divided into three layers (1-30 cm, 30-60 cm, and 60-100 cm). The chemical analyses were used to measure the content of heavy metals (Fe, Mn, Zn, Cu, Cr, Ni, Co, Pb, Cd, and As) in plants and soil layers using an ICP-MS device. The major nutrients N, P, and K were measured in the plant samples, and the length and weight of the plant were recorded. The results showed that fertilization with sludge and irrigation with treated wastewater increased the concentrations of Fe, Mn, Zn, Cu, and macronutrients N, P, and K in barley plants. Also, fertilization with sludge increased the weight of barley plants. The results confirmed that Vertisol soil is more fertile than calcareous soil. Fertilization with sludge increased the movement of heavy metals in the soil column and their filtration into the bottom layer. Sludge can be considered a source of plant nutrients as it contains macronutrients such as nitrogen, phosphorus, and potassium and micronutrients such as iron, zinc, and manganese.

Keywords: Calcareous soil; column experiment; heavy metals; vertisols soil.

Chapter One
Introduction

1.1 Research background

The agricultural sector contributes to both economic growth and food safety. With the increase in the population, the demand for agricultural products increases, and therefore, increasing the cultivated area has become a necessity.

One of the most important challenges facing the agricultural sector in Palestine is the lack of irrigation water (Castro et al., 2011). The traditional water sources are under unfair Israeli control, and it is imperative to deal with available resources wisely and sustainably. In recent years, treated wastewater has been used to irrigate certain crops, as well as play a positive role in preventing the pollution of agricultural lands and conventional water sources with wastewater (Rajaa et al., 2015). Treated wastewater offers an opportunity to reduce pressure on fresh water (FW) and contains nutrients that can replace mineral fertilizers (Maaß et al., 2016). Globally, there is a significant trend toward using treated wastewater for irrigation, especially in arid and semi-arid regions. As a solution to reduce pressure on fresh water, it was proposed to use treated water in agriculture (Mahjoub et al., 2016) and specifically claimed to be safe if used according to standards recommended by the World Health Organization to reduce risk (Ines et al., 2017).

The treated wastewater may contain high levels of salts, toxic ions, heavy metals, and organic residues. This component may be transported to the food chain through the soil and plants (Khaskhoussy et al., 2015). Heavy metals (HM) are defined as metallic elements that have a relatively high density compared to water. While heavy metals are naturally present in the soil, geologic and anthropogenic activities like vehicle exhausts, miming, industries, domestic and industrial effluents, waste disposal, pesticides, and fertilizers increase the concentration of these elements to levels that are harmful to both plants and animals (Chibuike and Obiora, 2014; Tuzen, 2003). It is a major source of pollution in the environment. It enters the human body in several ways, such as ingestion and absorption, and becomes toxic when its accumulation rate is greater than its discharge rate (Sardar et al., 2013). Some heavy metals are needed by living organisms in small quantities, and if they exceed the permissible limit, they become harmful, such as Co, Cu, Fe, Mn, Mo, Ni, V, and Zn. On the other hand, some heavy metals, such as Pb, Cd, Hg, and As, have no beneficial effects on living organisms and are considered very harmful to them (Koupai et al., 2006).

Plants need certain heavy metals for their growth and assistance in their vital functions, but if the amount of these minerals increases, they can become toxic to plants. Some of the direct toxic effects of high mineral concentrations are inhibition of cytoplasmic enzymes and damage to cellular structures due to oxidative stress. As for the indirect toxic effects, for example, they replace the essential nutrients in the cation exchange sites for plants (Chibuike and Obiora, 2014).

Barley (Hordeum vulgare), a member of the grass family, is one of the most important food grains since ancient times, and among the grains, it ranks fourth in the world in terms of area and quantity of production. It is popular in cultivation due to its versatility and ability to adapt to unfavorable climatic conditions and soil conditions (Arendt and Zannini, 2013). Approximately 65% of the barley crop is consumed as animal feed, 30% for malting, and only 2–3% for human consumption (Aldughpassi et al., 2016).

Soil is the uppermost layer of the earth's surface, and it is thin and crumbly. The plant can permeate it through the roots and absorb water from it for its life. Soil is usually described as the mantle or skin that covers the land mass of the planet; in some places the bare rocks are exposed and the thickness of the soil is a few centimeters, and in some places the thickness of the soil is several meters, and it is in the form of layers that reveal the composition or history of soil deposition. Soil is the natural medium for plant growth and has a thickness that is determined by the depth of rooting in plants. It consists of solids (mineral and organic), liquids, and gases (Eswaran and Reich, 2004).

Vertisol is a clay soil with deep and wide cracks occurring throughout the year. Shrinks when dry and swells when wet, has a high swelling capacity, and undergoes natural shrinkage with no air entering during the drying cycle. It is suitable for mechanized cultivation if there is a lot of rain or irrigation water. The permeability of this soil is low, and irrigation of this soil may lead to waterlogging and salinity buildup (Eswaran and Reich, 2004).

Calcareous soil contains more than 15% of calcium carbonate in the soil, which may appear in different forms (powders, nodules, crusts, etc.). It lacks nitrogen, often has a deficiency of phosphorous, and suffers from a deficiency of micronutrients such as zinc and iron, which requires good soil management that suits the needs of agricultural crops (FAO). In countries with a Mediterranean, dry, and desert climate, the soil contains calcium in one or more horizons because the rocks in these areas, such as basalt, are rich in calcium. The second reason is the frequent exchange of dry and wet periods, and the length of the dry season is not suitable for deep leaching of solutions in the soil (FAO).

The negative effect of heavy metals on the growth and activities of soil microorganisms may also indirectly affect the growth of plants. For example, a decrease in the number of beneficial microorganisms in the soil due to a high concentration of minerals may lead to a decrease in the decomposition of organic matter, which leads to a decrease in the nutrients in the soil. Enzyme activities useful for plant metabolism may also be impaired due to heavy metals interfering with the activities of soil microorganisms (Chibuike and Obiora, 2014).

The treated wastewater and sewage sludge was used in the experiment are generated from the Jenin wastewater treatment plant, which is located in Jenin city and served 70% of the population of 53,000 in 2021. The plant treats domestic and industrial wastewater. The current daily production volume of the plant is 9000 m^3.

1.2 Research question

1. What is the effect of sewage sludge fertilization and treated wastewater irrigation on the accumulation of heavy metals in calcareous soil, vertisol soil, and the barley plant (Hordeum vulgare)?

2. What is the effect of soil properties on the accumulation of heavy metals in soil and plants after application of sewage sludge and treated wastewater?

1.3 Research objectives

The main objective of this research is to assess the accumulation and transport of heavy metals in calcareous soil, vertisol soil, and the barley plant (Hordeum vulgare) due to sewage sludge fertilization and treated wastewater irrigation.

1.4 Research motivation

In Palestine, the trend towards reusing treated wastewater is increasing as a solution to pollution resulting from wastewater, especially in agriculture, as it is the largest sector that consumes water. The treated wastewater contains salts, toxic substances, organic matter, and minerals, including heavy metals. Heavy metals are minerals that are harmful to the environment and living organisms, and they pose a threat to humans if they are transmitted through the food chain. Also, some heavy metals are necessary for plant nutrition and can be a substitute for chemical fertilizers. Heavy metals are increasing in concentration in Palestine with the passage of time due to natural and human factors. The climatic diversity in Palestine has an impact on the diversity of the soil and its different characteristics, and thus the diversity of agricultural patterns. The importance of this study is that it examines the possibility of using sludge and treated wastewater as a source for fertilizing plants through their content of useful heavy metals and the accumulation of heavy metals in plant tissues and soil. It searches for potential problems and has prospects for working on finding solutions to overcome any environmental risks or problems and providing solutions such as choosing appropriate crops, irrigation programming, farm management in a way that prevents expected risks, and resorting to supporting activities during irrigation to reduce risks.

Chapter Two
Literature review

2.1 Heavy metal

The relevance of heavy metals has been a topic of study and discussion over the years. These metals have an impact on humans, plants, animals, and soil, in addition to the water. In addition to being harmful to the general public's health, this

In countries that suffer from a shortage or scarcity of water, agriculture is considered a major source of income, and the need arose to search for other sources of irrigation, such as reusing domestic and industrial wastewater to irrigate agricultural crops, as this wastewater contains significant amounts of heavy metals (Rattan et al., 2005). The treated wastewater can be used as a resource for agricultural irrigation because it contributes to the phosphorous and organic materials necessary for the plant (Castro et al., 2011).

Heavy metals are naturally occurring elements that have a high atomic weight and a density at least five times greater than that of water. Because of their industrial, domestic, agricultural, medical, and technological uses, they have spread widely in the environment, raising concerns about their potential effects on human health and the environment (Tchounwou et al., 2012).

Sources of heavy metals in the environment include geological, industrial, agricultural, pharmaceutical, sewage waste, and atmospheric sources. Although heavy metals occur naturally and are found throughout the earth's crust, most environmental pollution results from human activities such as mining, smelting, industrial production, and domestic and agricultural use of components containing minerals (Tchounwou et al., 2012).

Heavy metal concentrations in sewage effluents are typically modest, but prolonged usage of these wastewaters in agricultural areas frequently leads to the accumulation of high levels of metals in soils (Lu et al., 2015).

Heavy metals enter the food chain and can cause disturbances and health problems for humans when their concentrations exceed safe limits (Rajaa et al., 2015). The World

Health Organization therefore established guidelines for the levels of heavy metals in soil and plants (see table A.9 in Appendix A).

The degree of toxicity with heavy metals depends on several factors, such as dose amount, method of exposure, chemical species, age, sex, genetics, and nutritional status of the exposed individuals. Heavy metals are not subject to chemical and biological degradation and generally do not leak from the surface soil, which gives them an opportunity to contribute to environmental pollution (Tchounwou et al., 2012).

Some heavy metals, such as Co, Fe, Mn, Ni, V, and Zn, are necessary for living organisms in minute quantities, but if they are excessively increased, they become harmful to living organisms. Other heavy metals such as Pb, Cd, Hg, and As do not have beneficial effects on living organisms (Chibuike and Obiora, 2014).

Cadmium is an element that is easily absorbed by plant roots and moves to the upper parts of the plant. It is a worrisome element because it is very persistent in the environment and highly toxic to animals and plants, Thus, it poses a danger to consumers (Gvozdena et al., 2013). It is a very important pollutant because of its high toxicity and great water solubility (Gubrelay et al., 2013).

Cadmium is toxic because of its long-term accumulation in the human body, and it is naturally present in the soil. It enters the body primarily through food, and the Cadmium crisis in Japan (Itay Etay) is still fresh in our minds. As a result of soil pollution in Cadmium and its transmission to rice crops, about 150 Japanese died (Tomoyuki, 2003). The amount involved should not exceed 70 micrograms per day according to the recommendations of the World Health Organization, as Cadmium is similar to calcium in its behavior, as it is deposited in the bones in the form of triglycerosis, Cadmium, and it is gradually replaced by calcium, which leads to osteoporosis and damage to the spine. The cumulative effect of Cadmium is due to the strong bonding of cadmium with body proteins, which makes the body get rid of cadmium. Cadmium is included in several industries, such as batteries and plastic, and is mixed with lead and zinc metals and in phosphate fertilizers of sedimentary origin. The age of cadmium in the soil ranges between 15 and 1100 years. Cadmium concentration in the soil is estimated at about 1 mg/kg and is concentrated in the surface layer, especially in the dry climate. Cadmium is found in the soil solution freely and in the form of complexes (Cadmium chloride, sulfate, or bicarbonate). Because Cadmium tends to be available in the soil and to plant more than other cations, it is widely contaminated in crops consumed, such as wheat and rice (Al-Khatib, 2004).

Chromium is a naturally occurring element found in the Earth's crust. Exposure to chromium has health risks based on its oxidative states (Tchounwou et al., 2012). Chromium is found in the air, rocks, soil, water, and vital materials (Soni, 1990). The amount of chrome in the ground shell is greater than that of copper, zinc, lead, and nickel. Chrome enters the environment in two ways: one is normal and the other is industrial, occurring naturally, such as in volcanoes, the vital cycle, and rocks. As for the sources, industrial includes burning oil, charcoal, fertilizers, etc. (Abbasi et al., 1998).

In one of his studies (Kaufman et al., 1976) on chromium, it was found that 10 mg/kg of body weight may cause a pyramid in the liver, kidney inflammation, and death. Also, chromium may cause many health problems when it is deposited in the affected sites of a fracture in the body or chronic wounds. Also, at its lowest levels, it can irritate the skin and lead to ulceration. As for its highest levels, it may harm the kidneys and damage the liver, as well as nerve tissues.

Zinc is an important element and has an important role in the physiological process and metabolism of many organisms. High concentrations of zinc can be toxic to living organisms (Nazir et al., 2015).

Lead is harmful to human health and is toxic. There is no safe level of exposure to lead, which is a common soil pollutant (Tong et al., 2000). The concentration of lead in the ground shell is 5.12 mg/kg, and the arrangement of this element in the ground shell is thirty –sixth (Soni, 1990). Lead is one of the elements with a natural presence in the ground shell; the concentration rate is about 16 mg/kg of soil. It is found in nature in the form of mineral materials, which is lead sulfide (PbS). There are also other forms of lead, such as lead carbonate, lead church and lead sulfate, this is one of the naturally contaminated natural sources. As for the industrial point of view, it enters many industries such as dyeing, fireworks, small and large shells, batteries, and welding wires (Al-Khatib, 1998).

Soil pollution caused by car exhausts and others remains on the surface of the soil, and it is difficult to wash it for many years. The level of lead concentration in the soil is added to the sewage waste; this is due to the high concentrations of lead in these wastes.

The majority of lead in the soil does not move to the top of the plant. This is because the absorbed bullets collect in the roots, which are transferred in very small quantities to the top of the plant. Until now, no toxic symptoms of bullets have been recorded, but the increase in the concentration of lead in plants causes a danger to animals because of the high lead content. The danger of lead to human health lies in plant contamination with lead-contaminated soil particles that can be ingested by humans and grazing animals. In general, the transfer of lead from the soil to the ingested part of the plant is low. The range of lead concentration in the soil ranges from 10–48 mg/kg (Al-Khatib, 1998).

Lead is one of the cations of the elements commonly found in contaminated soils, and its compounds tend to accumulate in the soil (Davies, 1990). There have been many studies in terms of the effect of the accumulation of cations of heavy elements in the environment. In the study, a layer of soil with high fertilizer rates was used, with a depth of up to 20 cm. They found that lead accumulated in the first 1 cm of the soil surface, and its concentration increased with depth. In some cases, they found lead at a depth of 30–40 cm (Malavolta, 1994). Lead accumulates in soil due to its low solubility (Alloway, 1990). The concentration of lead in the world's soil ranges between 10 and 20 mg/kg (Warren et al., 1971).

The sources of lead additions to the soil are diverse, but the most important of them are those resulting from the combustion of motor fuels, and the rate of addition may reach 50 g per ha per year (Cartwright et al., 1977). Phosphate fertilizers are another source of added lead (Modaihsh et al., 2004).

Lead is an example of a fine particle that is dangerous to the life of living organisms due to its toxicity. Lead is added to car fuel to muffle the sound that occurs when the fuel-air mixture ignites in the engine. Lead comes out of the car exhaust in the form of lead oxides, chlorides, and bromides, and it turns into lead carbonate in the atmosphere. It is a toxic metal that has the characteristic of gathering and accumulating inside living tissues, and it causes many diseases, from headaches to kidney failure (Zidan, 1996).

Copper is an element that occurs naturally in soil, water and air (WHO, 2004).

Iron and manganese are common elements in the earth's crust and found in water, and they are not dangerous (Dvorak and Skipton, 2014).

Nickel is a chemical element found in abundance on Earth, and its concentration in the soil has increased in the past few years (Duda-Chodak and Blaszczyk, 2008).

2.1.1 Classification of heavy metals

Heavy metal cations are divided into four groups according to their ability to be absorbed by plants and then reach the end of the food chain (Chaney, 1980). The first group contains cations of elements that are poorly soluble in the soil solution and are less available to the plant to the extent that they do not allow their transfer to the plant and thus do not pose a danger even if they are found in large quantities, such as chromium (Cr). The second group contains cations of elements that can be absorbed by plant roots but do not transfer to the rest of the plant at rates that could pose a danger to humans and animals, such as cations of lead and arsenic. The third group represents the cations of the elements that are absorbed by the roots and easily transferred to the rest of the plant parts, but they are toxic to it, and animals and humans are spared them because the toxicity of the plant prevents its transfer to the food chain as a result of the death of the plant, and this group contains copper and nickel cations. As for the fourth group, it is the one that poses health risks to humans and animals, as it can be transmitted from the soil to the roots of plants and then to the rest of the other parts of the plant; there is no toxicity to the plant, and it can reach humans and animals. The most important element in this group is cadmium.

Heavy elements are divided into two groups in terms of their ability to form chemical bonds. Cations metals A and B exist, as well as a common cation metal between them (Kamel et al., 2010).

Group A: It tends to form vehicles with fluoride ions and tends to connect with vehicles containing oxygen atoms. So it is more strongly attracted to water than it is to aluminum or cyanide. Therefore, it is deposited or formed into unjust complexes with ions PO_4, CO_3, and OH. It also includes the major nutrients of plants such as Ca, Mg, K, and Cr.

Group B: It tends to connect with vehicles containing I, N, and S atoms; it also tends to connect with ammonia more than water.

2.2 Treated waste water

The large increase in population, water scarcity, the increasing demand for food products, and the increase in agricultural areas have led to an increase in water needs and the search for non-traditional water sources such as wastewater treatment.

Long-term irrigation of agricultural crops with wastewater may lead to the accumulation of heavy metals in the soil. Heavy metals can be transmitted to plants through their absorption from the soil and thus their transmission to the food chain, which poses a health risk to animals and humans who consume these plants that contain high concentrations of heavy metals. Therefore, the presence of metals in contaminated soil aroused the interest of researchers and environmental agencies (Rattan et al., 2005).

