



# **Assessment Of Some Heavy Metals And Organic Pollutant In Soil And Water Obtained From Pompomari Irrigation Water Channel Within Damaturu Metropolis, Yobe state**

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## **ABSTRACT**

The study was aimed at determining the levels of manganese (Mn), zinc (Zn), lead (Pb), chromium (Cr) and Cadmium (Cd) in soil and water samples of Damaturu town which was used to sensitize the general population of Damaturu town on the importance of environmental conservation, hence inform the authorities in environment management on the level of heavy metal pollution in Damaturu town. The results from the study highlights the remedial action to be taken including treatment of the water to remove the heavy metals where the levels are too high.

**Keywords:** environmental conservation, heavy metals, irrigation water

## **INTRODUCTION**

Pollution is a worldwide problem and its potential in influencing health of the human population is great (Khan and Ghouri, 2011). The impact of pollution in the vicinity of overcrowded cities and from industrial effluents and automobiles has reached a disturbing magnitude and is arousing public awareness (Begum *et al.*, 2009). Excessive levels of pollution are causing a lot of damage to human and animal health, plants including tropical rain forests as well as the wider environment (Khan and Ghouri, 2011). Pollution is the cause of many diseases, which affect not only the old but also the young and the energetic and all animals and plants (Kanmony, 2009). The WHO report points out those twenty million children worldwide suffer from pollution which has become critical because of overpopulation (Kanmony, 2009; Pain, 2008). An estimated 1.2 billion people drink unclean water which is the source of water related diseases that kill between five-ten million people mostly children around the world (Ahuja, 2009).

The natural substances which cause water pollution includes, gases, soils, minerals, humus materials, waste created by animals and other living organisms present in water. Water resources continue to be scarce and insufficient in most areas to meet the growing demands of a rapidly increasing population and because of this scarcity, the resources have continued to be overexploited leading to its salinity, increased pollution and eutrophication due to intensive agricultural practices (Al-Weher, 2008). Estimation indicates that more than fifty countries of the world with an area of twenty million hectares area are treated with polluted or partially polluted water including parts of all continents and this poor quality water causes health hazards and death of human beings, aquatic life and also disturbs the production of different crops (Khan and Ghouri, 2011). The most common environmental pollutants in the world are heavy metals (Papatilippaki *et al.*, 2008). The presence of heavy metals at trace level and essential elements at elevated concentration causes toxic effects if exposed to human population (Fong *et al.*, 2008). The knowledge of heavy metal accumulation in soils, the origin of these metals and their possible interactions with soil properties are a priority in many environmental monitoring (Qishlaqi and Moore, 2007). The accumulation of heavy metals in agricultural soils is of increasing concern due to food safety issues and potential health risks as well as its detrimental effects on soil ecosystems (Qishlaqi and Moore, 2007).

Food chain contamination by heavy metals has become a burning issue in recent years because of their potential accumulation in biosystems through contaminated water, soil and air (Begum *et al.*, 2009).

Heavy metals can accumulate in the soils to toxic levels as a result of long term application of untreated waste waters and fertilizers. Soil irrigated by waste water accumulate heavy metals in surface soils and when the capacity to retain heavy metals is reduced due to repeated application of waste water, heavy metals leach into ground water or soil solution available for plant uptake (Papatilippaki *et al.*, 2008). Research findings indicate that application of heavy doses of fertilizer, pollute ground water by nitrates and heavy metals through leaching and this affects the quality of water (Mico *et al.*, 2006).

Metal poisoning arises from heavy metals that have toxic properties leading to adverse effects on human and ecosystem health (Voet *et al.*, 2008). Although acute poisoning from heavy metal poisoning is rare through ingestion or dermal contact chronic exposure to even small doses can be disastrous (Sherameti and Varma, 2010). Chronic exposure to heavy metals leads to accumulation in the food chain which leads to an increased stock in biota, therefore magnifying the human dose (Voet *et al.*, 2008). The chronic problems associated with long term heavy metals exposure include; Serious hematological and brain damage, anaemia and kidney malfunctioning (Sonayei *et al.*, 2009). Heavy metals such as Lead (Lead (Pb)) and Cadmium (*Cadmium (Cd)*) are lethal even in very small doses. Lead has a negative influence on the somatic development, decreases the visual acuity and additive thresholds (Simeonov *et al.*, 2010). Acute exposure to lead causes brain damage, neurological symptoms, brain damage and could lead to death (Simeonov *et al.*, 2010). Cadmium (Cd) exposure on the other hand, causes renal dysfunction, calcium metabolism disorders and also increased incidence of some forms of cancer possibly due to the inhibition by Cadmium (Cd) of DNA mismatch remediation (Kumar, 2009). Malignant neoplastic and skin ulcers have been reported due to various occupations with exposure to chromium compounds. Chromium (VI) inhalation is responsible for bronchial asthma (Sakar, 2005). Manganese toxicity affects the central nervous system, visual reaction time, hand steadiness and eye-hand coordination (Calkins, 2009). A syndrome named manganism characterized by feelings of weakness and lethargy, tremors, a masklike face and psychological disturbance. Respiratory effects have also been noted in male workers afflicted with manganism (Calkins, 2009). Zinc toxicity is rare, but at concentrations in water up to 40 mg/l, may induce toxicity characterized by symptoms of irritability, muscular stiffness and pain (Al-Weher, 2008). In Damaturu, chemicals and agro-based wastes are the major source of pollution and this arises from agro-chemicals and fertilizers used in the highlands of Pompomari, where rice and vegetables is grown on large scale.