Domestic and industrial wastewater contains inorganic substances and potentially toxic elements such as arsenic, cadmium, chromium, copper, mercury, and zinc. If heavy metals are found in high concentrations that affect humans, then the use of treated wastewater is subject to limitations (Pescod, 1992).

In an experiment conducted on different crops of grains, fodder, and vegetables grown using groundwater and wastewater, Samples were taken from soil, plants, groundwater, and wastewater and analyzed. The result was that the wastewater contained a much higher amount of P, K, S, Zn, Cu, Fe, Mn, and Ni compared to groundwater. With regard to the mineral contents of some vegetable crops grown in soil irrigated with wastewater, it was found that they can be consumed safely (Rattan et al., 2005). In another study, the use of wastewater in horticultural crops achieved socio-economic benefits but had bad environmental and health effects, such as land degradation. As a result of the analysis of the samples, the use of wastewater in horticultural crops has increased the concentration of heavy metals in the soil in a way that may cause potential environmental and health risks to the crops (Mapanda et al., 2005). In another study, the grass was irrigated using treated municipal wastewater and compared with regular irrigation water to study the effect of irrigation with treated wastewater on the properties of the soil and its content of nutrients and heavy metals and to evaluate the effects of the continuous use of treated water on soil and crop yield. In this study, two plots of land planted with grass and irrigated with drinking water and treated wastewater were monitored over a period of two years. Physical and chemical factors in soil and plant tissues were analyzed. The study showed that the treated wastewater can be used as a resource for agricultural irrigation because it contributes to the phosphorous and organic materials necessary for the plant (Castro et al., 2011). In another research project, it aims to compare the effects of irrigation with fresh water and treated water on the soil in Tunisia in terms of the effect of irrigation water quality on soil properties, nutrients, and heavy metal content in order to establish the safe use of treated water in agriculture. Treated wastewater is considered a source of nutrition for the plant, as the plant has the ability to absorb heavy metals. When these minerals accumulate in the soil, the plant absorbs toxic and non-toxic metals. Six plots of land were allocated for irrigation application using fresh water and treated wastewater, with the aim of studying the effect of irrigation on different soil depths where the soil was divided into five layers, each with a thickness of 30 cm. The region suffers from a scarcity of water resources, and the study aims to search for sources of irrigation through wastewater treatment. Also, irrigation with environmentally treated wastewater is better than direct disposal of wastewater in nature. Also, treated wastewater is considered a source of plant nutrients and nutrients necessary for soil fertility, but the study assumes that the main risk of using treated water is its chemical content of salts, toxic ions, heavy metals, and organic residues, as the accumulation of these pollutants can cause damage in fields that are irrigated for a long time. It poses a threat to agricultural production and the environment (Khaskhoussy et al., 2015).

2.3 Treated waste water in Palestine

Palestine suffers from a scarcity of renewable water, and the average water consumption is 72 liters per person per day in the West Bank and in Gaza, which reaches 90 liters per person per day, and this rate is much lower than the standards recommended by the World Health Organization (World Bank, 2004).

More than half of the available groundwater is used for domestic water supply, which limits the volume available for industrial and agricultural development (PCBS, 2009).

Groundwater quality has deteriorated due to leakage of untreated sewage or direct sewage discharge into the environment without treatment (World Bank, 2009).

As a result of water scarcity and water resource pollution, wastewater treatment has become a priority to provide a partial solution to the water problem in the West Bank and Gaza Strip by reusing treated water as another source for agricultural irrigation, which consumes 70% of the water (PCBS, 2009).

The Palestinian Standards Institute, the Environmental Quality Authority, Palestinian ministries, and universities have set specific regulations for the reuse of treated wastewater by setting physical, chemical, and biological standards for non-hazardous liquid waste if it is used for irrigation (PSI, 2012).

2.4 Sewage sludge

Wastewater treatment plants produce large quantities of sludge on a daily basis, and the sludge contains soluble and insoluble impurities. Sludge is rich in nutrients such as nitrogen, phosphorus, and potassium, contains useful organic material, and can be considered a cheap fertilizer. To reduce the negative effects of sludge on human health and reduce environmental pollution, it needs proper management that includes reuse in agriculture in a way that prevents harmful effects on the environment and living organisms (Sharma et al., 2022). Sludge also contains pathogenic organisms and can be an odor source. Treatment is done after digestion, and sludge disinfection is required before sludge can be used in agricultural lands. For the agricultural use of sludge, one hectare is required for a population of 1,000. Poisonous metals may be concentrated in the sludge and may limit the value of its use on the land (Scholz, 2016).

Sewage sludge is the residual solid or semi-solid material or slurry that is produced as a by-product of wastewater treatment processes. These residues are classified as primary and secondary sludge. Primary sludge is generated from chemical precipitation, sedimentation, and other primary processes, while secondary sludge is the biomass of active waste resulting from biological treatments (Encyclopedia Britannica).

The high concentration of heavy metals in sewage sludge hinders its application on the ground. The total concentration of heavy metals in the sludge is estimated at 2%–5% of the total solids (Pathak et al., 2009). Because the heavy metals in the soil are not biodegradable, the use of sludge for a long time in agricultural lands leads to the accumulation of heavy metals and results in environmental toxicity, increasing soil pollution and reducing its productivity, in addition to being washed into the soil and transferred to groundwater (Suanon et al., 2016).

A study was conducted on soil treated with sewage sludge to measure the depth and movement of Cd, Cu, Ni, and Zn out of the surface sludge layer into the soil. The study was carried out using the soil column experiment, where six soil columns made of Polyvinyl chloride (PVC) pipes, 120 cm long and 10 cm in diameter, were used and they were built in a greenhouse. Soil samples were taken at a depth of 1 m, and sludge was applied to the columns three times. The columns were then irrigated with the water of the Colorado River by drip irrigation, and after 25 months, the columns were dismantled and divided into layers 10 cm thick and analyzed in the laboratory. The results of the analysis showed an increase in the concentration of minerals in the upper layer, while the bottom of the columns did not show an increase in the concentration of minerals (Emmerich et al., 1982).

2.5 The effect of heavy metal on soil

Minerals exist either as separate entities or in combination with other soil constituents. These constituents may include exchangeable ions adsorbed on the surfaces of inorganic solids, non-exchangeable ions, insoluble mineral compounds such as carbonates and phosphates, a soluble metal compound or free metal ions in soil solution, a mineral complex of organic matter, and minerals bound to silicates of minerals. Minerals associated with silicate minerals represent a concentration of soil minerals and do not cause pollution problems compared to minerals present as separate units (Chibuike and Obiora, 2014).

Soil properties affect mineral availability in a variety of ways, with pH being the main factor affecting mineral availability in soil. It has been proven that organic matter and aqueous ferric oxide reduce the availability of heavy metals through abiotic inactivation of these metals. Significant positive correlations were recorded between heavy metals and some soil physical properties, such as moisture content and water holding capacity. Other factors affect the availability of minerals in the soil, such as the density and type of charge in soil colloids, the degree of complexity of bonds, and the relative surface area of the soil. Also, soil aeration, microbial activity, and mineral composition affect the availability of heavy metals in the soil (Chibuike and Obiora, 2014).

Heavy metals may modify soil properties, especially biological properties. Heavy metals affect the number, diversity, and activities of soil microorganisms. The toxicity of these metals to microorganisms depends on a number of factors, such as temperature, pH, clay minerals, organic matter, anions, inorganic cations, and chemical forms of the metal. The presence of one heavy metal may affect the ability of other minerals in the soil and thus the plant (Chibuike and Obiora, 2014).

The study was also conducted with the aim of evaluating the accumulation of heavy metals in the soil and the edible part of the artichoke plant irrigated with secondary and tertiary treated wastewater. The research was carried out in a field planted with artichokes, where the properties of the soil were studied and three types of irrigation water were applied: fresh water (FW), secondary treated wastewater (SWW), and tertiary treated wastewater (TWW). Water samples and soil samples were taken at two depths (0–40 cm and 40–80 cm), and artichoke samples were taken. After analyzing the samples, the results showed that the concentrations of heavy metals in FW were lower than those in SMM and TWW. While the concentrations of Al, Cr, Cu, Ni, Zn, and Mn in the upper layer of soil were higher than in the lower layer, the concentration of Fe in the lower layers was higher than in the upper layer. The content of heavy metals in the artichoke plant irrigated with SWW and TWW was higher than in the plant irrigated with FW (Gatta et al., 2018).

Soil heavy metal cations occur naturally in one or more of the following six forms: Dissolved in the soil solution, adsorbed on the mineral solid part (soil granules), associated with the organic matter, precipitated as a solid as secondary minerals in the soil, and contributing to the building of crystals of the primary elements in the soil. However, the presence of cations of heavy elements in the soil in the first four forms may be greatly contributed by man as a result of his various activities in agriculture and industry, in addition to their presence naturally as a result of their dissolution during weathering processes in the soil (Langmuir et al., 2003). It was found that the concentration of heavy metal cations in the soil is higher than the concentration in surface and groundwater, and this is due to the evaporation-transpiration process, in addition to the higher ratio of the solid part to the liquid part in the soil as a result of the effect of dilution in water (Langmuir et al., 2003). The increase in the concentration of heavy metal cations causes them to turn into toxic elements, which leads to serious health problems if they enter the food chain (Oliver, 1997).

Agricultural activity results in the accumulation of many pollutants as a result of the use of agricultural chemicals such as fertilizers, pesticides, and sewage waste, which add to the balance of heavy metal cations in the soil and increase its quantities annually according to the type and nature of the pollutant. In a study in which more than 70 samples of fertilizers produced locally or imported and traded in the market were collected as they are commonly used, the content of these fertilizers was estimated from some heavy element cations (nickel, chromium, lead, cadmium, and cobalt). The cations of heavy elements vary according to the type of fertilizer and the type of element cation. They also concluded that phosphate fertilizers contain the most heavy element cations compared to the multi-component solid fertilizers and the soluble fertilizers, as the latter have a very low content of heavy element cations (Modaihsh et al., 2004).

Environmental studies on the inputs and outputs of cations of heavy elements and the environment's balance showed that there is an increase in the concentrations of cations of heavy elements in the surface soil at the global level as a result of the increase in industrial and agricultural activities. There is much evidence that the surface soil layer is affected by pollutants locally or transported from one place to another in the long term (Abdel Bari, 2000).

The use of wastewater in agricultural irrigation has a significant impact on most of the physical, chemical, and biological properties of the soil, as it was found that the accumulation of heavy element cations increases year after year, which leads to soil deterioration, and in the end, the concentrations of element cations may reach limits that are difficult to control (Al-Jailani, 1994). The pollution of water, soil, and plants with cations of heavy elements is one of the most important health and environmental risks as a result of irrigation with wastewater, especially when the pollution rate is high. And that the transfer of elemental cations differs according to the plant and environmental conditions, and what increases the risk of this matter is the accumulation of elemental cations in the root spreading area (5–40 cm) in conditions of dry environments, where irrigation with this water causes toxicity to the plant, and the severity of this toxicity depends on the sensitivity of the crop and the concentration of the elemental toxicity and the duration of exposure to this concentration (Chang et al., 1995).

The greater the amount of cadmium in the soil cultivated with grains, the higher its concentration in the grains, and he noted that the amount of cadmium was less in barley than in wheat (Amar et al., 2001). The process of transferring heavy metal cations from wastewater sediments to the soil, and then to groundwater and plants has serious environmental impacts (Bhogal, 2003). The chromium spreads between 0 and 5 cm, while lead may reach a depth of 60 cm, so lead represents a threat to food and ground water. He added that lead is absorbed by the plant in all stages of its growth and that chromium affects the germination process, as the corn crop failed to germinate as a result of the presence of chromium cation in the irrigation water by 10 ppm (Al-Khatib, 2004).

The pH is the main force that controls the adsorption of heavy metal cations (Schwertmann and Taylor, 1988). Adsorption of cadmium depends more on pH than that of lead. The adsorption of cadmium was less than or equal to the amount of negative surface charge. This indicates that the cadmium was most likely adsorbed by electrostatic surface interactions (Apple and Ma, 2002). The adsorption efficiency increases with the increase in pH when using a solution contaminated with cations of chromium, cadmium, copper, iron, and lead, due to the decrease in competition with hydrogen ions with the increase in pH (Agbozu and Emoruwa, 2014). The decrease in adsorption at low pH values is most likely due to increased competition between hydrogen ions for adsorption sites. And that the continuous increase in the adsorption of elemental cations by soil colloids after pH 6 can be explained by the precipitation of the elemental cation (Horsfall and Siff, 2004).

The degree of pH is an important factor in controlling the solubility of cations of the elements and their availability in the soil because of the extent of the difference in soil texture and its content of organic matter (Filiuse et al., 1998). The high pH values increase in the process of adsorption and sedimentation associated with the cations of heavy elements with iron oxides (Carmen and Murray, 2001).

The size distribution of the soil particles affects their content of chromium, lead, and nickel, and the clay particles contain a greater amount of these elemental cations than other cations because of their greater specific surface area than the coarse particles (Dzombak et al., 1990). The rate of pollutant transfer depends on the physical properties of the soil, specifically the particle size distribution and bulk density, because both of these properties affect the movement of water and air through the soil by influencing the porosity and hydraulic conductivity of the soil(Al-Khatib, 1998) . The proportion of organic matter and clay minerals explains most of the differences in the exchange capacity of the soil, and the important part of the organic matter in the soil is closely related to the inorganic components of the soil, such as clay minerals and oxides that are acidified when they are coated with clay minerals (Eriksson, 1998). The effect of the chemical and physical properties of the soil on the adsorption of cations of heavy elements is reflected in the calculation of the constants of their rates of movement and transmission within the soil (Hamad et al., 1997). The accumulation of heavy metal cations and their readiness for uptake by plants are highly correlated with the basic soil properties, the most important of which are pH, organic matter, clay content, soil content of calcium carbonate, equilibrium time, and cation exchange capacity (Murray et al., 1999).

Nickel and chromium accumulate in the upper layer between 25 and 30 cm, while the accumulation of lead appears at 15 cm (Lawes, 1993). An accumulation of heavy metal cations and some fertile element cations occurs in the surface layer between 0 and 15 cm (Shuman and Das, 2002). By studying several sites, it was found that cations of heavy elements accumulate in the surface layer of the soil (0–30 cm) and decrease with depth, and they noticed that the movement of cadmium in most of the sites was within the depth of 0–20 cm only, while lead reached a depth of more than 50 cm (Kibria et al., 2013).

The effect of an element's cation depends more on the available fraction than on its total concentration in the soil (Alen et al., 1995). The dependence on the available part gives a clear indication of the concentration of the element available to the plant because it is similar to its presence in the soil solution to a large extent in terms of the exchange process that takes place between the adsorbent complex and the soil solution (Al-Abdullah, 2007). The high concentration of some heavy metal cations in the soil may make the soil unproductive, and this may cause the bio-accumulation of heavy metal cations in the soil and their transmission to animals and humans (Abel-Sahab et al., 1994).

The process of retaining heavy metal cations present in wastewater sediment inside the soil is either through their adsorption or due to the formation of insoluble salts (Alloway and Jackson, 1990). The increase in the concentration of heavy metal cations in the soil makes them turn into toxic elements, which lead to serious health problems if they leak into the food chain (Oliver, 1997).

Heavy metal cations behave in a variety of ways in the environment after they are added, where they can be volatilized or disintegrated into other compounds by sunlight, or transported by surface runoff or movement within the soil sector to ground water, or adsorbed on soil colloids, or microbially degraded. The behavior of heavy metal cations requires in soil and plant to further studies (Abdel Bari, 2000). Some heavy metal cations remain forever once they reach the soil, due to their poorly soluble nature, as they quickly precipitate in the soil and turn into an insoluble form. Others of these elements enjoy solubility and a high ability to move, and these are more harmful, as they can move due to their high solubility to the bottom of the soil sector, causing groundwater pollution, and also because their solubility makes them in a form that is available to plants, so the quantities that plants absorb from these elements increase. Elements, and then enter the food chain and pass from plants to animals and humans (Wu, 2004).

Soil contamination with heavy metal cations leads to the inhibition of the enzymatic activity of microbes as well as a reduction in the diversity of plant and animal communities in the soil. Soil pollution also leads to an imbalance between the physical, chemical, and biological processes in the soil, which are the processes on the basis of which soil fertility is built. Milk and meat of animals fed on plants growing in contaminated soil all lead to the transfer of cations of heavy elements to humans (Forstner, 1991).

2.6 The effect of heavy metal on plant

Heavy metals available for plant uptake are those that are present as soluble components in the soil solution or those that are easily dissolved by root secretions (Chibuike and Obiora, 2014).

The ability of the plant to collect and absorb essential elements enables it to obtain other non-essential elements. Since minerals cannot be broken down, when sedimentation inside the plant exceeds optimum levels, it negatively affects the plant directly and indirectly (Chibuike and Obiora, 2014).

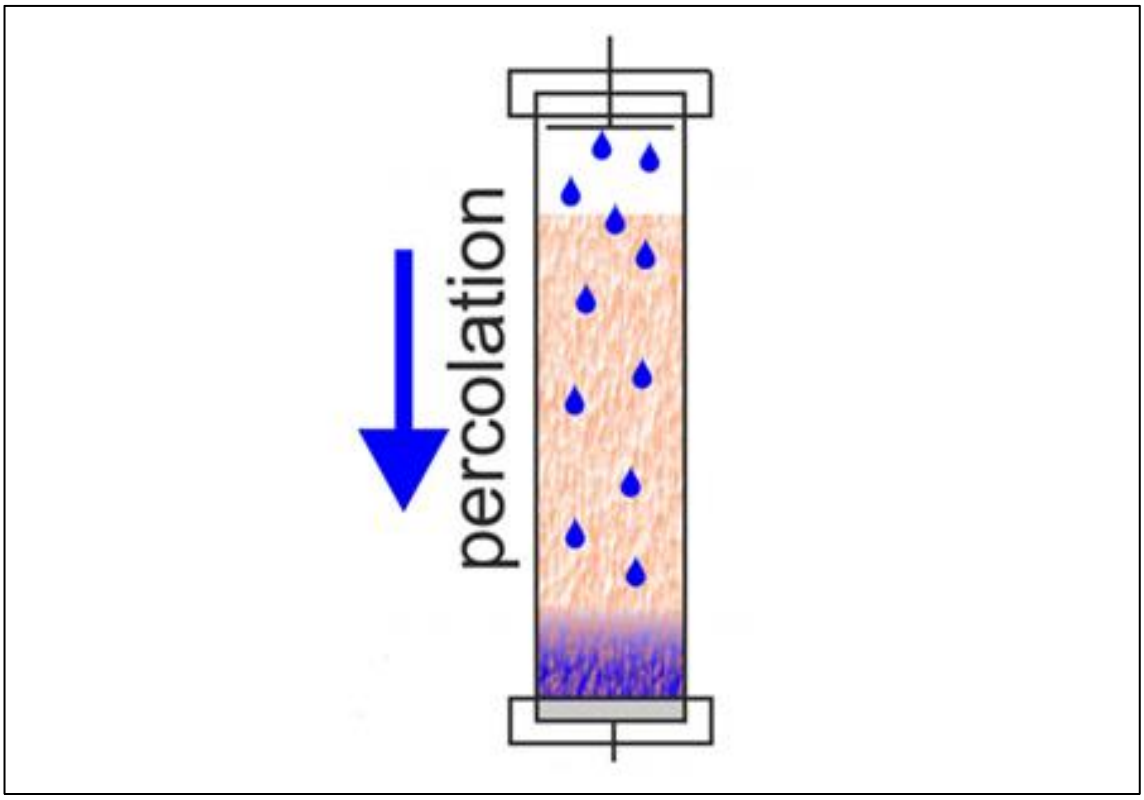
Some of the direct toxic effects of high concentrations of the metal include inhibition of cytoplasmic enzymes and damage to cellular structures due to oxidative stress. The negative effect of heavy metals on the growth and activities of soil microorganisms may indirectly affect the growth of plants. A decreased number of beneficial microorganisms in the soil due to the higher concentration of minerals may lead to decreased decomposition of organic matter, resulting in a decrease in soil nutrients. These direct and indirect toxic effects lead to a decrease in plant growth, which sometimes leads to plant death (Chibuike and Obiora, 2014).

2.7 Column experiments

Through the utilization of soil column experiments, we may simulate field procedures on the soil column. A soil column is defined as a discrete block of soil located either outdoors or in a laboratory that allows control and measurement of the infiltration and incorporates equipment for the total recovery of the effluent. This is usually achieved by encasing the soil column in a rigid and impermeable shell material. The column material must be rigid to ensure that the column offers the structural strength at the desired column size and wall thickness and inert to avoid any chemical interference with any solute. Column tests essentially consist in the percolation of a feed solution through a cylindrical column filled with soil under conditions that are aimed to be representative of the field site (Figure 2.1) (DEMEAU).

Figure 2.1

Injecting the irrigation solution into the soil column



Chapter Three
Methodology

3.1 Experimental setup

3.1.1 Experimental site

The experiment was conducted under controlled conditions in the greenhouse at the National Agriculture Research Center (NARC) in Qabatya – Jenin, north of the West Bank during growing seasons (2021/2022).

A factorial experiment was applied because it includes studying two or more factors and the interactions between the factors. The experiment was arranged according to a randomized complete block design (CRD) with three replications. The experiment consisted of two factors, soil type and sludge fertilization, and was arranged as shown in the table (3.1).

Table 3.1

Factorial experiment to CRD design arrangement of soil column experiment

S	F	R1	R2	R3
s1	f1			
	f2			
s2	f1			
	f2			

S: soil type (s1: vertisol soil and s2 calcareous soil).
F: fertilization (f1 fertilized with sludge and f2 without fertilized).

3.1.2 Soil material

The experiment was carried out using two types of soil: the first type was Vertsols soil taken from the village of Habla in the Qalqilya governorate, and the second type was calcareous soil taken from the village of Hajjah in the Qalqilya governorate. The soil was taken in the form of a longitudinal section with a depth of 1 meter for each type. Analyze the soil in the laboratory to see its properties such as pH, electrical conductivity, bulk density, cation exchange capacity (CEC), organic matter, moisture content (table 3.2) and heavy metals (table 3.3).

3.1.2.1 Soil properties analyze

a. pH

- Weigh 50 g of air-dried soil into a 100 ml glass beaker.
- Add 50 ml distilled water.
- Mix the suspension well, then leave it for 30 minutes.
- After an hour, stir the suspension well.
- The combined electrode of the pH meter is placed in the suspension at a depth of 3 cm.
- The device reading is taken after 30 seconds.

b. Electrical conductivity

- Prepare a suspension at a ratio of 1:1 (soil: water).
- Filter the suspension using a vacuum filtration system.
- The filtrate is placed in a 50 ml vial, then the conductivity cell is immersed in the solution and the result is taken.

c. Cation exchange capacity (CEC)

- Place 4 g of air-dried soil in a 40 ml tube, then add 33 ml of sodium acetate solution (NaOAc),1N, then plug the tube and shake for five minutes.
- Remove the stopper and place the tube in the centrifuge (3000 rpm) until the supernatant becomes clear. After that, the supernatant is completely filtered.
- Repeat the process with 33 ml of sodium acetate four times, and each time we drain the liquid, then add 33 ml of ethanol (95%), plug the tube and shake for 5 minutes, then remove the stopper from the tube and place it in the centrifuge until the supernatant liquid becomes clear.
- Wash the sample with 33 ml of ethanol (95%) solution three times, each time filtering the supernatant so that the electrical conductivity is less than 400 $\mu\delta/cm$.

- Replace the sodium (Na^+) absorbed from the sample by adding 33 ml of ammonium acetate solution three times. Each time, we shake the tube for 5 minutes and place the tube in the centrifuge until the supernatant liquid becomes clear.
- Filter the three liquids into a 100 ml volumetric flask. The volume is supplemented with ammonium acetate solution and the contents are mixed well.
- A series of appropriate standard solutions are measured and a calibration curve is drawn.
- Samples are measured and spectrum readings are taken using a flame photometer at wavelength 767 nm.
- The concentration of sodium (Na) is calculated according to the calibration curve.
- The following equation applies:

$$\text{CEC (meq/100 g)} = Na\ (meq/l) \times \frac{A}{Wt} \times \frac{100}{1000} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

A = Total volume of extract.

Wt = Weigh dry soil aerobically.

d. organic matter

- Weigh 1 g of soil into a 500 ml Baker's cup.
- Add 10 ml of $K_2Cr_2O_7$, 1 N solution, then add 20 ml of concentrated sulfuric acid and stir the cup well.
- Leave the suspension for 30 minutes.
- Add 200 ml of distilled water, then add 10 ml of concentrated phosphoric acid.
- Add 10 drops of diphenylamine, then place the cup on a magnetic stirrer.
- Calibration is carried out with a solution of ferrous sulphate and ammonium until the color changes from blue-violet to green.
- We apply the following equations:

$$M = \frac{10}{\text{V blank}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

$$Oxidized\ organic\ carbon\ \% = \frac{(V\ blank - V\ sample) \times M \times 0.3}{Wt} \ldots\ldots\ldots (3)$$

$$Total\ organic\ carbon\ \% = Oxidized\ organic\ carbon\ \%\ \times 1.334$$

$$Organic\ matter\ \% = Total\ organic\ carbon\%\ \times 1.724$$

M = Systemic solution of ferrous and ammonium sulphate (M 0.5).

$V\ blank$ = The volume of ferrous and ammonium sulphate solution needed to calibrate the control (ml).

$V\ sample$ = The volume of ferrous sulphate and ammonium solution needed to calibrate the sample (ml).

Wt = Weigh dry soil aerobically (gm).

$0.3 = 100\ \times 10^{-3} \times 3$, Number 3 is the equivalent weight of carbon.

e. Moisture content

- Weigh 10 g of air-dry soil for each sample.
- Dry the samples in the oven at 105°C.
- The next day, the samples are placed out of the oven and cooled for at least 30 minutes, then the samples are weighed again.
- The following equation applies:

$$Soil\ moisture\ \theta\ \% = \frac{Wet\ soil - dry\ soil}{dry\ soil} \times 100 \ \ldots\ldots\ldots.. (4)$$

Table 3.2

Soil properties for Vertisols soil and Calcareous soil

Properties	Vertisols soil	Calcareous soil
pH	6.7	8.2
EC (dS/m)	0.094	1.67
CEC [cmolc/kg]	51.82	12.2
Organic matter (%)	4	0.98
Sand (%)	16	64
Silt (%)	26	15
Clay (%)	58	21
Soil texture	Clay	Sandy clay loam
Bulk density (g /cm^3)	1.1	1.25
Total N (mg/kg)	2400	300
Available P (mg/kg)	10.2	4.2
Available K (mg/kg)	438	320

Table 3.3

HM concentration (ppm) in Vertisols soil, Calcareous soil, sewage sludge and treated wastewater

Type	Fe	Mn	Zn	Cu	Cr	Ni	Co	Pb	Cd	As
Vertisols	48.09	2.946	0.159	0.057	0.097	0.113	0.042	0.026	0	0.004
Calcareous	85.897	0.061	0.075	0.042	0.193	0.301	0.017	0.007	0.001	0.01
Sewage sludge	430	410	951.25	263.68	53.47	31.34	67	52.26	1.39	1.01
Treated wastewater	1.278	0.003	0.021	0.001	0.041	0.001	0	0	0.003	0.036

3.1.3 Plant material

The experiment was carried out using local barley.

3.1.4 Treated wastewater sewage sludge

TWW and SS used in the experiment were taken from the Jenin treatment plant, which treats domestic and industrial wastewater.

3.2 Column experiment

Has been created a soil experiment column by placing soil samples in plastic (PVC) tubes 1 m long and 72 mm in diameter (Figure B.1 in Appendix B), such that the arrangement of soil layers in the tube represents the natural arrangement of the sample (Figure B.13 and B.14 in Appendix B). (Six tubes from each soil).

3.3 Fertilization

After preparing the soil columns, 200 ml of sewage sludge was added to 3 columns of each soil type. The sewage sludge was taken from the Jenin treatment plant.

3.4 Irrigation

3.4.1 Irrigation network

After preparing the soil columns, a drip irrigation network were established consisting of a water tank and a network of pipes (Irrigation pipe diameter 16 mm), so that each soil column is under an irrigation drip (Figure B.2 in Appendix B).

3.4.2 Irrigation process

Each column was irrigated with 1 liter of treated wastewater. After 3 days of adding sewage sludge, the pipes were irrigated and 3 grains of barley were planted in each tube. Irrigation was repeated twice a week for a period of 3 months, and the amount of irrigation was 7 liters/column. The water used for irrigation is treated wastewater from the Jenin treatment plant and was transported to the experimental site using plastic containers.

3.5 Collecting Plant Samples-end of experiment

After 3 months of cultivation, the barley plant was harvested, and a sample of each column was placed in a bag and sent to the laboratory for chemical analysis.

3.6 Collecting soil Samples-end of experiment

After harvesting the plant, the soil columns were dried and then each column was cut into 3 layers (1-30 cm), (30-60 cm) and (60-100 cm). The soil samples were placed in bags and sent to the laboratory for chemical analysis.

3.7 Trace Elements Analysis

The following method was used to determine the levels of heavy metals in each sample that was collected.

3.7.1 Chemical Analysis

ICP-MS (Inductively Coupled Plasma Mass Spectrometry), a type of mass spectrometry that can identify metals in a sample at concentrations as low as 1 part per trillion, was used to conduct chemical analysis in the lab. The ICP-MS can be used as a qualitative instrument to identify the metal speciation in a sample or as a quantitative tool to calculate the concentration of a particular analyte.

3.7.1.1 Plant

The steps used were conducted from "Analysis of Major, Minor and Trace Elements in Plant Tissue Samples using ICP-OES and ICP-MS." (University of Wisconsin – Madison, 2005)

a. Procedure (before digestion)

Samples were dry stored in a 5-gram vial or equivalent after being dried at 60 ºC for two days. Dry samples of weigh 0.50±0.01 g. the samples were allowed to cool for an hour before being handled once they were cooled, After adding 5 mL of concentrated nitric acid (HNO3 - 70%), samples were allowed to soak for 2–3 hours at room temperature.

b. Procedure (Hot plate digester)

To decrease the evaporation of the water, tubes were put in the block heater and covered with plastic film. The block heater was then turned on and set to 70°C for three days. The block heater's tubes were removed, the film cover was taken off, and it was appropriately discarded. After that, 1 mL of 30% hydrogen peroxide was applied to each sample. After being put on the block heater, the tubes were heated for 20 to 30 minutes. The 50 mL mark, block heater, and all of the tubes were then removed. They were then let to sit for at least 30 minutes.

c. Measurement by ICP-MS

In order to determine the final concentration of heavy metals, 14 ml of the diluted sample was obtained and placed in falcon tubes.

3.7.1.2 Soil

The steps used were conducted from "Analysis of Major, Minor and Trace Elements in Soil and Sediment Samples with ICP-OES and ICP-MS" (University of Wisconsin – Madison, 2005)

a. Procedure (before digestion)

First, samples were dried at 60°C for two days while being freed of any large stones, boulders, or plant matter. For airtight storage, they were kept in a 5-gram vial or something comparable. Samples should be dry and weighed into 50-mL digestive tubes that have been cleaned and allowed to air dry. To wet the samples, drops of 20–30% (v/v) nitric acid were added. The samples were then given 5 mL of concentrated nitric acid.

b. Procedure (Hot plate digester)

To avoid the water from evaporating too quickly, all tubes were placed inside the block heater and covered with plastic film. The block heater was then set for three days at 70°C. The film cover was then taken off and disposed of properly. Tubes were removed from the block heater and allowed to cool for a while. Then, 1 mL of 30% hydrogen peroxide was applied to each sample. Then, for 20 to 30 minutes, all tubes were placed on the block heater. Finally, the block heater's tubes were removed. 5 ml of hydrofluoric acid [HF] (40%) were added after digestion and left for 24 hours. Water was then added up to the 50 mL mark, and the mixture was allowed to settle for at least 30 minutes. After this digestion, the samples were combined and left overnight to allow the particles to settle.

c. Measurement by ICP-MS

To obtain the final concentrations of heavy metals, 14 ml of the diluted sample were extracted and placed in falcon tubes.

3.7.2 Statistical analysis

Analysis of variance (ANOVA) was performed on laboratory data (heavy metal concentrations) and field data (plant height and weight), where univariate analysis was applied to heavy metal concentrations in the soil and plants, and multivariate analysis was applied to plant weight and height. Program was used SPSS.

Chapter Four

Result

4.1 Heavy metals concentration in plant

In order to test whether there is an effect of soil, sludge, and the interaction between those variables on heavy metals, univariate analysis was used. The results of this test appear in the table 4.1.

Table 4.1

Results of univariate analysis of heavy metal concentration in barley plant

Source	Sum of squares	df	Mean squares	F	Sig
Soil	0.013	1	0.013	0.092	0.763
Fertilization	9.584	1	9.584	65.927	0.000
Soil× fertilization	2.809	1	2.809	19.325	0.000
Soil× metal	0.385	9	0.043	0.294	0.974
Fertilization× metal	81.090	9	9.010	61.978	0.000
Soil× fertilization× metal	24.512	9	2.724	18.734	0.000
Error	11.630	80	0.145		
Total	1159.333	119			

From table (4.1) we can test the following hypothesis

1. H_0: there is no significant effect of soil on HM concentration in barley plant at level of significant ($\alpha = 0.05$).

   From the results in table (4.1) the p-value (sig) = 0.763 this value greater than $\alpha = 0.05$ which means don't reject the hypothesis which means al level of significant $\alpha = 0.05$ there is no sufficient evidence that there is an effect of soil on HM concentration in barley plant.

2. H_0: there is no significant effect of fertilization on HM concentration in barley plant at level of significant ($\alpha = 0.05$).

   From the results in table (4.1) the p-value (sig) = 0.000 this value less than $\alpha = 0.05$ which means reject the hypothesis which means al level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of fertilization on HM concentration in barley plant.

3. H_0: there is no significant effect of interaction between soil and fertilization on HM concentration in barley plant at level of significant ($\alpha = 0.05$).

   From the results in table (4.1) the p-value (sig) = 0.000 this value less than $\alpha = 0.05$ which means reject the hypothesis which means at level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of the interaction between soil and fertilization on HM concentration in barley plant.

4. H_0: there is no significant effect of interaction between soil and metal on HM concentration in barley plant at level of significant ($\alpha = 0.05$).

   From the results in table (4.1) the p-value (sig) = 0.974 this value greater than α =0.05 which means don't reject the hypothesis which means at level of significant $\alpha = 0.05$ there is no sufficient evidence that there is an effect of the interaction between soil and metal on HM concentration in barley plant.

5. H_0: there is no significant effect of interaction between fertilization and metal on HM concentration in barley plant at level of significant ($\alpha = 0.05$).

   From the results in table (4.1) the p-value (sig) = 0.000 this value less than $\alpha = 0.05$ which means reject the hypothesis which means at level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of the interaction between fertilization and metal on HM concentration in barley plant.

6. H_0: there is no significant effect of interaction between soil, fertilization and metal on HM concentration in barley plant at level of significant ($\alpha = 0.05$).

   From the results in table (4.1) the p-value (sig) = 0.000 this value less than $\alpha = 0.05$ which means reject the hypothesis which means at level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of the interaction between soil, fertilization and metal on HM concentration in barley plant.

Table (4.2) shows the concentration of heavy metals in samples of barley taken from a normal field, while Table (4.3) shows the concentration of heavy metals in the barley plant after the experiment. Concentration of Fe, Mn and Co (14.12, 0.596 and 0.013 ppm, respectively) was higher in VS treatment, whereas the concentration of Zn, Cu, Ni, Pb and Cd (0.684, 0.073, 0.088, 0.005 and 0.0005 ppm, respectively) was higher in

CS treatment, and the concentration of Cr and As (0.213 and 0.022 ppm, respectively) was higher in C treatment.