### **Problem statement and justification**

The environment has continued to suffer from pollution due to increased population and industrialization (Goel, 2009). On the other hand resources like land have remained constant leading to overcrowding of population around towns and main cities. The biggest challenge resulting from this overcrowding is waste disposal. The overcrowding has led to domestic and industrial wastes being disposed of in water bodies like rivers and dams and this has led to the contamination of soil and the water bodies, especially from heavy metals (Lesamana, 2009).

Lack of planning in Damaturu town, has resulted in overcrowding with no proper sewage disposal system for waste, hence waste disposal into drainage system has become an order of the day.

This drainage system were usually washed during raining season and deposited around the irrigation farms.

This could therefore lead to both soil and water pollution, which of course could pollute the plantation around the area.

This study therefore proposes to determine the levels of heavy metals and other pollutant in both soil and water around the irrigation farm land in Damaturu town.

### **Aim and Objective of the study**

#### **Aim**

The aim of this study is to determine the heavy metal content and organic pollutant in soil and water around drainage and irrigation channel within damaturu town.

#### **The objective of the study**

- i. To determine the levels of heavy metals in both soils and water around Pompomari irrigation area of Damaturu town.
- ii. To determine the levels of organic pollutants in soil and water around Pompomari irrigation area of Damaturu town.

- iii. To determine the concentration of toxic elements in soil and water samples obtained from Pompomari irrigation area.

#### **Significance of the study**

The determination levels of manganese (Mn), zinc (Zn), lead (Pb), chromium (Cr) and Cadmium (Cd) in soil and water samples of Damaturu town will be used to sensitize the general population of Damaturu town on the importance of environmental conservation. The study will also inform the authorities in environment management on the level of heavy metal pollution in Damaturu town hence providing a reference for future studies. The results from the study will also be used to determine the remedial action to be taken including treatment of the water to remove the heavy metals where the levels are too high.

#### **Scope and limitations**

There are many heavy metals but this study considered only five heavy metals which from literature are prevalent in soils and water in Damaturu town. The study also covered only one area of Damaturu town because of its expansiveness, inaccessibility and resource availability. Some heavy metals could be present in the underlying rocks and this could be reflected in the levels and assumed to be from anthropogenic sources.

#### **LITERATURE REVIEW**

Pollution includes natural as well as manmade substances or energy that may have an adverse impact on human health or well-being or on the natural or cultural heritage. Environmental degradation due to pollution in poor countries is pervasive, accelerating and unabated (Farmer, 2002). In developed countries, a lot of resources have been used to ensure that there is cleaner air, drinking water, sewage treatment, safe food laws and food refrigeration (Hill, 2010). It is usually easy to see the effects of pollution on the earth and in plants and animals, but it is more difficult to reduce the amount of pollution put into the environment (Shafi, 2005). For example it is estimated that at least 1.6 million lives are lost each year through lack of access to sanitation and drinking water (Farmer, 2002) and more millions of people are left chronically ill from the water they must drink (Hill, 2010). It is important to note however that even a very small concentration of persistent pollutants may cause irreparable damage to the ecosystem. Organisms susceptible even to low concentration may get eliminated (Shafi, 2005). Scientific analysis is important for those pollutants with threshold for impacts to determine the nature of the threshold and for those without thresholds, to determine the significance at a level of "acceptable" impact (Farmer, 2002).

The major sources of pollution include; burning fossil fuel in engines, waste disposal, accidental spills of chemicals from factories and use of agricultural chemicals on farms (Greenaway *et al.*, 2002). There are several types of pollution including air pollution, water pollution and soil pollution.

#### **Soil pollution**

Soil is a very important natural resource to man as it is a source of his life on this planet. Without soil the earth would be as barren as the moon hence lifeless (Misra and Mani, 2009). Despite its importance, soil is often contaminated by human activities and this is reflected in the high horizontal and vertical variability brought about by the anthropogenic influence on soil formation and development (Fong *et al.*, 2008). A variety of human activities including municipal waste disposal, industrial emissions, military testing and agricultural practices have left their impacts on soils in the form of elevated and high level of toxicants (Van and Krivolutsky, 1996).

Materials that find their entry into the soil system persist and accumulate in toxic concentrations becoming sources of pollution in the soil (Misra and Mani, 2009). The concentration of heavy metals in soil and their impact on ecosystems can be influenced by many factors such as the parent rock, climate and anthropogenic activities (Jia *et al.*, 2010). Among the pollutants that persist and accumulate in the soils include; inorganic toxic compounds for example fertilizers, organic wastes, organic pesticides and radio nucleides (Misra and Mani, 2009; Jia *et al.*, 2010). The soil is thus becoming increasingly polluted with chemicals and other pollutants which can reach the food chain, surface water or ground water and ultimately be ingested by man (Misra and Mani, 2009).

#### **Water pollution**

Pollution of water still remains one of the most significant environmental problems of recent times. Water can be regarded polluted when it gets changed in its quality or composition either naturally or as

a result of human activities so as to become less suitable for drinking, domestic, agricultural, industrial, recreational, wildlife and other uses for which it would have been otherwise suitable in its natural or unmodified state (Goel, 2009). Gross pollution of water has its origin mainly in urbanization, industrialization, agriculture and increase in human population being observed (Calhoun, 2005; Goel, 2009). In addition to toxic chemicals, water pollutants occur in many other forms, including pathogenic microbes, excess fertilizers and trash floating on streams, lakes and beaches. Water pollution can also take the form of sediment eroded from stream banks, large booms of algae, low levels of dissolved oxygen or abnormally high temperatures (Calhoun, 2005). Water pollution threatens our health and environment and therefore we need to implement an expanding array of techniques for its assessment, prevention and remediation (Calhoun, 2005).