Table 4.2

HM concentration (ppm) in barley plant from normal field

Fe	Mn	Zn	Cu	Cr	Ni	Co	Pb	Cd	As
2.878	0.434	0.405	0.041	0.088	0.040	0.003	0.001	0.000	0.005

Table 4.3

The an average of HM concentration (ppm) in barley plant after treatments

Treatment	Fe	Mn	Zn	Cu	Cr	Ni	Co	Pb	Cd	As
VS	14.12	0.596	0.679	0.055	0.104	0.055	0.013	0.003	0.0003	0.006
V	5.608	0.511	0.564	0.059	0.119	0.045	0.004	0.001	0.0002	0.004
CS	11.14	0.165	0.684	0.073	0.183	0.088	0.004	0.005	0.0005	0.009
C	8.66	0.248	0.471	0.062	0.213	0.076	0.003	0.003	0.0003	0.022

VS: Vertisols soil with sludge fertilization.

V: Vertisols soil.

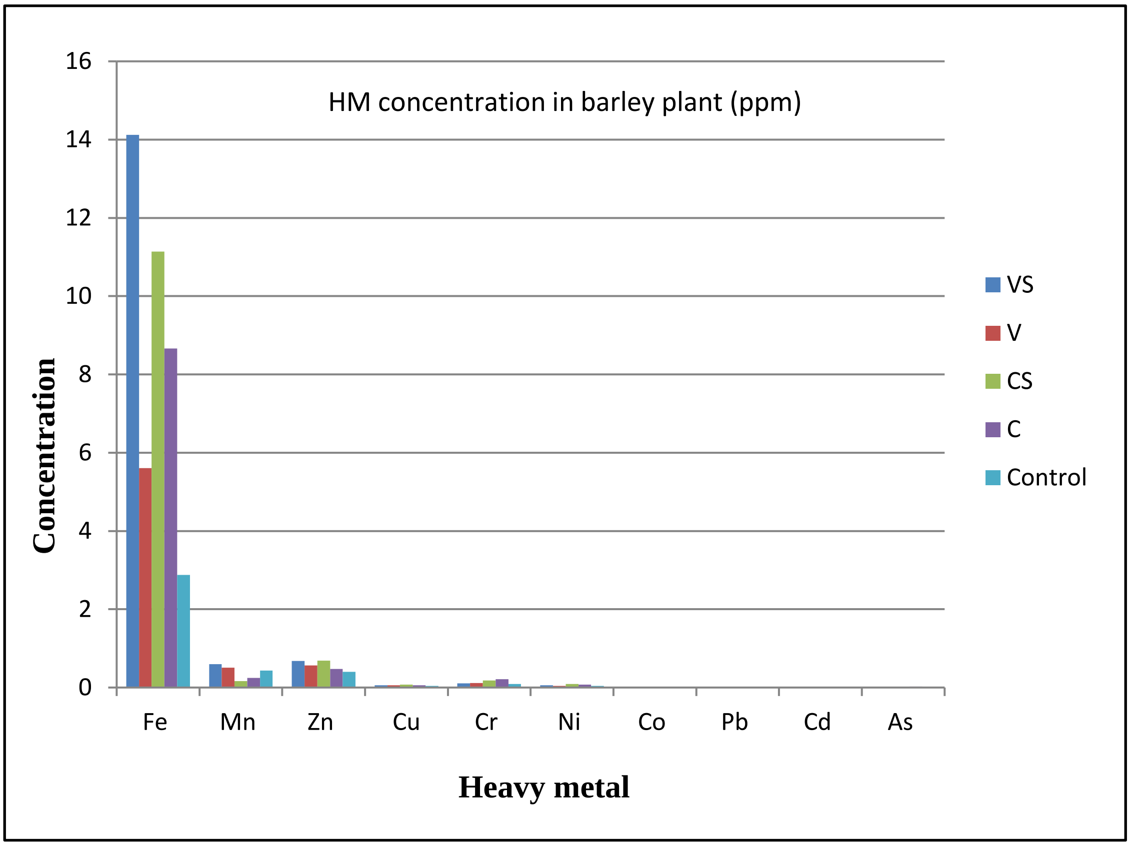
CS: Calcareous soil with sludge fertilization.

C: Calcareous soil.

Ferrous (Fe) concentration was significantly high in VS and CS (14.12 and 11.14 ppm, respectively), in contrast treatment C and V had the lowest concentration (8.66 and 5.608 ppm, respectively). For Manganese (Mn) concentration was significantly high VS and V (0.596 and 0.511 ppm, respectively), in contrast treatment C and CS had the lowest concentration (0.248 and 0.165 ppm, respectively). Zinc (Zn) concentration showed the highest value in CS, then in VS, then in V and then in C (0.684, 0.679, 0.564 and 0.471ppm, respectively). Copper (Cu) concentration showed the highest value at CS and C (0.073 and 0.062 ppm, respectively) whereas the lowest at VS and V (0.055 and 0.059 ppm, respectively). C treatment showed the highest concentration at Chromium (Cr) 0.213 ppm, thin in CS 0.183 ppm whereas the lowest at V and VS (0.119 and 0.104 ppm, respectively). Nickel (Ni) concentration showed the highest value at CS and C (0.088 and .076 ppm, respectively), whereas the lowest at VS and V (0.055 and 0.045 ppm, respectively). For Cobalt (Co) treatment VS showed the highest concentration (0.013 ppm) whereas no differences were observed between V, CS and C treatment. Lead (Pb) showed the highest value at CS (0.005 ppm) and the lowest at V (0.001 ppm) whereas no differences observed between VS and C. Cadmium (Cd) no differences observed between all treatments. Arsenic (As) showed the highest concentration at C (.022 ppm) whereas no differences observed between VS, V and CS.

Figure 4.1

HM concentration in barley plant



4.2 N P K concentration in plant

In order to test whether there is an effect of soil, sludge and the interaction between those variable in to barley plant Univariate analysis used. The results of this test appear in the table 4.4.

Table 4.4

Result of Univariate analysis of N P K concentration in barley plant after treatments

Source	Sum of squares	df	Mean squares	F	Sig
Soil	44.308	1	44.308	144.134	0.000
Fertilization	2505.604	2	1252.802	4075.376	0.000
Soil× fertilization	75.118	2	37.559	122.180	0.000
Soil× NPK concentration	0.712	1	0.712	2.317	0.141
Fertilization× NPK concentration	11.616	2	5.808	18.894	0.000
Soil×fertilization×NPK concentration	1.010	2	0.505	1.643	0.214
Error	7.378	24	0.307		
Total	2653.290	35			

From table (4.4) we can test the following hypothesis:

1. H_0: there is no significant effect of soil on N P K concentration at level of significant ($\alpha = 0.05$).

   From the results in table (4.4) the p-value (sig) = 0.000 this value less than $\alpha = 0.05$ which means reject the hypothesis i.e. at level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of soil on N P K concentration.

2. H_0: there is no significant effect of fertilization on N P K concentration at level of significant ($\alpha = 0.05$).

   From the results in table (4.4) the p-value (sig) = 0.000 this value less than $\alpha = 0.05$ which means reject the hypothesis i.e at level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of fertilization on N P K concentration.

3. H_0: there is no significant effect of interaction between soil and fertilization on N P K concentration at level of significant ($\alpha = 0.05$).

   From the results in table (4.4) the p-value (sig)= 0.000 this value less than ($\alpha = 0.05$) which means reject the hypothesis i.e at level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of the interaction between soil and fertilization on N P K concentration.

4. H_0: there is no significant effect of interaction between soil and NPK concentration on NPK concentration at level of significant ($\alpha = 0.05$).

   From the results in table (4.4) the p-value (sig) = 0.141 this value greater than ($\alpha = 0.05$) which means don’t reject the hypothesis i.e. at level of significant ($\alpha = 0.05$) there is no sufficient evidence that there is an effect of the interaction between soil and NPK concentration on NPK concentration.

5. H_0: there is no significant effect of interaction between fertilization and NPK concentration on NPK concentration at level of signحificant ($\alpha = 0.05$).

   From the results in table (4.4) the p-value (sig)= 0.000 this value less than ($\alpha = 0.05$) which means reject the hypothesis i.e. at level of significant (α = 0.050 there is a strong evidence that there is an effect of the interaction between fertilization and NPK concentration on NPK concentration.

6. H_0: there is no significant effect of interaction between soil, fertilization and NPK concentration on NPK concentration at level of significant ($\alpha = 0.05$).

From the results in table (4.4) the p-value (sig)= 0.214 this value greater than ($\alpha = 0.05$) which means don’t reject the hypothesis i.e. at level of significant ($\alpha = 0.05$) there is no sufficient evidence that there is an effect of the interaction between soil, fertilization and NPK concentration on NPK concentration.

Table (4.5) shows the average concentration of essential nutrients for plants N P K for all treatment. The results showed that VS is the highest concentration, then decreases in V, and then decreases in CS, and C is the lowest concentration.

Table 4.5

The an average of N P K concentration in barley plant

Treatment	N%	P%	K (ppm)
VS	1.0997	0.071	22.888
V	0.7893	0.061	19.618
CS	0.6677	0.055	15.835
C	0.5327	0.052	14.07
Control	0.512	0.05	33.347

Nitrogen (N) concentration was significantly high in VS (1.0997%), and showed at a lower value in V, then in CS and thin in C (0.7893, 0.6677 and 0.5327%, respectively). Phosphorus (P) showed the highest concentration at VS and V (0.071 and0.061%, respectively) whereas the lowest concentration at CS and C (0.055 and 0.052%, respectively). Potassium (K) showed the highest concentration at VS and V (22.888 and 19.618 ppm, respectively) whereas the lowest concentration at CS and C (15.835 and 14.07 ppm, respectively).

Figure 4.2

Nitrogen (N) concentration in barley plant

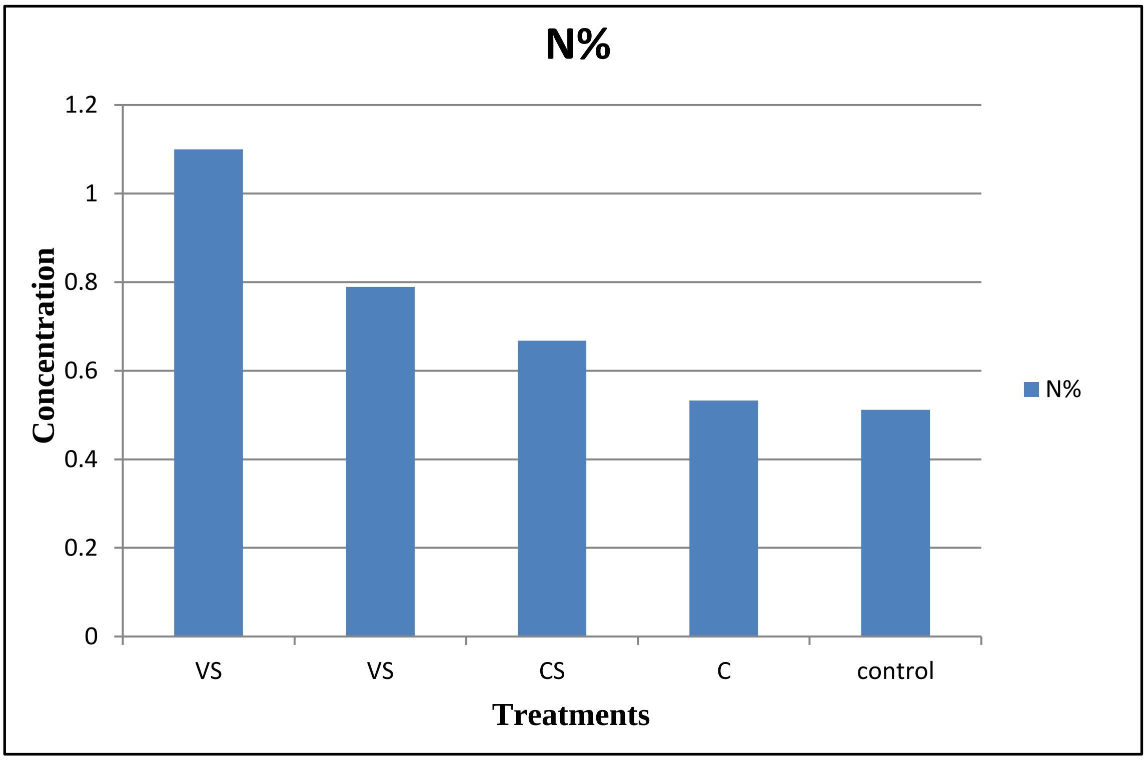


Figure 4.3

Phosphorous (p) concentration in barley plant

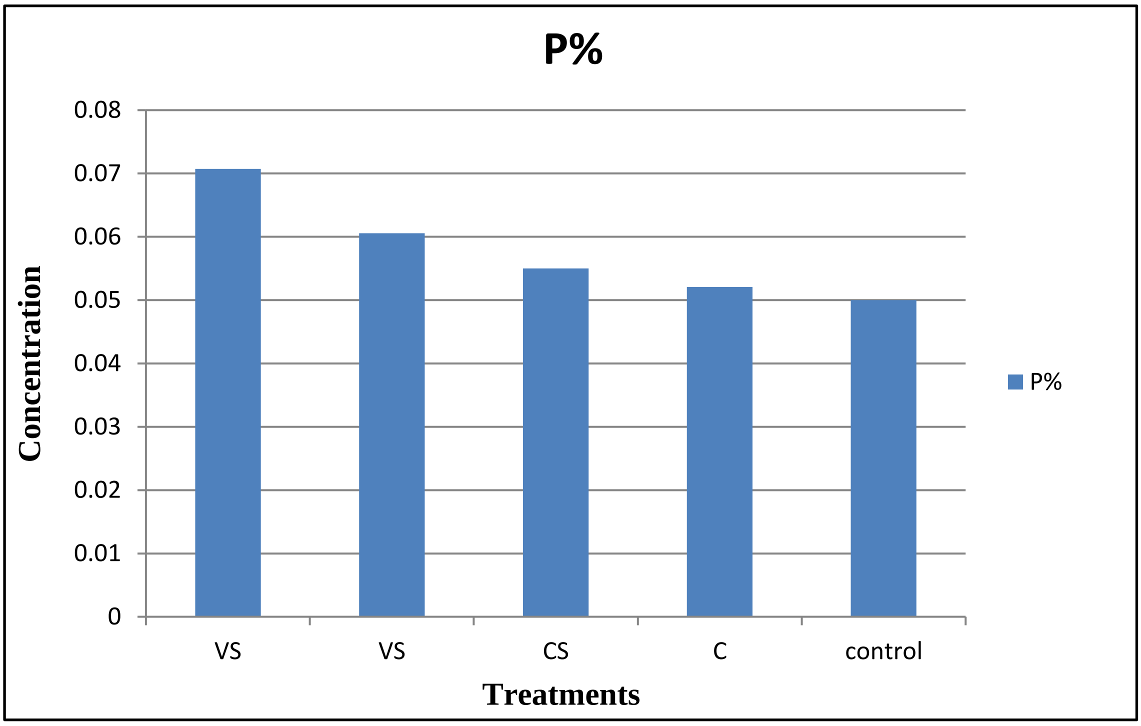
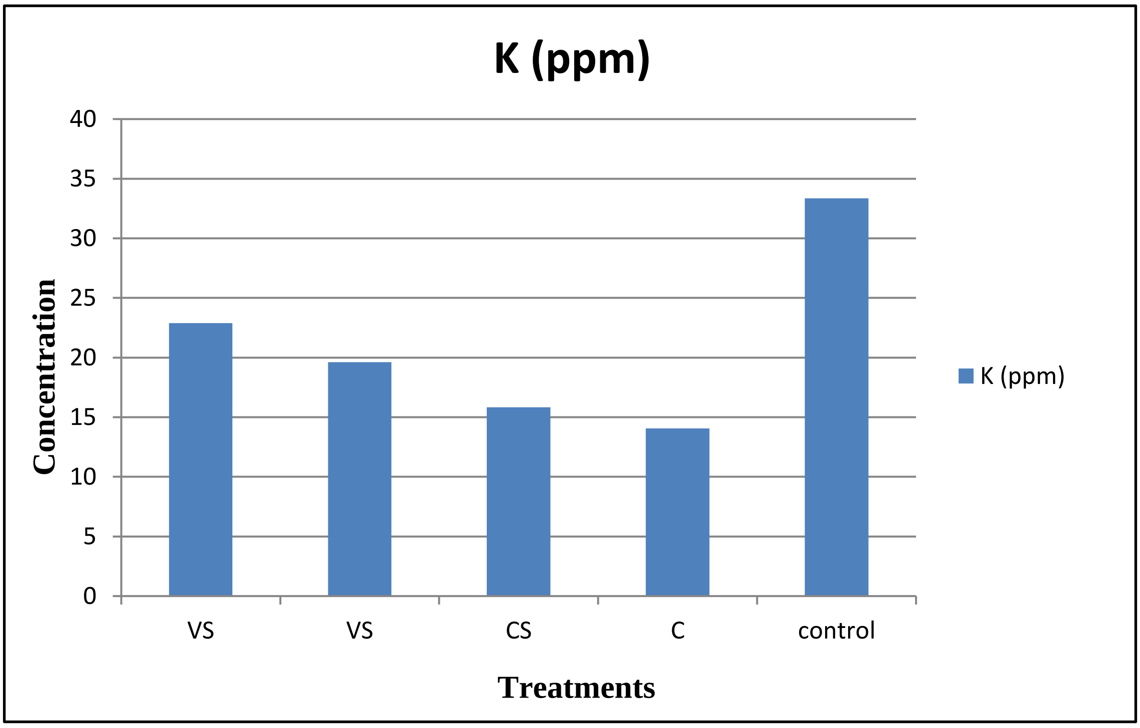


Figure 4.4

Potassium (K) concentration in parley plant



4.3 Morphological characteristics of barley plants

Differences appeared in the cultivated barley plants in terms of height, biomass weight and growth speed in the experiments.

In order to study the morphological characteristics of barley plants, multivariate analysis was used. The results of this test appear in the following table.

Table 4.6

Multivariate test

Effect		Value	F	Sig.
Intercept	Pillai's Trace	1.000	14573.173[b]	0.000
	Wilks' Lambda	0.000	14573.173[b]	0.000
	Hotelling's Trace	4163.764	14573.173[b]	0.000
	Roy's Largest Root	4163.764	14573.173[b]	0.000
Soil	Pillai's Trace	0.959	80.870[b]	0.000
	Wilks' Lambda	0.041	80.870[b]	0.000
	Hotelling's Trace	23.106	80.870[b]	0.000
	Roy's Largest Root	23.106	80.870[b]	0.000
Fertilization	Pillai's Trace	0.907	34.267[b]	0.000
	Wilks' Lambda	0.093	34.267[b]	0.000
	Hotelling's Trace	9.790	34.267[b]	0.000
	Roy's Largest Root	9.790	34.267[b]	0.000
Soil × fertilization	Pillai's Trace	0.622	5.765[b]	0.033
	Wilks' Lambda	0.378	5.765[b]	0.033
	Hotelling's Trace	1.647	5.765[b]	0.033
	Roy's Largest Root	1.647	5.765[b]	0.033

From table (4.6) all p-value (sig) less than ($\alpha = 0.05$) for all independent variables, and the interaction between the independent variables less than $\alpha = 0.05$, so we can expect that there is a significant effect of independent variables and the interaction on at least one of the dependent variables.

Table 4.7

Results of multivariate analysis

Source	Dependent variable	Sum of squares	df	Mean squares	F	Sig
Soil	Weight	752.083	1	752.083	111.420	0.000
	long	85.333	1	85.333	34.133	0.000
Fertilization	Weight	216.750	1	216.750	32.111	0.000
	long	12.000	1	12.000	4.800	0.060
Soil× fertilization	Weight	0.083	1	0.083	0.012	0.914
	long	5.333	1	5.333	2.133	0.182
Error	Weight	54.000	8	6.750		
	long	20.000	8	2.500		
Total	Weight	1022.917	11			
	long	122.667	11			

1. Soil

H_0: At level of significant $\alpha = 0.05$, there is no significant effect of soil on barley plant weight.

From the result of table (4.7) the p-value (sig) = 0.000 which is less than α=0.05 this lead us to reject the hypothesis which means at $\alpha = 0.05$ there is a strong evidence that there is a significant effect of soil on barley plant weight.

H_0: At level of significant $\alpha = 0.05$, there is no significant effect of soil on barley plant long

From the result of table (4.7) the p-value (sig) = 0.00 which is less than α=0.05 this lead us to reject the hypothesis which means at $\alpha = 0.05$ there is a strong evidence that there is a significant effect of soil on barley plant long.

2. Fertilizer

H_0: At level of significant $\alpha = 0.05$, there is no significant effect of fertilization on barley plant weight.

From the result of table (4.7) the p-value (sig) = 0.00 which is less than α=0.05 this lead us to reject the hypothesis which means at $\alpha = 0.05$ there is a strong evidence that there is a significant effect of fertilization on barley plant weight.

H_0: At level of significant α = 0.05, there is no significant effect of fertilization on barley plant long

From the result of table (4.7) the p-value (sig) = 0.06 which is greater than α=0.05 this lead us to don't reject the hypothesis which means at α = 0.05 there is no sufficient evidence that there is a significant effect of fertilization on barley plant long.

3. Soil × Fertilizer

H_0: At level of significant α = 0.05, there is no significant effect of the interaction between soil and fertilization on barley plant weight.

From the result of table (4.7) the p-value (sig) = 0.914 which is greater than α=0.05 this lead us to don't reject the hypothesis which means at α = 0.05 there is no sufficient evidence that there is a significant effect of interaction between soil and fertilization on barley plant weight.

H_0: At level of significant α = 0.05, there is no significant effect of the interaction between soil and fertilization on barley plant long

From the result of table (4.7) the p-value (sig) = 0.182 which is greater than α=0.05 this lead us to don't reject the hypothesis which means at α = 0.05 there is no sufficient evidence that there is a significant effect of the interaction between soil and fertilization on barley plant long.

The growth of barley was faster and stronger in vertisol soil than in calcareous soil, and it was faster and more prolific in sludge-fertilized soil than in non-fertilized soil.

The average plant height in the VS was 77 cm, while the V was 73.6 cm. In the CS, the average height was 70.3 cm, while C was 69.6 cm.

The average plant weight in the VS was 148.6 gm, while V was 140 gm. In the CS, the average weight was 133 gm, while the C was 124.3gm (Table A.1 in appendix A).

Figure 4.5

The average long in barley plant

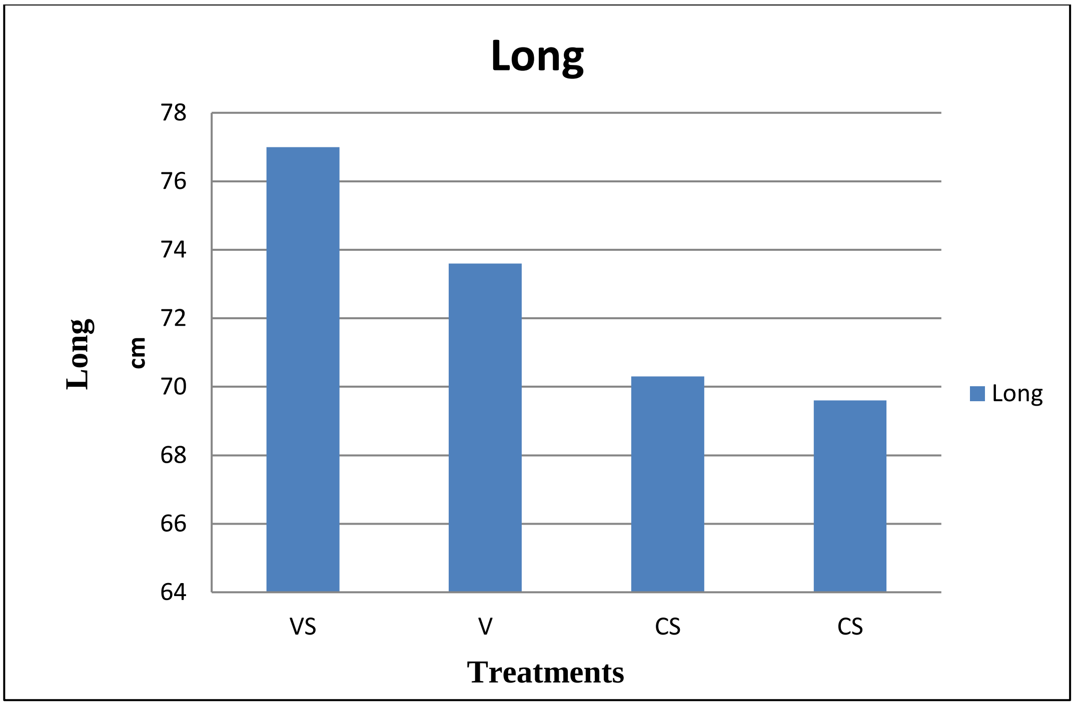
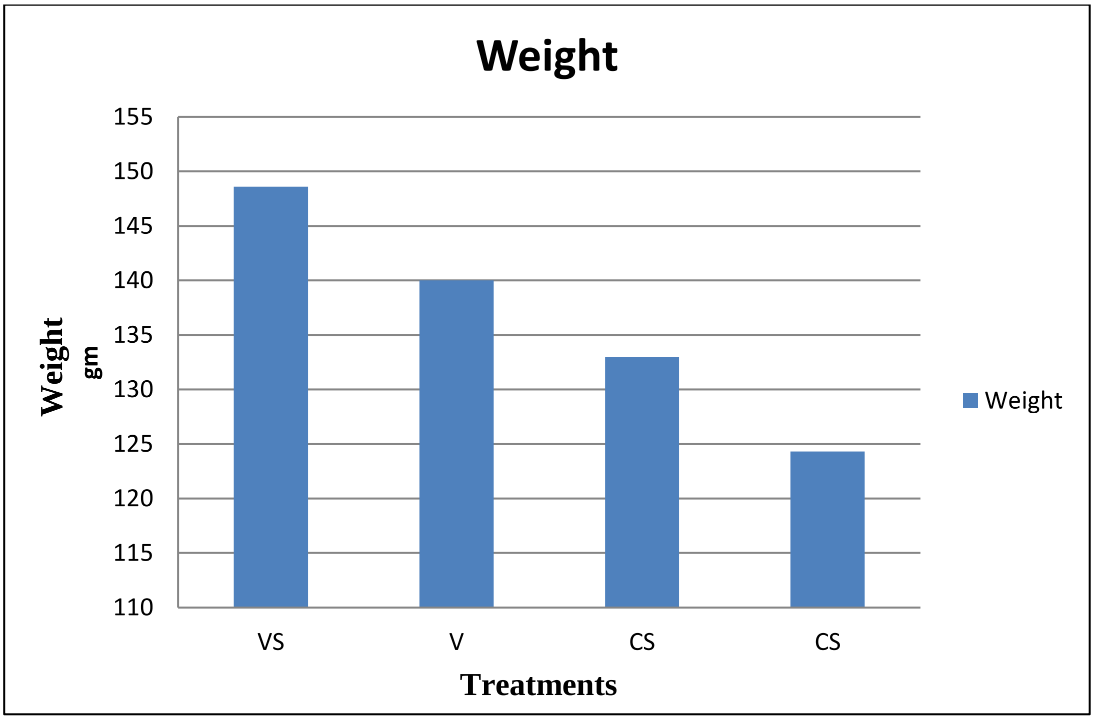


Figure 4.6

The average weight in barley plant



4.4 Heavy metal concentration in soil

In order to test whether there is an effect of heavy metal concentration in soil samples, the interaction between those variables was studied using univariate analysis. The results of this test appear in the table (A.2) (see Appendix A).

From table (A.2) (see Appendix A) we can test the following hypothesis:

1. H_0: there is no significant effect of layer soil on HM concentration in soil at level of significant ($\alpha = 0.05$).

   From the results in table (A.2) (see Appendix A) the p-value (sig) = 0.049 this value less than $\alpha = 0.05$ which means reject the hypothesis i.e at level of significant $\alpha = 0.05$ there is an evidence that there is an effect of layer soil on HM concentration in soil.

2. H_0: there is no significant effect of soil on HM concentration in soil at level of significant ($\alpha = 0.05$).

   From the results in table (A.2) (see Appendix A) the p-value (sig) = 0.000 this value less than ($\alpha = 0.05$) which means reject the hypothesis i.e. at level of significant ($\alpha = 0.05$) there is a strong evidence that there is an effect of soil on HM concentration in soil.

3. H_0: there is no significant effect of fertilization on HM concentration in soil at level of significant $\alpha = 0.05$

   From the results in table (A.2) (see Appendix A) the p-value (sig) = 0.708 this value greater than ($\alpha = 0.050$ which means don't reject the hypothesis i.e. at level of significant ($\alpha = 0.05$) there is no sufficient evidence that there is an effect of fertilization on HM concentration in soil.

To study the concentration of HM in soil layers.

For layer A (0-30 cm) the results of analysis appear in table (A.12) (see Appendix A).

From results of table (A.3) (see Appendix A):

1. H_0: there is no significant effect of soil on HM concentration on layer A at level of significant $\alpha = 0.05$

From the results in table (A.3) (see Appendix A) the p-value (sig) = 0.001 this value less than $\alpha = 0.05$ which means reject the hypothesis i.e at level of significant $\alpha = 0.05$ there is a strong evidence that there is an effect of soil on HM concentration on layer A.

2. H_0: there is no significant effect of fertilization on HM concentration on layer A at level of significant $\alpha = 0.05$

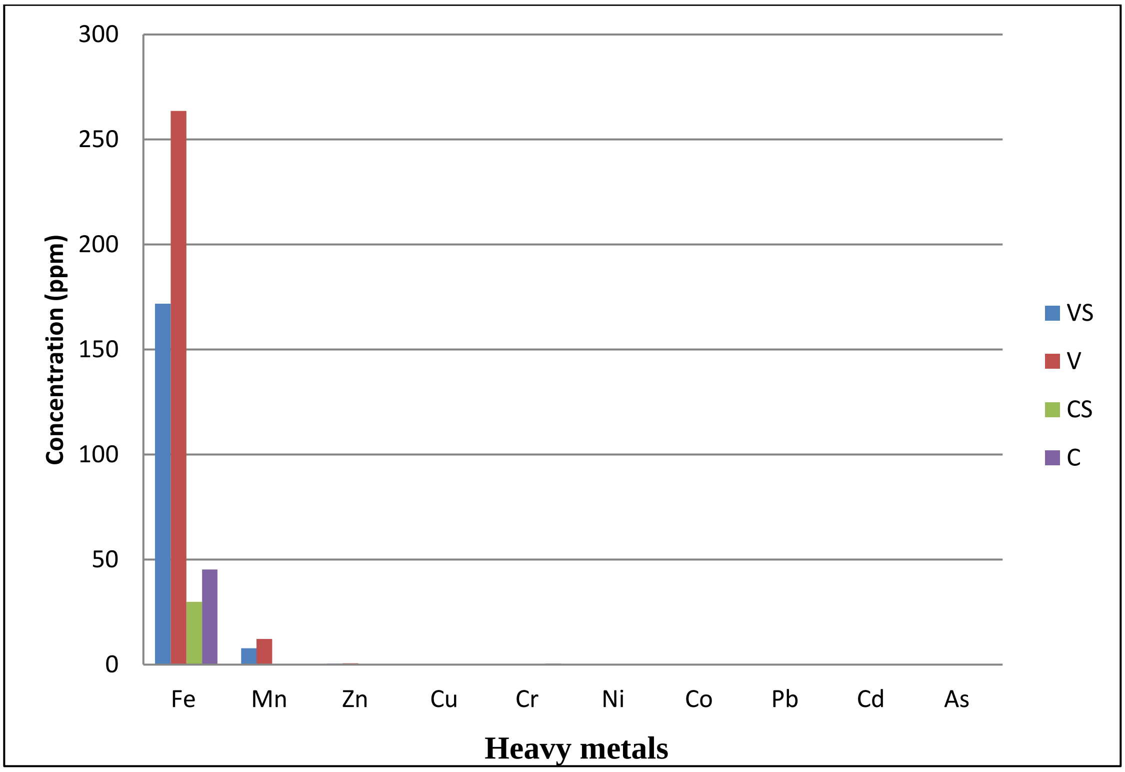
From the results in table (A.3) (see Appendix A) the p-value (sig) = 0.318 this value greater than ($\alpha = 0.05$) which means don't reject the hypothesis i.e. at level of significant ($\alpha = 0.05$) there is no sufficient evidence that there is an effect of fertilization on HM concentration on layer A.

Table (A.4) (see Appendix A) shows the concentration of heavy metal in soil layer A (0-30 cm) after the experiment. Concentration of Fe, Mn, Zn, Cu, Ni, Co and Pb was higher in V treatment, whereas the concentration of Cr, Cd and As was higher in C treatment.

Ferrous (Fe) concentration was significantly high in V and VS (263.672 and 171.761ppm, respectively), in contrast treatment C and CS (45.2435 and 29.8403 ppm, respectively). Manganese (Mn) concentration was significantly high in V and VS (12.187 and 7.729 ppm, respectively), in contrast treatment C and CS (0.151 and 0.098 ppm, respectively). Zinc (Zn) concentration was significantly high in V and VS (0.642 and 0.460 ppm, respectively), in contrast treatment CS and C (0.219 and 0.162 ppm, respectively). Copper (Cu) concentration showed the highest value at V and VS (0.224 and 0.159 ppm, respectively), whereas the lowest at C and CS (0.108 and 0.078 ppm, respectively). C treatment showed the highest concentration at Chromium (Cr) (0.450 ppm), thin in V (0.378 ppm) and thin in CS (0.320 ppm), whereas the lowest at VS (0.238 ppm). Nickel (Ni) concentration showed the highest value at V (0.351 ppm), thin in C (0.260 ppm) and thin in VS (0.238 ppm), whereas the lowest at CS (0.184 ppm). Cobalt (Co) concentration showed the highest value at V and VS (0.179 and 0.115 ppm, respectively), whereas the lowest at C and CS (0.014 and 0.009 ppm, respectively). Lead (Pb) concentration showed the highest value at V and VS (0.104 and 0.086 ppm, respectively), whereas the lowest at C and CS (0.015 and 0.013 ppm, respectively). Cadmium (Cd) no differences observed between all treatments. Arsenic (As) showed the highest concentration C (0.027 ppm), thin in CS (0.018 ppm) and thin in V (0.013 ppm) whereas the lowest at VS (0.009 ppm).

Figure 4.7

HM concentration in soil layer A (0-30 cm)



a. For layer B (30-60 cm) the results of analysis appear in table (A.5) (see appendix A).

   From the result of table (A.5):

1. H_0: there is no significant effect of soil on HM concentration on layer B at level of significant ($\alpha = 0.05$).

   From the results in table (A.5) (se Appendix A) the p-value (sig) = 0.001 this value less than α = 0.05 which means reject the hypothesis i.e. at level of significant (α = 0.05) there is a strong evidence that there is an effect of soil on HM concentration on layer B.

2. H_0: there is no significant effect of fertilization on HM concentration on layer B at level of significant ($\alpha = 0.05$).