#### ***Heavy metal and environmental pollution***

There are different types of pollution among which pollution caused by toxic level of heavy metal pollutants is called heavy metal pollution (Bose and Hemantaranjan, 2005). Heavy metals are elements having a density greater than 5 g/cm<sup>3</sup> in their elemental form (Bose and Hemantaranjan, 2005; Misra and Misra and Mani, 2009). Heavy metal pollution has received the attention of researchers all over the world, mainly due to their harmful effects on living beings (Misra and Misra and Mani, 2009). Human biology is full of instances where heavy metal toxicity has led to mass deaths (Shrivastav, 2001). All heavy metals are toxic to living organisms at excessive concentrations, but some are essential for normal healthy growth and reproduction by plants at low but critical concentrations (Bose and Hemantaranjan, 2005). The heavy metals essential in trace elements to plants include Co, Cu, Fe, Mo and Zn and for animals are Cr, Ni and Sn. The heavy metals Cadmium (Cd), Hg and Lead (Pb) have not been shown to be essential for either plants or animals (Misra and Misra and Mani, 2009).

It is important to note however that the concentrations of individual metals in living tissues must be kept very low and should be maintained within narrow limits to permit the optimum biological performance of most organisms (Misra and Mani, 2009). Heavy metals are non-biodegradable and once they enter into an environment, they will stay there for a longtime (Voet *et al.*, 2008). Heavy metals are considered serious pollutants because of their toxicity, persistence and nonbiodegradable conditions in the environment, thereby constituting a threat to human beings and other forms of biological life (Adeleken and Abegunde, 2011). Heavy metals occur in atmosphere basically in particulate form. Hence, the transfer of airborne particles to land or water surfaces by dry, wet and occult deposition constitutes the first stage of atmospheric heavy metals (Shrivastav, 2001).

Adeleken and Abegunde (2011) note that heavy metals have low environmental mobility as a result of this, a single contamination could set a stage for a long term exposure of human, microbial, fauna, flora and other edaphic communities to heavy metals. The problem of atmospheric heavy metal pollution is not going to disappear overnight. On the contrary it will remain a legacy of mass industrial activity for many generations and is likely to escalate further in future. In this regard, the compilation of past and present catalogues of atmospheric heavy metal concentration is an activity of great importance (Shrivastav, 2001).

#### ***Soil pollution from heavy metals***

Heavy metal pollution in soils refers to cases where the quantities of the elements in soils are higher than maximum allowable concentrations and this is potentially harmful to biological life at such locations (Adeleken and Abegunde, 2011). Heavy metals occur at typical background in all ecosystems, however, anthropogenic releases can result in higher concentrations of these metals relative to their normal background values hence the pollution (Adeleken and Abegunde, 2011). Heavy metals released from vehicular emission can accumulate in surface soils and their deposition over time can lead to abnormal enrichment, thus causing metal contamination of the surface soils (Fong *et al.*, 2008).

High concentrations usually occur in soils below or near landfills and agricultural lands that have been irrigated with contaminated water (Mamtaz and Chowdhury, 2006). Studies have shown that both long term and short term contamination of soils have effects on microbial activity and enzyme activities of the soil (Adeleken and Abegunde, 2011). The toxicity and mobility of heavy metals in soils depend not only on the total concentration but also on their specific chemical form, bonding state, metal properties, environmental factors, soil properties and organic matter content (Osu and Okoro, 2011). Exposure of children, generally accepted as the highest risk group who have a higher adsorption rate of heavy metals because of their active digestion system and sensitivity of haemoglobin, to heavy metals, can greatly

increase ingestion of metal laden soil particles via hand –to-mouth activities. In addition, adults may be exposed to threat since inhalation is easier pathway for toxic metals to enter their body (Fong *et al.*, 2008).

### Heavy metals in water

The contamination of fresh waters with a wide range of pollutants has become a matter of great concern over the last few decades (Al-Weher, 2008). The aquatic systems receive a large amount of heavy metals from natural occurring deposits and natural processes and anthropogenic activities (Wogu and Okaka, 2011). Anthropogenic sources arising from human activities such as industrial, municipal effluents, as well as non-point source run off are the main sources of metals in rivers (Sonayei *et al.*, 2009).

Discharge of heavy metals into rivers or any other aquatic environment can change both aquatic species diversity and ecosystems due to their toxicity and accumulative behaviour (Al-Weher, 2008). Heavy metals dissolved in water also endanger the lives of the public who use it for drinking and also irrigation. When used for irrigation heavy metals have the danger of being incorporated in food chain and therefore ingested by the public (Wogu and Okaka, 2011). Heavy metals accumulate in the soils at toxic levels as a result of long term application of untreated waste water and therefore soils irrigated by wastewater accumulate heavy metals in their soil surface (Sonayei *et al.*, 2009). When the capacity of the soil to retain heavy metals is reduced due to repeated application of waste water, the metals leach into ground water or soil solution available for uptake (Sonayei *et al.*, 2009). Table 2.1 shows metal limits in water set by national and international organizations.

**Table 2.1 Heavy metal limits (µg/l) in water set by different national and international organizations Cadmium (Cd)**

	Cadmium (Cd)	Cr	Mn	Lead(Pb)	Zn
<b>USEPA</b>	5	100	50	10	5000
<b>EU</b>	5	50	50	10	Nm
<b>WHO</b>	3	50	400	10	NGL
<b>Iranian</b>	10	50	500	50	Nm
<b>Australian</b>	2	50	500	10	3000
<b>Indian</b>	10	50	100	100	5000
<b>New Zealand</b>	4	50	400	10	1500

Nm- not mentioned

NGL – No guideline, because it occurs in drinking water at concentrations well below those at which toxic effects may occur  
Source; Mebrahtu and Zerabruk (2011).