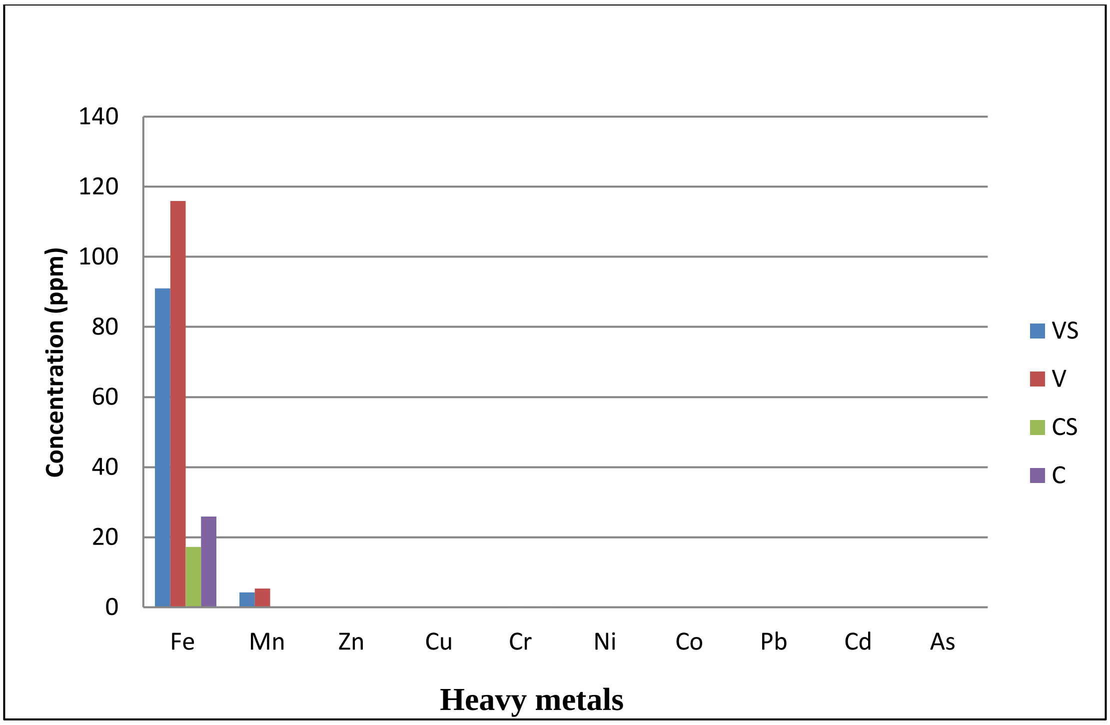
From the results in table (A.5) (see Appendix A) the p-value (sig) = 0.476 this value greater than ($\alpha = 0.05$) which means don't reject the hypothesis i.e. at level of significant ($\alpha = 0.05$) there is no sufficient evidence that there is an effect of fertilization on HM concentration on layer B.

Table (A.6) (see appendix A) shows the concentration of heavy metal in soil layer B (30-60 cm) after the experiment. Concentration of Fe, Mn, Zn, Cu, Co and Pb was higher in V treatment, whereas the concentration of Cr, Ni, Cd and As was higher in C treatment.

Ferrous (Fe) concentration was significantly high in V and VS (115.921 and 91.009 ppm, respectively), in contrast treatment C and CS (25.865 and 17.193 ppm, respectively). Manganese (Mn) concentration was significantly high in V and VS (5.372 and 4.274 ppm, respectively), in contrast treatment C and CS (0.110 and 0.042 ppm, respectively). Zinc (Zn) concentration was significantly high in V and VS (0.185 and 0.134 ppm, respectively), in contrast treatment C and CS (0.086 and 0.049 ppm, respectively). Copper (Cu) concentration showed the highest value at V and VS (0.097 and 0.060 ppm, respectively), whereas the lowest at C and CS (0.041 and 0.027 ppm, respectively). Chromium (Cr) concentration showed the highest value at C and V (0.265 and 0.214 ppm, respectively), whereas the lowest at CS and VS (0.181 and 0.170 ppm, respectively). Nickel (Ni) concentration showed the highest value at C and V (0.156 and 0.145 ppm, respectively), whereas the lowest at VS and CS (0.111 and 0.105 ppm, respectively). Cobalt (Co) concentration showed the highest value at V and VS (0.075 and 0.057 ppm, respectively), whereas the lowest at C and CS (0.010 and 0.004 ppm, respectively). Lead (Pb) concentration showed the highest value at V and VS (0.081 and 0.063 ppm, respectively), whereas the lowest at C and CS (0.006 and 0.005 ppm, respectively). Cadmium (Cd) no differences observed between all treatments. Arsenic (As) showed the highest concentration C (0.013 ppm), whereas no differences observed between VS, V and CS.

Figure 4.8

HM concentration in soil layer B (30-60 cm)



b. For layer C (60-100 cm) the results of analysis appear in table (A.7) (see Appendix A).

From results of table (A.7):

1. H_0: there is no significant effect of soil on HM concentration on layer C at level of significant ($\alpha = 0.05$).

   From the results in table (A.7) in Appendix A) the p-value (sig) = 0.116 this value less than ($\alpha = 0.05$) which means don't reject the hypothesis i.e at level of significant ($\alpha = 0.05$) there is no evidence that there is an effect of soil on HM concentration on layer C.

2) H_0: there is no significant effect of fertilization on HM concentration on layer C at level of significant ($\alpha = 0.05$).

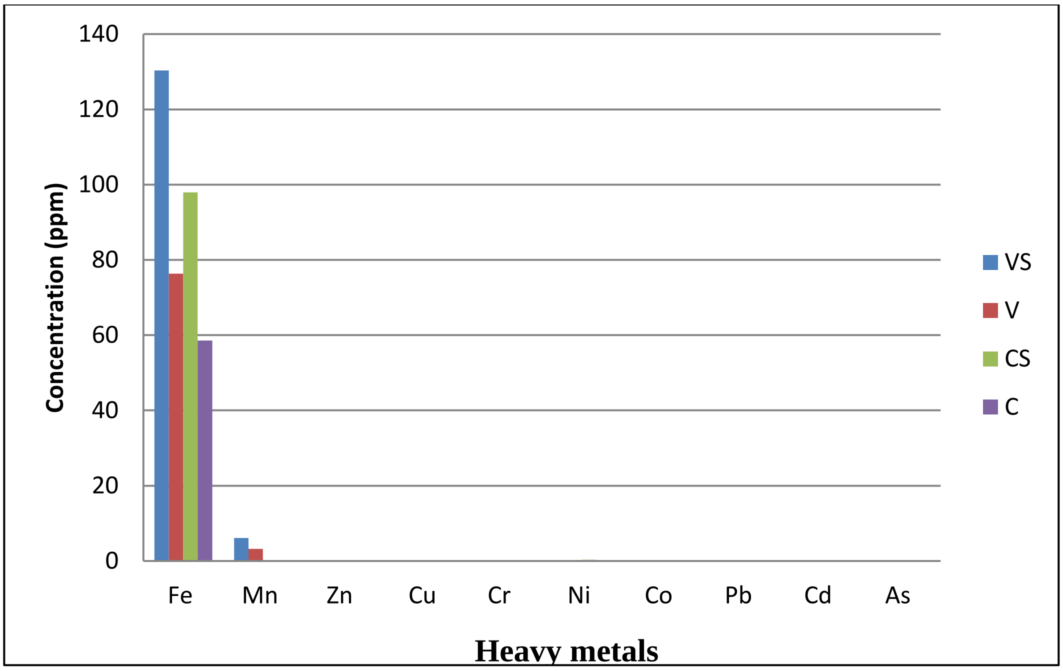
   From the results in table (A.7) in Appendix A) the p-value (sig) = 0.011 this value less than ($\alpha = 0.05$) which means reject the hypothesis i.e. at level of significant ($\alpha = 0.05$) there is a sufficient evidence that there is an effect of fertilization on HM concentration on layer C.

Table (A.8) (see Appendix A) shows the concentration of heavy metal in soil layer C (60-100 cm) after the experiment. Concentration of Fe, Mn, Zn, Cu, Cr, Co and Pb was higher in VS treatment, whereas the concentration of Cd and As was higher in C treatment and higher concentration of Ni in CS treatment.

Ferrous (Fe) concentration was significantly high in VS and V (130.311 and 76.366 ppm, respectively), in contrast treatment CS and C (97.904 and 58.537 ppm, respectively). Manganese (Mn) concentration was significantly high in VS and V (6.080 and 3.239 ppm, respectively), in contrast treatment C and CS (0.133 and 0.062 ppm, respectively). Zinc (Zn) concentration was significantly high in VS (0.148ppm) and the concentration was equal in V and CS (0.091ppm), whereas the lowest in V (0.049 ppm). Copper (Cu) concentration showed the highest value at VS (0.072 ppm), then in CS (0.039 ppm), then in C (0.035 ppm) and then in V(0.033 ppm). Chromium (Cr) concentration showed the highest value at VS and CS (0.022 and 0.214 ppm, respectively), whereas the lowest at C and V (0.174 and 0.155 ppm, respectively). Nickel (Ni) concentration showed the highest value at CS and C (0.343 and 0.265 ppm, respectively), whereas the lowest at VS and V (0.186 and 0.086 ppm, respectively). Cobalt (Co) concentration showed the highest value at VS and V (0.092 and 0.043 ppm, respectively), whereas the lowest at CS and C (0.020 and 0.017 ppm, respectively). Lead (Pb) concentration showed the highest value at VS and V (0.045 and 0.036 ppm, respectively), and the concentration was equal in CS and C (0.004 ppm). Cadmium (Cd) concentration showed the highest value at C (0.001ppm), whereas no differences observed between VS, V and CS. Arsenic (As) showed the highest concentration at C and CS (0.020 and 0.012 ppm, respectively), whereas the lowest at VS and V (0.003 and 0.001 ppm, respectively).

Figure 4.9

HM concentration in soil layer C (60-100 cm)



4.4.1 Movement of heavy metals in the soil

a. Fe

Iron appeared in high concentration in layer A for all treatments and then decreased in layers B and C in equal concentration (Figure B.3 in Appendix B).

b. Mn

In the VS treatment, manganese appeared in a higher concentration in layer A, decreased in layer B, and then increased in layer C. As for treatment V, the concentration was higher in layer A, then decreased in layer B, and decreased to a lesser extent in layer C. In CS and C treatments, the concentration was highest in layer A, decreased in layer B, and then increased in layer C (Figure B.4 in appendix B).

c. Zn

In VS, CS, and C treatments, the concentration was higher in layer A, decreased significantly in layer B, and then slightly increased in layer C. In treatment V, the concentration appeared higher in layer A, decreased in layer B, and decreased in layer C (Figure B.5 in Appendix B).

d. Cu

In treatments VS and CS the copper concentration appeared higher in layer A, decreased in layer B, and then increased slightly in layer C, but in treatments V and C, the concentration was higher in layer A, decreased in layer B, then decreased in layer C (Figure B.6 in Appendix B).

e. Cr

In treatments VS and CS the concentration of chromium appeared higher in layer A, decreased in layer B, then increased slightly in layer C, but in treatments V and C, the concentration was higher in layer A, decreased in layer B, then decreased in layer C (Figure B.7 in Appendix B).

f. Ni

In treatment VS, the concentration of nickel appeared higher in layer A, then decreased in layer B, then increased in layer C. As for treatment V, the concentration was higher in layer A, decreased in layer B, and then decreased also in layer C. In treatment CS, the concentration appeared higher in layer C, decreased in layer A, and then decreased also in layer B. In treatment C, the higher concentration appeared equally in layers A and C, and the lower concentration in layer B (Figure B.8 in Appendix B).

g. Co

In treatment VS, the cobalt concentration was higher in layer A and lower in layer C, and the lowest concentration was in layer B. In treatment V, the concentration was highest in layer A, decreased in layer B, and then decreased in layer C. In treatment CS, the concentration was higher in layer C, decreased in layer A, and decreased in layer B. In experiment C, the concentration was equal in all layers (Figure B.9 in Appendix B).

h. Pb

In treatments VS and V, the lead concentration was higher in layer A, decreased in layer B, and decreased in layer C. As for treatments CS and C, the concentration was higher in layer A and decreased equally in layers B and C (Figure B.10 in Appendix B).

i. Cd

In treatments VS, V and CS, the cadmium concentration was higher in layer A, decreased in layer B, and then decreased in layer C. In treatment C, the concentration was higher in layer A, decreased in layer C, and then decreased in layer B (Figure B.11 in Appendix B).

j. As

In the treatments of VS and V, the concentration of arsenic was higher in layer A, decreased in layer B, and decreased in layer C. In treatment Y and Z, the concentration was highest in layer A, then in layer X, and the lowest concentration in layer B (Figure B.12 in Appendix B).

4.5 Discussion

4.5.1 Heavy metal concentration in barley plant

The concentration of plant nutrients Fe, Mn, Zn, and Cu in the barley plants that were fertilized with sludge and irrigated with treated wastewater was higher than the control sample and was within the standards table (A.9) (see Appendix A). Also, other elements that could cause toxicity, such as Cr, Ni, Co, Pb, Cd, and As, had low concentrations in experimental applications.

The results show that fertilization with sludge has improved the nutrition of barley plants and contributed to providing the plants with the necessary nutrients without risk.

Vertsols soil was more efficient in transferring nutrients to barley plants compared to calcareous soil, which shows that vertisols soil is more fertile and suitable for growing barley than calcareous soil.

4.5.2 N P K concentration in barley plant

The concentration of nitrogen and phosphorus in barley plants fertilized with sludge and irrigated with treated wastewater was higher than in the control sample, while the concentration of potassium was higher in the control sample.

Vertsols soil was more efficient in providing barley plants with essential nutrients than calcareous soil. Fertilization with sludge also improved soil fertility and its ability to supply plants with nutrients, as the concentration of nutrients in fertilized plants was higher than in non-fertilized plants for each soil type.

According to the results, Vertsols soil is more fertile and capable of nourishing barley plants, and its response to fertilizer is greater compared to calcareous soil.

4.5.3 Morphological characteristics of barley plant

During the study, growth in Vertsols soil was faster than in calcareous soil, and in soil fertilized with sludge, the growth rate was faster for each soil type. After the end of the experiment, the weights and lengths of barley plants grown in Vertsols soil were greater than those grown in calcareous soil.

The effect of fertilization with sludge was clear in the results, as the fertilized barley plants had a greater weight and length than non-fertilized plants for each soil type.

From the results, it is clear that Vertsols soil is more suitable for growing barley, and fertilizing with sludge improves soil fertility.

4.5.4 Heavy metal concentration in soil

The experiment revealed that the concentration of Fe and Mn nutrients in Vertsols soil was higher than the untreated soil sample after fertilizing with sludge and irrigation with treated wastewater. The concentration of Zn and Cu in layer A was higher than in soil not treated with sludge and treated wastewater, and it decreased in layers B and C.

Also in Vertsols soil, the concentration of elements Cr, Ni, Co, Pb, Cd, and As increased by small percentages in samples treated with sludge and treated wastewater compared to the untreated soil sample.

The concentration of Fe and Mn elements in Vertsols soil in layer C was higher than in layer B, which shows the effect of experimental applications on the transfer of elements Fe and Mn to the bottom layer.

In calcareous soil, the concentration of Fe in soil samples treated with sludge and treated wastewater was lower than in untreated soil, meaning that the experimental applications led to the leaching of Fe from the soil, as its concentration in layer C was greater than in layers A and B.

The concentration of Mn and Zn in layer A was greater in the calcareous soil samples treated with sludge and treated wastewater than in the untreated sample. As for Cu and Cr elements, their concentration was highest in layer A and decreased in layers B and C for calcareous soil samples treated with sludge and treated wastewater. After treating the calcareous soil with sludge and treated wastewater, the concentration of Ni and Co decreased.

Chapter Five

Conclusions and Recommendations

5.1 Conclusion

The main objective of this research was to study the effect of sludge fertilization and irrigation with treated wastewater on the accumulation of heavy metals in barley plant, vertisols and calcareous soils. The following can be concluded from the results of field measurements:

1. The use of treated wastewater for irrigation and sludge fertilization increased the concentration of heavy metals in the barley plant, especially elements associated with plant nutrients like Fe, Mn, Zn, and Cu.
2. The absorption of heavy metals by barley plants is influenced by the interplay between the type of soil and sludge fertilizer.
3. The concentration of vital plant nutrients N P K varies depending on the type of soil, with plants cultivated in Vertsols soil having a higher concentration than those grown in calcareous soil.
4. Barley plants fertilized with sludge showed higher concentrations of the major plant nutrients N P K than plants not fertilized in the same type of soil, indicating that sludge fertilization increased plant nutrient concentration.
5. The height and weight of barley plants grown in Vertsols soil were greater than those grown in calcareous soil.
6. Sludge fertilization enhanced the weight of the barley plant, with the fertilized plants weighing more for the same type of soil than the unfertilized ones.
7. At a depth of 60 cm, the type of soil has an impact on the concentration of heavy metals in the soil; however, this impact is lessened as depth increases.
8. Sludge fertilization had no effect on the concentration of heavy metals in soil layers that were 60 cm deep, but it increased the concentration of heavy metals in soil layers that were 60–100 cm deep, indicating that sludge fertilization enhances the movement of metals and their infiltration into the lower layers.

9. Because of its higher quantity of plant nutrients and higher capacity for nutrient absorption, vertisols soil is more fertile than calcareous soil.

5.2 Recommendations

1. It is possible to use sludge and treated wastewater produced from the Jenin treatment plant for fertilizer and irrigation on the barley plant because the results of heavy metal concentrations in the plant and soil were within the permissible limits, with the need to continue long-term studies to monitor the concentration of heavy metals.
2. Sludge is a source of plant nutrients, including major elements like nitrogen, phosphorous, and potassium, as well as minor elements like iron, zinc, and manganese, and can be utilized in composting.
3. The type of soil affects the quality of production. It is possible to improve the properties and quality of the soil through fertilization with sludge and irrigation with treated wastewater.
4. Fertilization with sludge should be in limited quantities and at frequent intervals because sludge helps in the movement of minerals and their seepage into the lower layers.