### Methods for heavy metal analysis

Several techniques for the determination of heavy metal elements are currently in use. These include flame atomic absorption spectroscopy, inductively coupled plasma atomic emission spectroscopy (ICP-AES) (Sonayei *et al.*, 2009), inductively coupled plasma –mass spectroscopy (ICP-MS) (Nassef *et al.*, 2006); X-ray fluorescence and neutron activation analysis (Magdaleno *et al.*, 2011). The AAS was used in this study because of its simplicity, reliability and sensitivity (Sarkar, 2005). A lot of studies on heavy metals in water, soil plants and animals have used atomic absorption spectroscopy as the method of analysis for the heavy metals. A study by Begum *et al.* (2009) in the analysis of lead, iron, zinc, nickel and copper in soil and plants employed the method for analysis. Similarly, Fong *et al.* (2008) in the analysis of copper, Cadmium (Cd), manganese, lead and zinc in urban roadside soils used atomic absorption spectroscopy in their analysis. Other researchers who have employed atomic absorption spectroscopy in their analysis of heavy metals include, Mantaz and Chowdhury (2006) who studied iron, copper, manganese and zinc levels in urban solid waste, Awokunmi *et al.* (2010) in their study of Cadmium (Cd), cobalt, chromium, copper, lead, manganese, nickel and zinc levels in soils from a dumpsite, Mico *et al.*, (2006) employed atomic absorption spectroscopy method in analysis of Cadmium (Cd), cobalt, chromium, copper, iron, manganese, nickel, lead and zinc in the agricultural soils of Segura River Valley in Spain. Al weher (2008), analysed levels of Cadmium (Cd), copper and zinc in three species of fish using atomic absorption spectroscopy method. Similarly Wieczorek *et al.* (2005)

employed the same method of analysis in determining the levels of lead in cereal grains and soils adjacent to roadways.

### Atomic spectroscopy

This technique is applicable to most gas phase elements over a wide range of concentrations and involves detecting, measuring and analyzing radiation that is either absorbed or emitted from the atoms or ions of the element of interest (McMahon, 2007). It involves three techniques: Absorption, emission and fluorescence. In all the above, the sample is decomposed by intense heat into hot gases consisting of free atoms and ions of the element of interest (McMahon, 2007). As atoms are the simplest and purest form of matter and cannot rotate or vibrate as a molecule does when subjected to high energy radiation, electrons within the atom undergo transitions. The high energy radiation is commonly produced by

1. Flame in flame atomic absorption spectroscopy (FAAS)
2. Electro thermal furnace in flameless graphite furnace atomic absorption spectroscopy (GFAAS)
3. Plasma in inductively coupled plasma-optical emission spectroscopy (ICP-OES).
4. X-ray in X-ray fluorescence spectroscopy (XRF) ((Lajunen and Paavo, 2007))

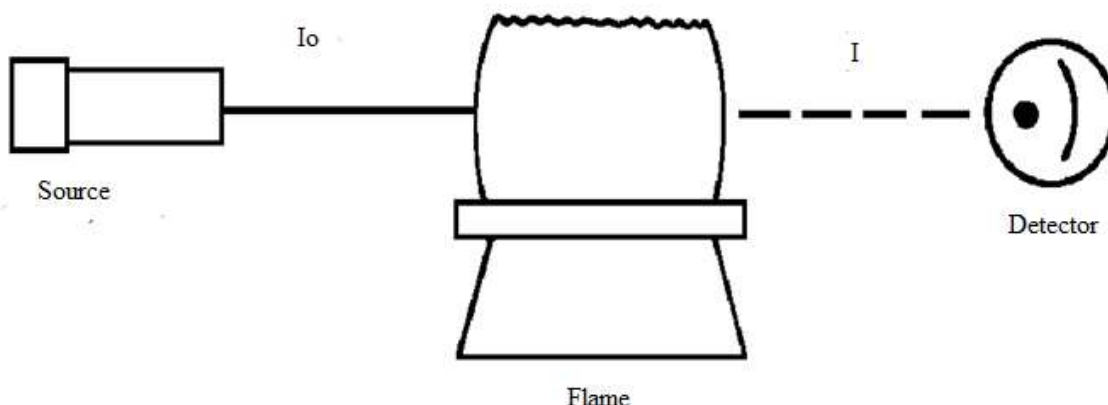
The above four belong to one of three major types of atomic spectroscopy namely absorption, emission and fluorescence ((Lajunen and Paavo, 2007)).

### Atomic absorption spectroscopy

#### Principle of AAS

An atom is made up of positively charged nucleus surrounded by a number of negatively charged particles necessary to provide neutrality. These atoms occupy discrete energy levels but it is possible for an electron to be moved from one level to another by introduction of energy. Such transitions will only occur if the available energy is equal to the difference between the two levels. Energy levels and the energies associated with electron transitions are unique for each element. When light (energy) of a characteristic wavelength enters an analytical system, outer shell electrons of corresponding atoms within the light path will be excited as energy is absorbed. The amount of light transmitted through the system from a source to the detector will be less. The loss of light is proportional to the number of atoms. The measurement of the radiation transmitted (using Beer-Lambert's law) in such a transition form the basis of AAS. Beer-Lambert's law relates absorbance,  $a$  to the concentration of metallic atoms in the atom cell,  $c$  as follows  $\text{Log}T-1 = a b c$  Where  $a$  is the absorptivity in grams per litre-centimetre  $b$  is the atom width in centimeters  $c$  is the concentration of atoms

The AAS involves the measurement of the drop in light intensity of initial radiation  $I_0$  to final radiation  $I$  depending on the concentration of the metal. Modern instruments automatically convert logarithmic values into absorbance (Nollet, 2011). Figure 2.1 below illustrates AAS instrumentation.



**Figure 2.1: Diagram to illustrate instrumentation of AAS**

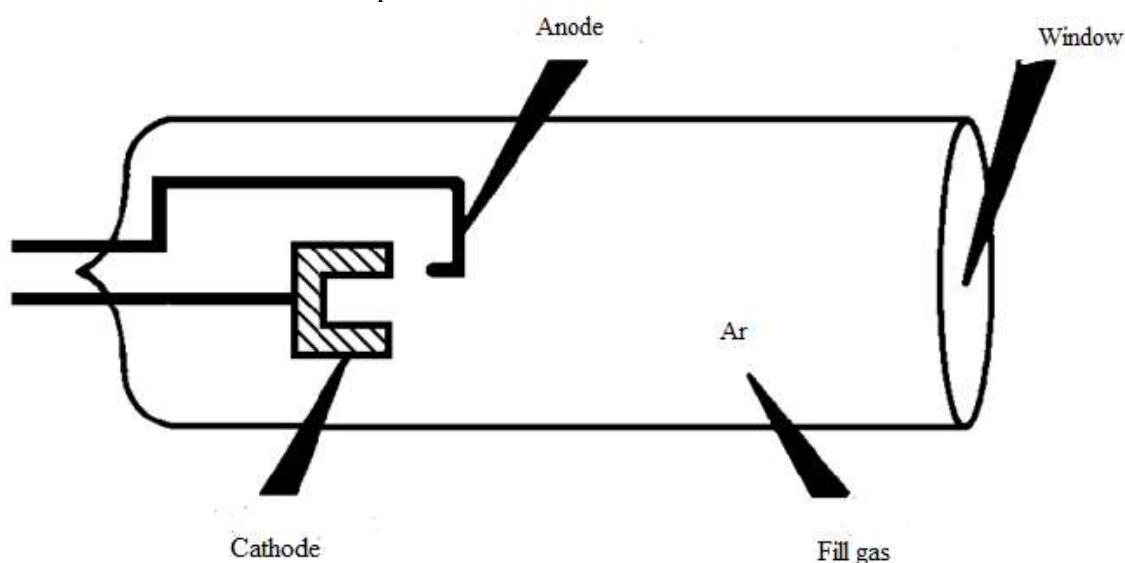
#### Instrumentation of AAS

Any atomic absorption spectrometer consists basically of a light source which emits the sharp line spectrum of elements to be determined, a method to produce atomic vapour of the sample to be

analyzed, a monochromator for the spectral dispersion of the source radiation, a detector connected to an amplified read out system and a computer.

### Light source

A continuous source of radiation is required. A series of sources which can give sharp emission lines for a specific element are used. A hollow cathode glow discharge lamp is used. A hollow cathode lamp has two electrodes; one is cup shaped and made of a specific element. The metal used for the cathode is the same as the metal to be analyzed. The lamp is filled with noble gas at low pressure. It will produce a glow discharge from the hollow cathode. Metal atoms are evaporated by sputtering. The atoms accept energy of excitation and emit radiation with the lines of the metal. Hollow cathode lamps made out of several elements are available (Khopkar, 1998). The figure 2.3 below shows one of the light source used by AAS machine; a hollow cathode lamp.



**Figure 2.2: A hollow cathode lamp.**

### Atomization

Several types of atomizers are used for atomization. These include flame, electrothermal, cold vapour technique for mercury and hydride generation (Nielsen, 2010). The flame atomizers consist of a nebulizer and a burner. The nebulizer is designed to convert the solution into a fine mist or aerosol. In flame atomization, atomization is carried out by flame. Heat energy is utilized to convert the metallic element to atomic dissociated vapour. The temperature should be controlled very carefully to convert it to atomic vapour. At too high or too low temperatures, atoms will be ionized and they will not be absorbed. In atomization, fuel and oxidant gases are fed into a mixing chamber which passes through baffles to the burner head. A flame is produced and the sample is aspirated through the air into the mixing chamber. Only droplets of a small size pass through the baffles to the burner head. A narrow burner is therefore preferred and careful readjustment of the gas (Khopkar, 1998; Nielsen, 2010).

In electrothermal atomization, electrothermal atomizers are used. Electrothermal atomizers are typically cylindrical graphite tubes connected to an electric power supply. The sample is introduced into the tube through a small hole using a microlitre syringe. The system is flushed with an inert gas to prevent the tube from burning and exclude air from the sample compartment. The tube is heated electrically to evaporate the solvent, the sample is then ashed and further heated to quickly vapourized and atomize the sample (Nielsen, 2010). Cold vapour technique of atomization works for mercury only. In this technique, mercury compounds in a sample are reduced to elemental mercury by the action of stannous chloride, a strong reducing agent. The elemental mercury is then carried in a stream of air or argon into an absorption cell where absorption takes place (Nielsen, 2010). In hydride generation technique of atomization, volatile hydrides of elements are formed by reacting samples with sodium borohydride. The hydrides are carried into an absorption cell and heated to decompose them into free atoms. The atomic absorption measurements are then carried out (Nielsen, 2010).