List of abbreviations

Abbreviation	Meaning
ANOVA	Analysis of variance
CEC	Cation exchange capacity
FW	Fresh water
HM	Heavy metal
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
NARC	National Agriculture Research Center
PbS	Lead sulfide
PVC	Polyvinyl chloride
SWW	Secondary treated wastewater
TWW	Tertiary treated wastewater

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Appendices

Appendix A

Tables

Table A.1

The an average of long and weight in barley plant

Treatment	Long	Weight
VS	77	148.6
V	73.6	140
CS	70.3	133
CS	69.6	124.3

Table A.2

Results of univariate analysis of heavy metal concentration in soil

Source	Sum of squares	df	Mean squares	F	Sig
Layer	2836.492	2	1418.246	3.042	0.049
Soil	9436.359	1	9436.359	20.243	0.000
Heavy metal	281622.809	9	31291.423	67.126	0.000
Fertilization	65.464	1	65.464	0.140	0.708
Error	161292.035	346	466.162		
Total	455253.158	359			

Table A.3

Result of univariate analysis of HM concentration in soil layer A

Source	Sum of squares	df	Mean squares	F	Sig
Heavy metal	174247.043	9	19360.783	20.516	0.000
Fertilization	950.547	1	950.547	1.007	0.318
Soil	10907.964	1	10907.964	11.559	0.001
Error	101921.071	108	943.714		
Total	288026.624	119			

Table A.4

The average of HM concentration in soil layer A (0-30 cm)

Treatment	Fe	Mn	Zn	Cu	Cr	Ni	Co	Pb	Cd	As
VS	171.761	7.72901	0.46053	0.159584	0.26961	0.23866	0.11533	0.08647	0.00184	0.00978
V	263.672	12.187	0.64207	0.224772	0.37868	0.3519	0.17997	0.10444	0.00253	0.01328
CS	29.8403	0.09865	0.21914	0.078247	0.32004	0.18429	0.00905	0.01301	0.00198	0.01857
C	45.2435	0.15146	0.16274	0.108315	0.45061	0.26057	0.01485	0.01596	0.00291	0.02796

Table A.5

Result of univariate analysis of HM concentration in soil layer B

Source	Sum of squares	df	Mean squares	F	Sig
Heavy metal	41789.209	9	4643.245	25.645	0.000
Fertilization	92.692	1	92.692	0.512	0.476
Soil	2265.709	1	2265.709	12.513	0.001
Error	19554.640	108	181.061		
Total	63702.250	119			

Table A.6

The average of HM concentration in soil layer B (30-60 cm)

Treatment	Fe	Mn	Zn	Cu	Cr	Ni	Co	Pb	Cd	As
VS	91.0094	4.2746	0.13441	0.060767	0.17014	0.11142	0.05714	0.06376	0.00117	0.00403
V	115.921	5.3723	0.18596	0.097874	0.21421	0.14536	0.07508	0.08117	0.00145	0.00581
CS	17.1931	0.04279	0.04931	0.027042	0.18183	0.10513	0.00413	0.00526	0.00108	0.00821
C	25.8657	0.11005	0.0865	0.041806	0.26554	0.15675	0.01052	0.00686	0.00156	0.0133

Table A.7

Result of univariate analysis of HM concentration in soil layer C

Source	Sum of squares	df	Mean squares	F	Sig
Heavy metal	88405.773	9	9822.864	93.718	0.000
Fertilization	699.316	1	699.316	6.672	0.011
Soil	262.840	1	262.840	2.508	0.116
Error	11319.862	108	104.814		
Total	100687.792	119			

Table A.8

The average of HM concentration in soil layer C (60-100 cm)

Treatment	Fe	Mn	Zn	Cu	Cr	Ni	Co	Pb	Cd	As
VS	130.311	6.08052	0.14879	0.07247	0.22026	0.18607	0.09272	0.04507	0.00062	0.00362
V	76.3669	3.23925	0.04946	0.033469	0.15577	0.08664	0.04362	0.03634	0.00043	0.00139
CS	97.904	0.06204	0.09185	0.039593	0.21428	0.34367	0.0209	0.00484	0.00097	0.01228
C	58.5374	0.13338	0.09154	0.035244	0.17446	0.26552	0.0172	0.00419	0.00188	0.0202

Table A.9

WHO permissible limits for heavy metal in plant and soil

Elements	Target value of soil (mg/kg)	Permissible value of plant (mg/kg)
Cd	0.8	0.02
Zn	50	0.60
Cu	36	10
Cr	100	1.30
Pb	85	2
Ni	35	10