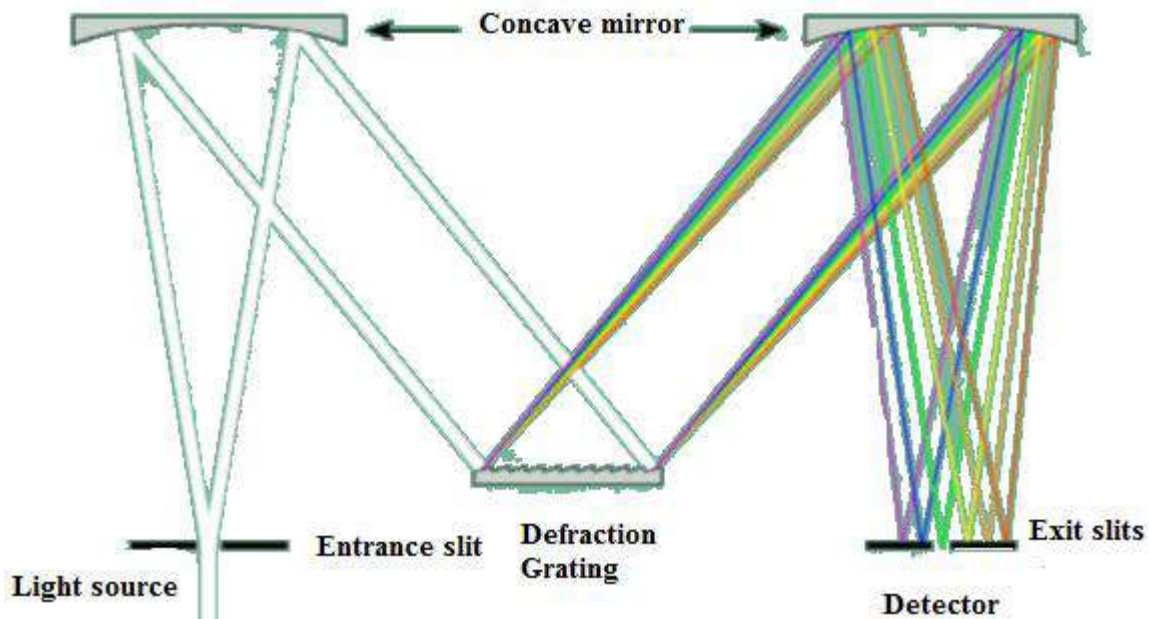
### A monochromator

A monochromator produces monochromatic light by removing unwanted wavelengths from the source light beam. It isolates a single atomic resonance line from the spectrum of lines emitted by hollow cathode lamp. Essentially it is an adjustable filter that selects a specific, narrow region of spectrum for transmission to the detector and excludes all wavelengths outside this region.

A monochromator comprises an entrance slit, a dispersion device and an exit slit.

- i. The entrance slit selects a defined beam of light from the source
- ii. The dispersion device causes the different wavelength of light in the source beam to be dispersed at different angles
- iii. The exit slit enables selection of a particular wavelength to produce the required monochromatic light

Figure 2.3 below shows a model of a monochromator used by an AAS machine



**Figure 2.3: A model of a monochromator**

There are two types of monochromators; prism and grating systems. Prisms and grating systems separate various wavelengths of light in different fashions. Prisms refract light at the interface of two surfaces with differing refraction indexes creating angular dispersion. Prisms have limitations, their resolution is significantly lower than a grating system and their separation technique is non-linear which creates mechanical problems with focusing a specific wavelength on the exit slit.

Diffraction gratings are materials with a large number of parallel and closely spaced slits or ridges. Diffraction causes constructive interference at unique points for each wavelength. The separated wavelengths are collimated with a concave mirror towards the exit slit. The tilt angle of the grating device determines the band of wavelength exiting the monochromator and reaching the detector.

### The detector

Two detectors are used in atomic absorption spectrometers; photomultiplier tubes and solid state detectors (Nielsen, 2010). Detectors convert the radiant energy reaching it into an electrical signal. The signal is processed to produce either an analogue or digital read out. Modern instruments are interfaced with computers for data collection, manipulation and storage. The photomultiplier tubes are the most common types of detectors used.



## MATERIALS AND METHODS

### Materials and Methods

#### The Study Area

The study area is located within Damaturu in Pompomari Irrigation Farm. Damaturu is the capital city of Yobe State of Nigeria, which is located on coordinates of 11°44' 55" N, 11°57' 50"E in the north-eastern part of Nigeria, with an area of 2,366 km<sup>2</sup>. The textural class of the soil was Clay loam. The vegetation of this area falls within the Sudan savannah.

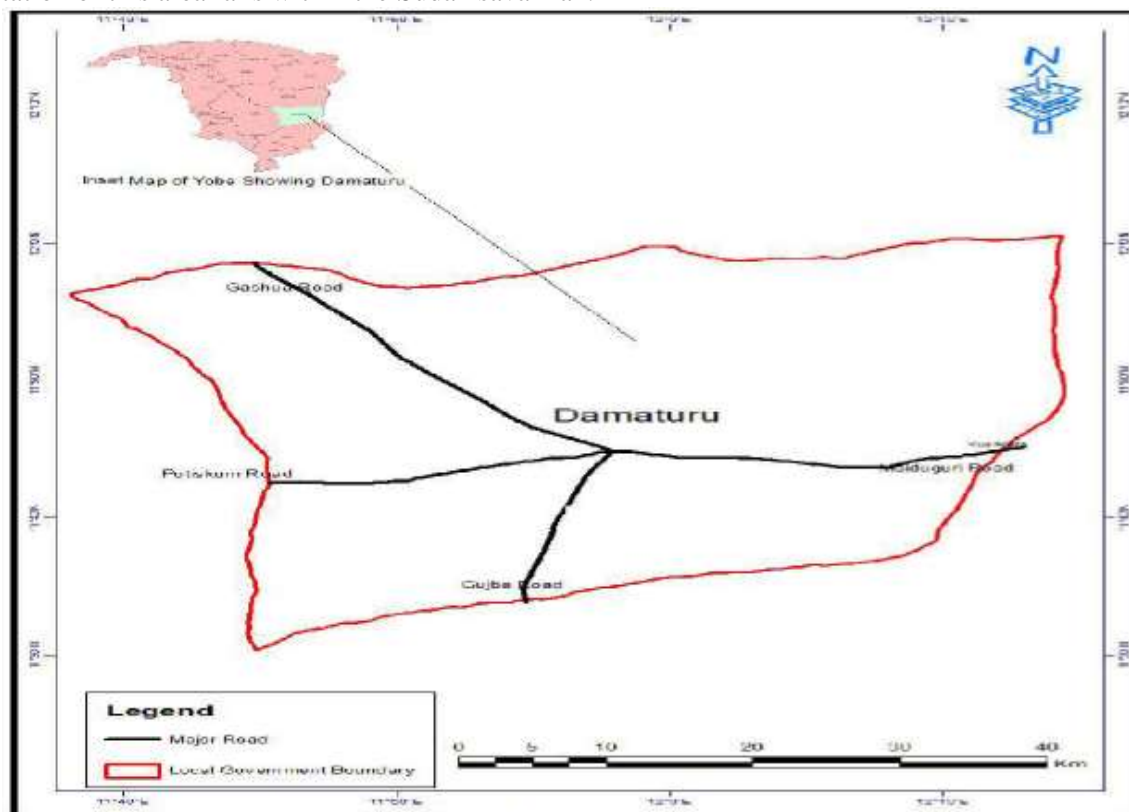


Fig.1: Map the study area (Adapted from Galadima, 2018)

#### Chemicals reagents

Chemicals and reagents to be use will be of analytical grade. They included; nitric acid, hydrochloric acid, distilled water, hydrogen peroxide, lead nitrate, zinc nitrate, Chromium oxide, manganese powder, ammonium chloride and Cadmium (Cd) nitrate. The chemicals were obtained from Chemistry laboratory Yobe State University.

#### Cleaning of glassware

All apparatus will be initially washed with detergents then soaked in 2 M nitric acid for 24 hours then washed thoroughly. They will be rinsed with aqua regia, followed by tap water and then rinsed in distilled water. The glassware is then dried in a hot oven at 1050C.

#### Sampling

##### Soil sampling

Sampling sites will be chosen in line with anthropogenic sources of heavy metals. At each sampling point, approximately 0.5 kg of soil will be collected 0 -10cm in depth using a stainless steel sampler. Soil samples along the roads will be collected at a distance of one meter away from the road and within an area of one square meter. Three samples will be collected from each point, thoroughly mixed in a clean plastic container to obtain a representative sample dried, crushed and sieved with 2 mm mesh before being stored in labeled polythene bags prior to the analysis.

The soil samples will be label according to the regions from which they will be obtained. Fig 3.1 show soil sampling sites



**Plate 3.1: Soil sampling from wheat farm in Pompomari Irrigation Farm**

#### **Water sampling and pre-treatment**

Water sample from stream will be obtain for analysis. Water from borehole will be sampled 10-15 cm below the water surface using label acid washed plastic containers to avoid unpredictable changes in characteristic as per standard procedures (Reza and Singh, 2010). The water will be label according to the source from which it will be obtain and also the region from which it will be obtain. The water will be acidified with 2 ml of analytical grade nitric acid in order to preserve metals and avoid precipitation (Kar *et al.*, 2008). The water samples will be stored at a 5°C temperature awaiting the transportation to the laboratory for analysis.

The picture in the Fig. 3.2 shows water sampling sites.



**Plate 3.3: Water sampling site, Pompomari Irrigation Farm**

### **Sample digestion**

#### **Water digestion**

The water sample bottles will be shaken thoroughly in their plastic containers by use of hand. A volume of 100 ml of the sample will be measured using a 100 ml volumetric flask and put in a conical flask and 5 ml of concentrated nitric acid will be added. The mixture will be heated slowly on a hot plate and evaporate to about 20 ml ensuring that the water did not boil. A further 5 ml of concentrated nitric acid will then be added and the beaker will be covered with a watch glass while heating continue. Nitric acid continue to be added until the solution appeared light coloured and clear. Lastly, 2 ml of concentrated hydrochloric acid will be added and heated slightly to dissolve any remaining residue. Few drops of hydrogen peroxide will then be added to ensure complete digestion had taken place. The solution will be filtered and the filtrate will be transfer to a 100 ml volumetric flask to cool and the filtrate will make up to the mark with distilled water (Radojovic and Bashkin, 2006).

#### **Soil digestion**

Well mixed samples of 1 g each will be weigh using a Scientech Zeta series electronic balance manufactured in the year 2000. The samples will be put into 250 ml glass beaker and digest with 24 ml of aqua regia and evaporate to near dryness. The soil samples will then dissolved in 10 ml of 2% nitric acid, filtered and then diluted to 100 ml with distilled water (Begum *et al.*, 2009).

### **Preparation of stock solutions and standards**

#### **Lead stock solution and standards**

Lead stock solution (1000 mg/l) will be prepare by dissolving 1.59 g of lead (ii) nitrate in 500 ml of distilled water and then make up to 1 liter of solution using distilled water. Through serial dilutions, standard working solutions of lead of 1, 2, 3, 4 and 5 mg/l will make which will be used to generate a calibration curve for lead.