Appendix B

Figures

Figure B.1

PVC Soil Column

Figure B.2

Design of experiment

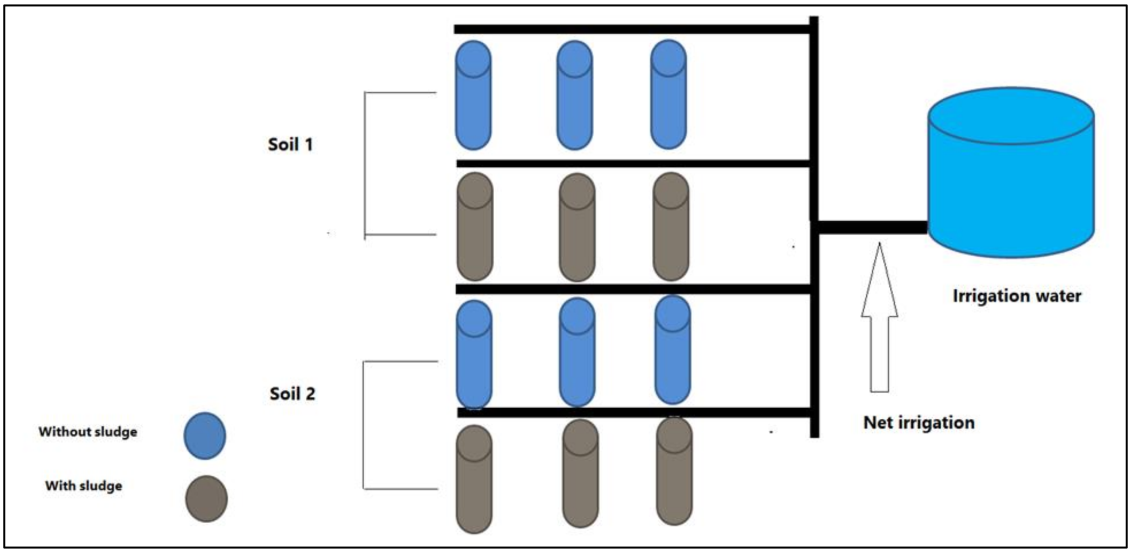

Figure B.3

Fe concentration in layer A B C

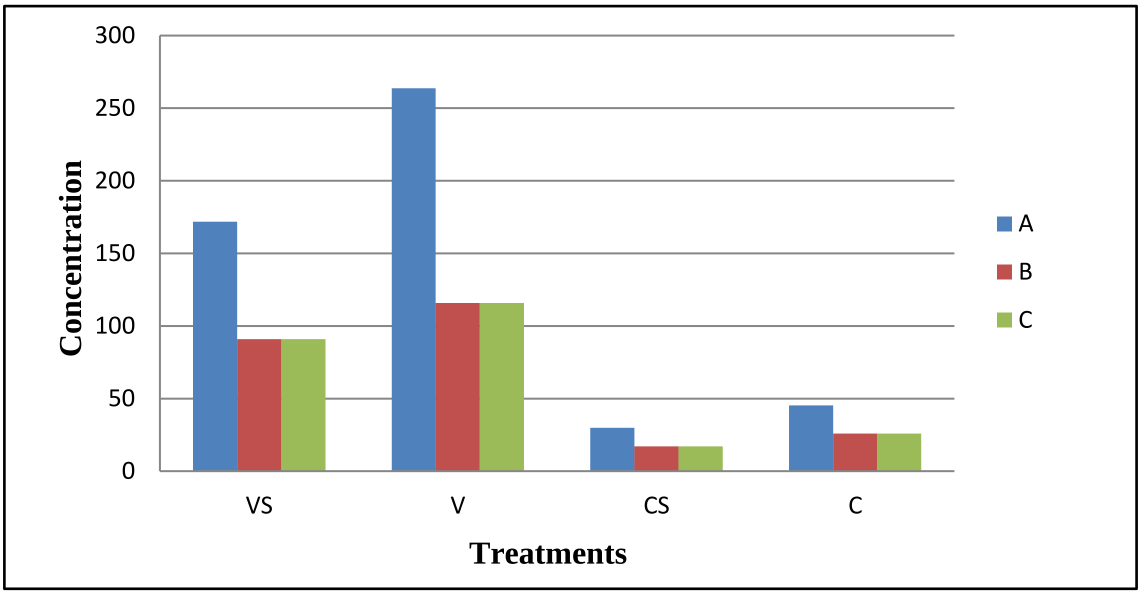

Figure B.4

Mn concentration in layer A B C

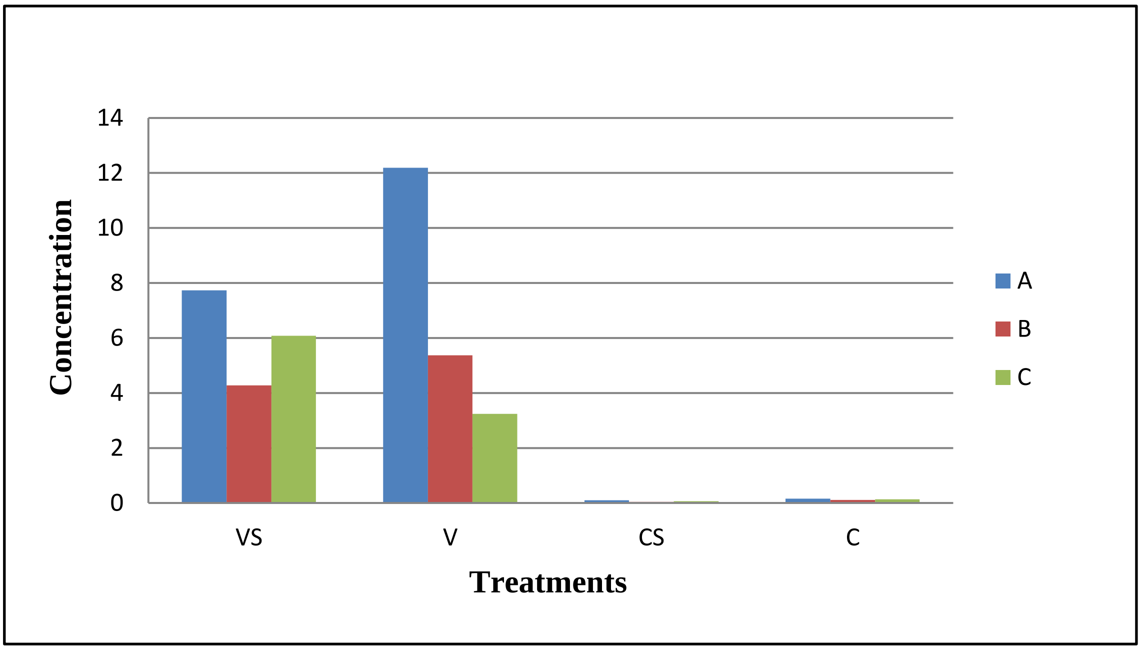

Figure B.5

Zn concentration in layer A B C

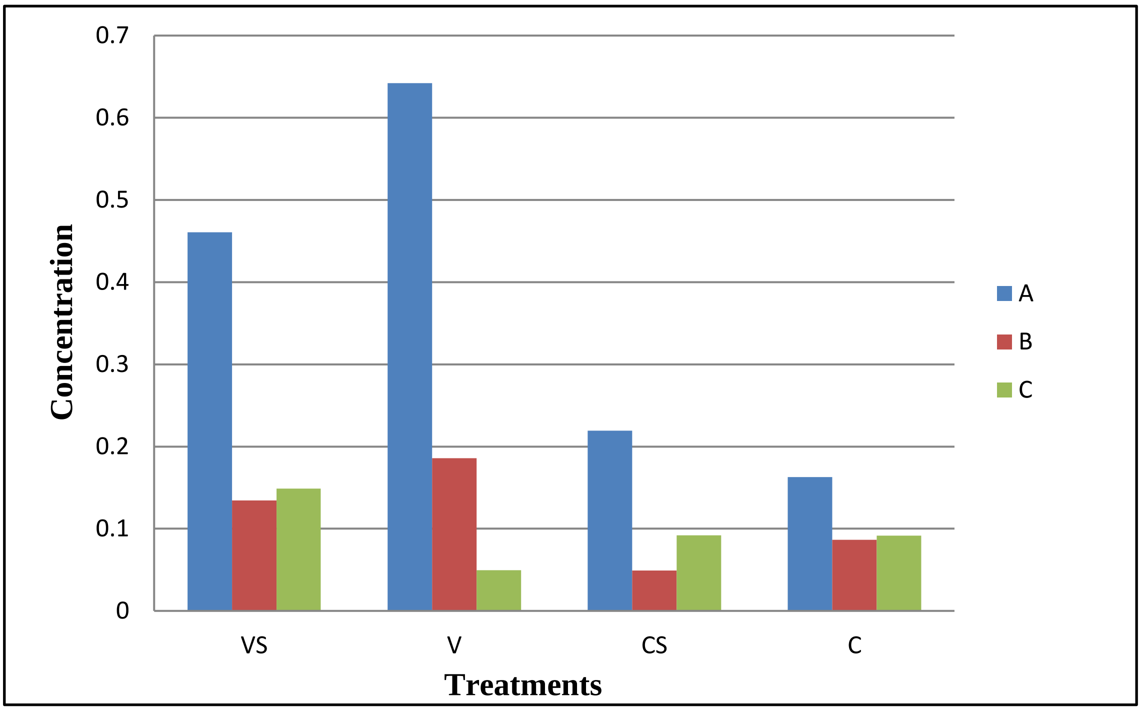

Figure B.6

Cu concentration in layer A B C

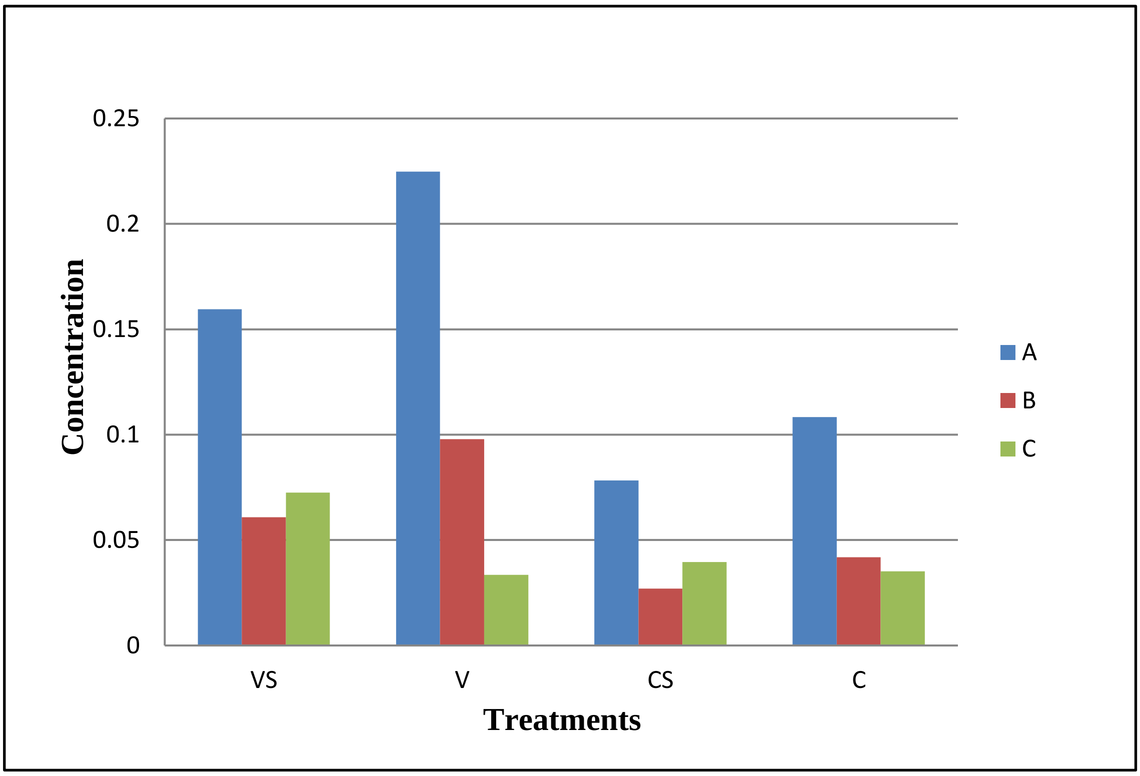

Figure B.7

Cr concentration in layer A B C

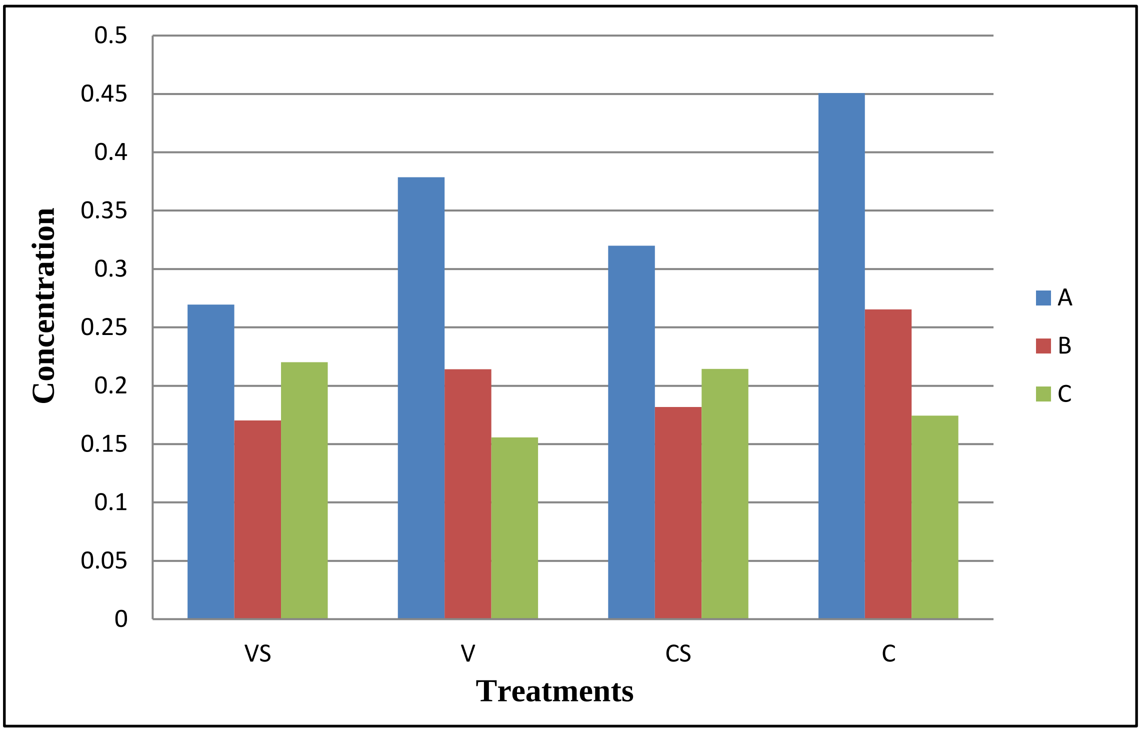

Figure B.8

Ni concentration in layer A B C

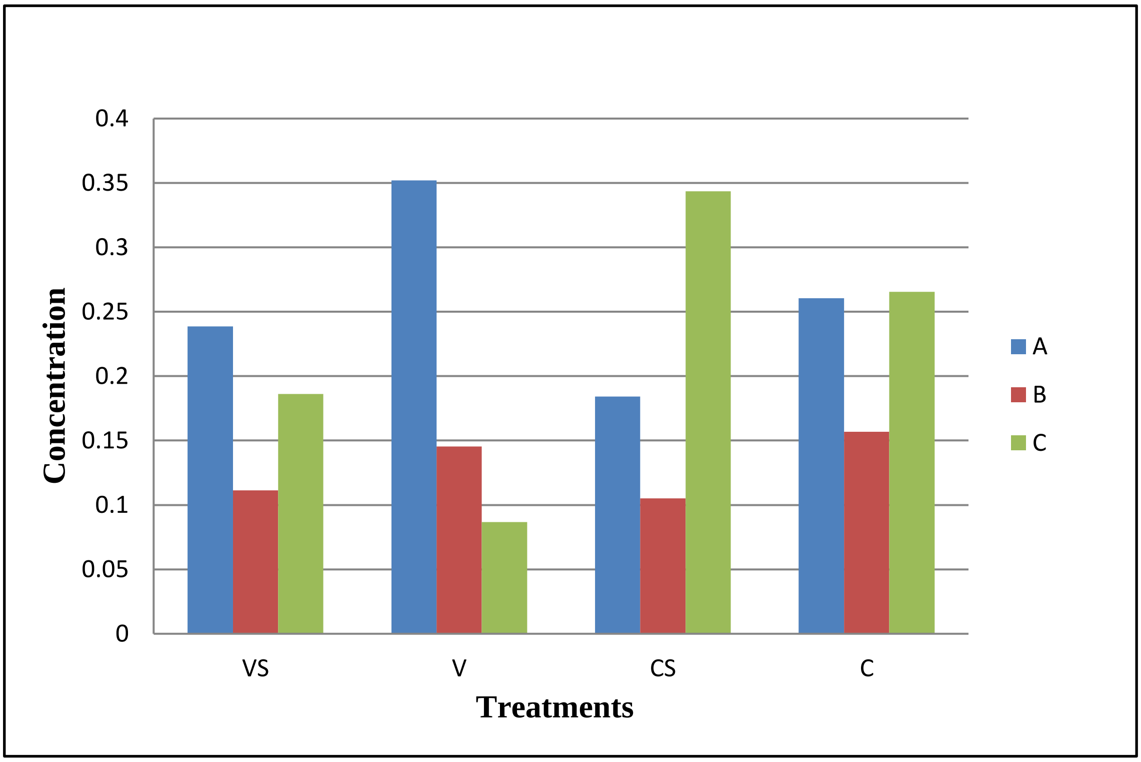

Figure B.9

Co concentration in layer A B C

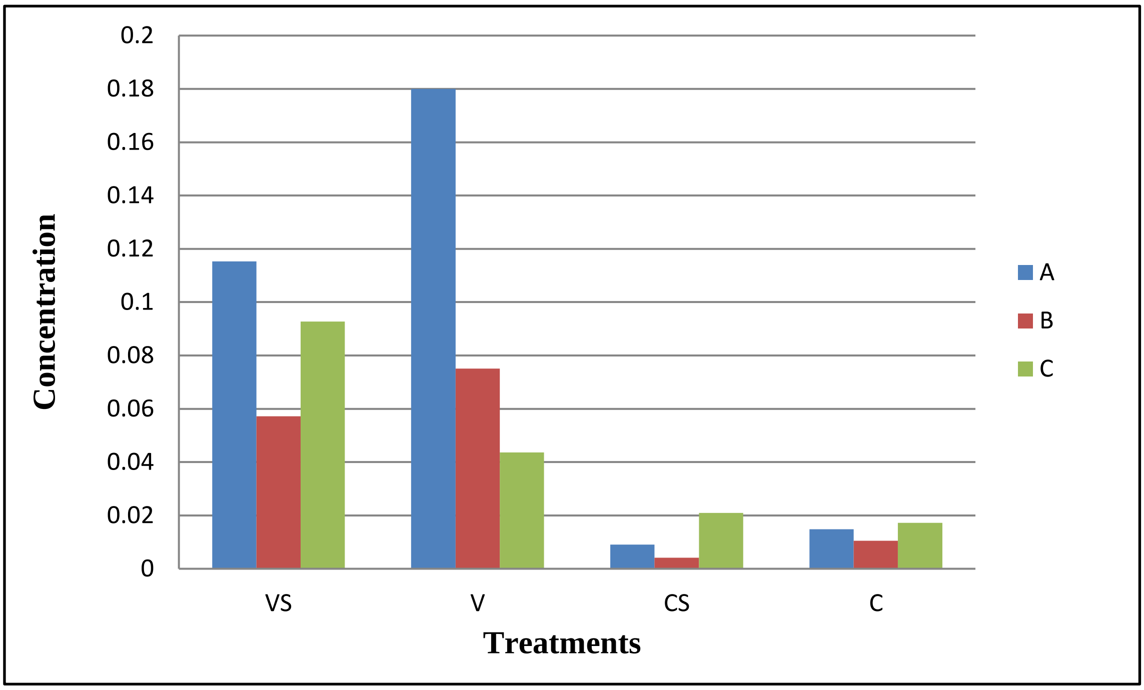

Figure B.10

Pb concentration in layer A B C

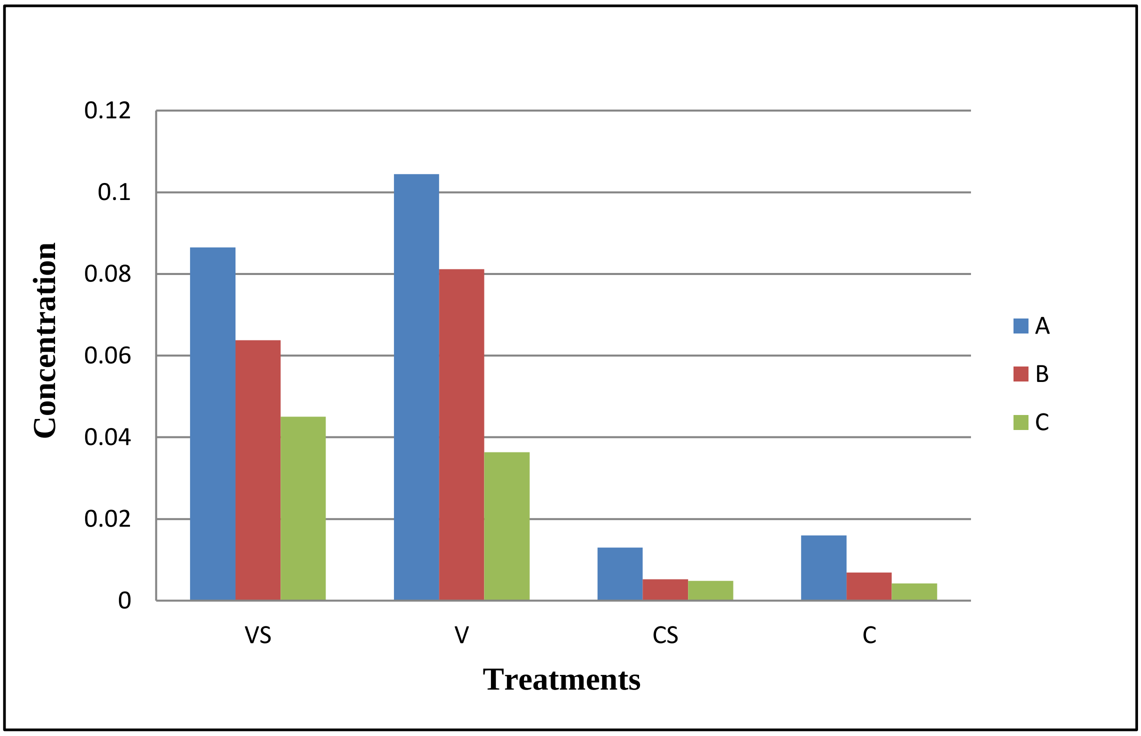

Figure B.11

Cd concentration in layer A B C

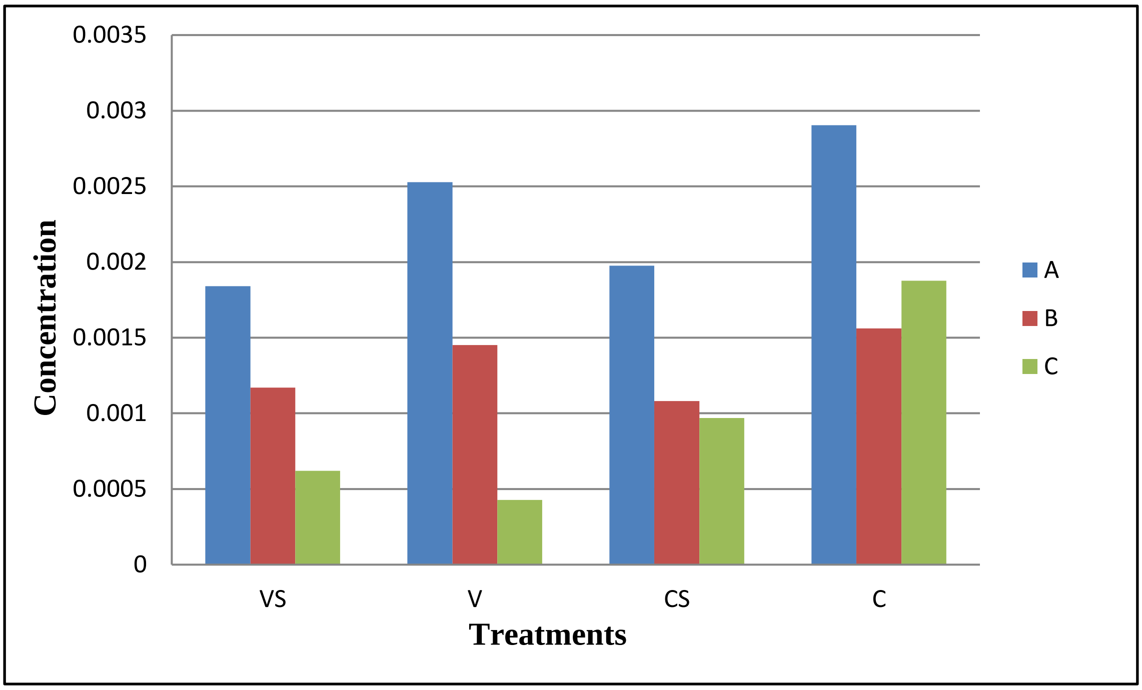

Figure B.12

As concentration in layer A B C

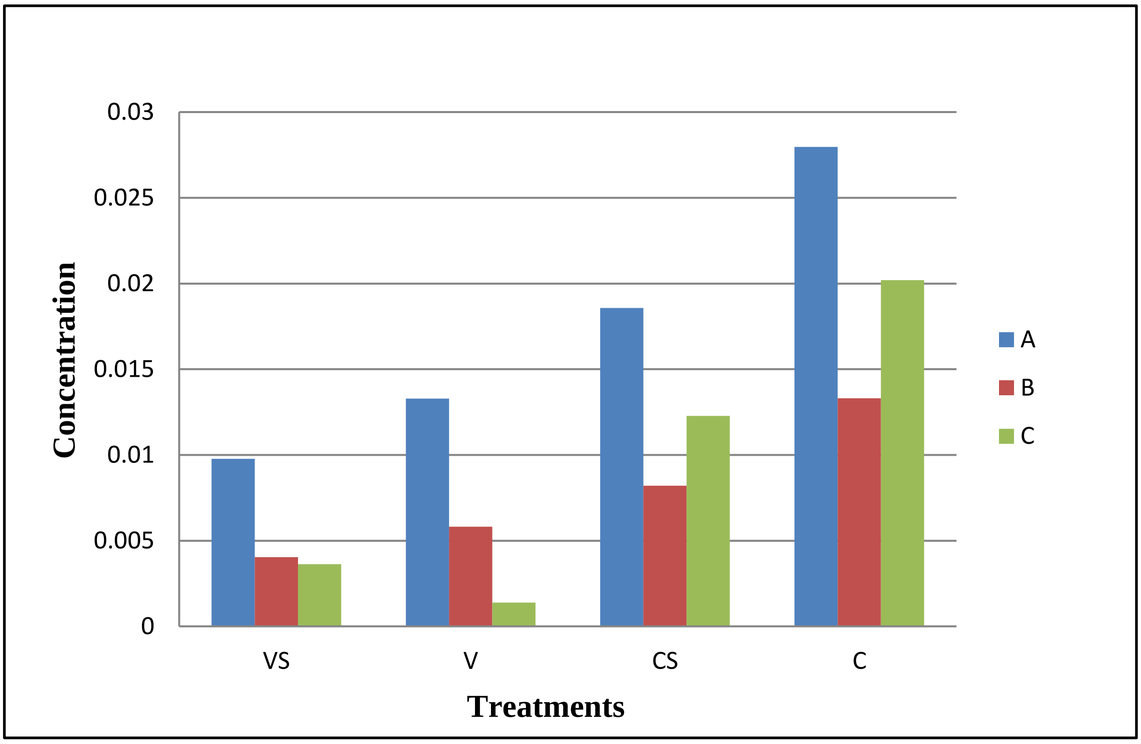

Figure B.13

Experience at the beginning of its establishment

Figure B.14

Experience while working on the project

جـامعـــة النجاح الوطنية
كليـــة الدراســـات العليـــا

تأثير التسميد بحمأة الصرف الصحي والري بالمياه العادمة المعالجة على تراكم المعادن الثقيلة في التربة الجيرية وتربة السهول العميقة ونبات الشعير

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قدمت هذه الرسالة استكمالا لمتطلبات الحصول على درجة الماجستير في العلوم البيئية، من كلية الدراسات العليا، في جامعة النجاح الوطنية، نابلس–فلسطين.

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تأثير التسميد بحمأة الصرف الصحي والري بالمياه العادمة المعالجة على تراكم المعادن الثقيلة في التربة الجيرية وتربة السهول العميقة ونبات الشعير

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الملخص

تم تطبيق هذه التجربة من أجل دراسة تأثير التسميد بالحمأه والري بالمياه العادمة المعالجة المنتجة من محطة معالجة جنين على تراكم المعادن الثقيلة في تربة السهول العميقة والتربة الجيرية ونبات الشعير، ونفذت هذه التجربة في بيت بلاستيكي ضمن ظروف بيئية متحكم بها في المركز الوطني للبحوث الزراعية في جنين. تم استخدام تجربة عمود التربة على نوعين من التربة هما تربة السهول العميقة والتربة الجيرية حيث تم أخذ مقطع من التربة بعمق 1م لكل نوع ومن ثم تمثيله في انابيب بلاستيكية طولها 1م وقطرها 72 ملم (ستة انابيب لكل نوع تربة)، وصممت التجربة بحث وضعت كل 3 اعمدة من التربة في خط (خطين لكل نوع تربة) وتم توصيلها بشبكة ري. بعد ذلك تم تسميد خط من كل نوع تربة ب 200 مل حمأة المياه المعالجة. بعد ذلك تم زراعة 3 بذور شعير في كل انبوب خلال الموسم الزراعي 2021/ 2022، واستمر الري بالمياه المعالجة لمدة 3 شهور. ومن ثم تم حصاد نبات الشعير وتجفيف اعمدة التربة وتقطيعها الى ثلاث طبقات (1−30 سم، 30−60 سم، 60−100سم). استخدمت التحليلات الكيميائية لقياس محتوى النبات وطبقات التربة من المعادن الثقيلة باستخدام جهاز ICP-MS. ايضا تم قياس محتوى عينات النبات من النيتروجين والفسفور والبوتاسيوم وملاحظة طول ووزن النبات. أظهرت النتائج أن التسميد بالحمأة الري بالمياه العادمة المعالجة زاد تركيز الحديد والمنغنيز والزنك والنحاس في نبات الشعير وكذلك بالنسبة للنيتروجين والفسفور والبوتاسيوم. أيضا التسميد بالحمأة زاد من وزن نبات الشعير. حركة المعادن الثقيلة في عمود التربة وتسربها للطبقات السفلية زادت بسبب التسميد بالحمأة.

وأكدت النتائج أن تربة السهول العميقة أكثر خصوبة من التربة الجيرية. يمكن اعتبار الحمأة مصدر للمغذيات النباتية بسبب محتواها من العناصر الكبرى مثل النيتروجين والفسفور البوتاسيوم وكذلك محتواها من العناصر الصغرى مثل الحديد والزنك والمنغنيز.

كلمات مفتاحية: تجربة عمود التربة؛ المعادن الثقيلة؛ تربة السهول العميقة؛ التربة الجيرية.