#### **Zinc stock solution and standards**

Zinc stock solution (100 mg/l) will be prepare by dissolving 0.289 g of zinc nitrate salt in 300 ml of distilled water and then make up to 1 liter of solution using distilled water. A working zinc standard solution (20 mg/l) will be made by diluting 20 ml of the stock solution to 100 ml of solution. The calibration graph will be made using solutions with the following concentrations; 0.5, 1, 1.5, 2, and 2.5 mg/l of zinc.

#### **Manganese stock solution and standards**

Manganese stock solution (100 mg/l) will be prepare by dissolving 0.10 g of manganese metal powder in 10 ml of concentrated hydrochloric acid mix with 1 ml of concentrated nitric acid. A 10 ml of nitric acid was then be added and the solution would finally be dilute to 1000 ml with distilled water. A working manganese standard solution (20 mg/l) will be made by diluting 20 ml of the stock solution to 100 ml of solution using distilled water. The calibration graph will be made using solutions with the following concentrations; 0.5, 1, 1.5, 2 and 2.5 mg/l of manganese.

#### **Cadmium stock solution and standards**

Cadmium stock solution (1000 mg/l) will be prepare by dissolving 0.275 g of Cadmium (Cd) nitrate salt in 500 ml of distilled water and make up to 1 litre of solution using distilled water. A working Cadmium (Cd) standard solution (10 mg/l) will be made by diluting 10 ml of the stock solution to 100 ml of solution with distilled water. The calibration graph will be made using solutions with the following concentrations; 0.2, 0.4, 0.6, 0.8, and 1.0 mg/l of Cadmium.

#### **Chromium stock solution and standards**

Chromium stock solution (1000 mg/l) will be prepare by dissolving 0.38 g of CrO<sub>3</sub> in a solution of 20 ml water and 4 ml of concentrated nitric acid and diluted to 200 ml using distilled water. Through serial dilution, standard working solutions of chromium will be made. The calibration graph will be made using solutions with concentrations of 1, 2, 3, 4 and 5 mg/l.

The sample analysis will be carried out using Atomic Absorption Spectrometer (AAS) for heavy metals while Gas Chromatography (GC) and other analytical equipment will be employed for organic pollutants analysis.

#### **Method Validation**

The digestion method and atomic absorption spectroscopy analysis were validated by recovery method. One gram of randomly selected soil powder was spiked with three different concentrations of heavy

metals one at a time (1.0, 1.5, 2.0 ppm) each run in with the AAS machine. This was followed by the digestion of the spiked samples and determination of metal concentration using AAS.

Blank or unspiked samples were digested through the same process and analyzed by same AAS. The amount that was recovered after digestion of the spiked samples was used to calculate % recovery (Alweher, 2008). A mean recovery of the matrix was evaluated at 95% confidence level (Borosova *et al.*, 2002).

### Sample analysis

Buck scientific (210 VGF) flame atomic absorption spectrophotometer machine was used in this analysis. Its parameters were set according to the specifications given in the manufactures manual including lamp current and fuel system of air/acetylene flame. The AAS machine had a picking meter that indicated when the optimum conditions had been realized. Its optimization was automatic. The Table 3.1 below shows elements and their wavelength of analysis in air acetylene flame.

**Table 3.1: Elements and their conditions of analysis.**

Element analyzed	Wavelength of analysis (nm)
Zinc	213.8
Chromium	357.9
Manganese	278.5
Lead	283.3
Cadmium (Cd)	228.8

### Data analysis

The data derived from various determinations was subjected to statistical analysis including mean, Pearson Correlation, t-test and ANOVA. The means for the levels in water and soil in the two divisions were determined. Using ANOVA and t-test, the means were compared to determine whether they were significantly different. Pearson correlation was used to relate the levels of heavy metals in water and soil.

## RESULT

Water samples from the research location were found to be colorless and odorless after examination. The analytical results Of metals in soil and water samples are reported in Table 1&2 However metal concentrations in all study locations had no Significant differences in concentrations when compared to WHO defined Allowed range of metal ions. According to average values, The content of heavy metals in soil samples decreases in Descending order Cr> Pb> Mn> Cd> Zn. the content of heavy metals in water samples decreases in descending order Ni> Cr>Fe> Zn>Pb> Cd. Moreover, pH value of Damaturu town is maximum (alkaline).

Table 1: Concentration of heavy metal in soil (mg/kg)

Element	Pompomari 1	Pompomari 2
Cr	0.97	0.84
Pb	0.89	0.72
Mn	1.98	1.24
Cd	0.90	0.37
Zn	2.51	1.24

**Table 2** Concentration of heavy metal in water (mg/L)

Element	Pompomari 1	Pompomari 2
Cd	0.07	0.04
Pb	0.09	0.06
Cr	0.60	0.78
Zn	0.17	0.20
Ni	0.07	0.01
Fe	0.42	0.99

## CONCLUSIONS

The study has shown that there was considerable amount of heavy metals in ground and surface water, though the levels were below WHO maximum permissible levels for Mn, zinc and Cr. In few cases the levels of Cadmium (Cd) exceeded the WHO maximum permissible limit. The mean of lead was above the WHO maximum permissible limits in ground and surface water from Pompomari area.

## RECOMMENDATIONS

The following may be recommended from this research and should be taken to the researchers in this area

- i. The levels of lead and Cadmium (Cd) in the water should be continuously monitored to check on their levels. Lead levels in both the surface and ground water is already beyond the WHO recommended maximum limit. The two heavy metals are very poisonous even in their smallest quantities.
- ii. Sources of heavy metals in soils like inorganic fertilizers, pesticides and acaricides need to be controlled. Fertilizers, pesticides and acaricides are known to be the sources of some of the heavy metals like lead, zinc and Cadmium (Cd) which have been detected both in soils and water at high levels.

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