

Concentration of Some Metals in Soil Samples Collected from Different Parts of Ethiopia

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Abstract

The amounts of some metals (K, Ca, Mg, Fe, Mn, Zn, Cu, Cd, and Pb) in the soil samples collected from the three different areas of Ethiopia (Bahir Dar, Bure and Debre Markos) were determined by using the Microwave Plasma-Atomic Emission Spectroscopy (MP-AES). After proper sample pretreatment, 5 mL HNO₃: 1 mL HClO₄, 240°C and 2:30 h were the optimized digestion conditions. The results showed that the mean concentration of the Fe content of the soils is the highest of all the other studied metals in all of the three sampling areas. The overall mean concentrations of the metals collected from the three sampling areas in mg/kg can be ordered as Fe (74089 ± 3) > Ca (4484 ± 3) > K (3567 ± 3) > Mg (1239 ± 1) > Mn (1196 ± 3) > Zn (152 ± 2) > Cu (39.0 ± 2) > Pb (4.20 ± 0.6) > Cd (2.10 ± 0.2). There was a significant difference between the metal concentrations of the soils collected from the three sampling areas at a confidence level of 95%. On the other hand, the soil samples were not free from the level of the toxic heavy metal Pb and Cd ranges from 3.10-5.25 mg/kg of Pb and from 1.09-3.14 mg/kg of Cd collected from the three sampling areas. The accuracy of the optimized procedure was evaluated by analyzing the digest of the spiked samples with standard solution and the percentage recoveries were varied from 92%-104%, which was within the acceptable range of 100% ± 10%.

Keywords: Accuracy; Digestion; Metals; Optimization; Soil.

Introduction

Soil is a vital component, medium of unconsolidated nutrients and materials, forms the life layer of plants. It is a basic life support components of biosphere. Plants get nutrient from environmental compartment (soil, water and air). Metals present in the soil fractions vary in degree of mobility. Soil is composed of minerals, Soil Organic Matter (SOM), water and air. The composition and proportion of these components greatly influence soil physical properties including texture, structure and porosity, the fraction of pore space in a soil.

The bioavailability of metals in plant depends on a number of physicochemical properties such as pH, organic matter contents, cation exchange capacity, redox potential, soil texture and clay contents. Soil pH refers to a soil's acidity or alkalinity and is the measure of hydrogen ions (H⁺) in the soil. Soil pH can affect Cation Exchange Capacity (CEC) and Anion Exchange Capacity (AEC) by altering the surface charge of colloids. A higher concentration of H⁺ (lower pH) will neutralize the negative charge on colloids, thereby decreasing CEC and increasing AEC. The opposite occurs when pH increases. The pH scale ranges from 0-14 and pH range of soils may be divided: 3.5-5.0 strongly acidic, 5.0-6.5 moderately acidic, 7.0 neutral and 8.5-10.0 strongly alkaline. The Solubility of mineral nutrients is greatly affected by soil pH, phosphorous is never readily soluble in the soil but is most available in soil with a pH range that centered around 6.5. Strongly acidic soils can have high concentration of soluble Al³⁺, Cu²⁺, Fe³⁺, and Mn²⁺ ion which may be toxic to the growth of some plants. A pH range of approximately 6-7 promotes the most readily available plant nutrients, while a pH above 7 (alkaline)

reduces the ability of plants to absorb elements such as iron, manganese, boron and other trace elements [1].

Soil pH is an important factor for plant growth, as it affects nutrient availability, nutrient toxicity and has a direct effect on the protoplasm of plant root cells. It also affects the abundance and activity of soil organisms responsible for transformations of nutrient. Since most mineral nutrients are readily available to plants when soil pH is near neutral (pH=6.5-7.5), species richness is high in such neutral soils, declining in both acidic and alkaline soils. Electrical Conductivity (EC) is a numerical expression of the inherent ability of a medium to carry an electric current. Soil EC is also one of the simplest and least expensive soil measurements available to check soil quality. Soil EC is a measurement that integrates many soil properties affecting crop productivity. These include water content, soil texture, salinity and exchangeable calcium and magnesium. EC indicates how much dissolved salt is in a given sample. The quality of soil is controlled by physical, chemical and biological components of a soil and their interactions.

The term salinity refers to the presence of the major dissolved inorganic solutes (essentially Na⁺, Mg²⁺, Ca²⁺, K⁺, Cl⁻, SO₄²⁻, HCO₃⁻, NO₃⁻ and CO₃²⁻) in aqueous samples. As applied to soils, it refers to

the soluble plus readily dissolvable salts in the soil or operationally in an aqueous extract of a soil sample. Salinity is quantified in terms of the total concentration of such soluble salts or more practically in terms of the electrical conductivity of the solution, because the two are closely related. Theoretically, the Electrical Conductivity of the soil solution (ECs) is a better index of soil salinity. ECw has not been

widely adopted for routine appraisals of soil salinity because methods for obtaining soil water samples are not practical at typical field water contents. Salt in soil comes from the fertilizer we apply but also from irrigation water and dissolving soil minerals. Soils contain some salts, which are essential for plant growth. However, excess salts will hinder plant growth by affecting the soil-water balance. The electrical conductivity measurement detects the amount of cations or anions (salts) in solution; the greater the amount of anions or cations, the greater the electrical conductivity reading [2].

The Soil Organic Matter (SOM) is a product from the decomposition of plant residues, roots and soil organisms whether living or dead. The soil organic matter consists of humic and fulvic acids and humin, which contain functional groups such as R-COO-, R-C=O, R-COH, R-SH and others. Three macro plant food nutrients (N, P and S) are constituents of soil organic matter. Many methods are available for measuring SOM; wet oxidation and loss-on-ignition among the methods. From these methods Loss-On-ignition is an inexpensive, convenient and accurate method for estimating SOM, which involves combusting samples at high temperatures and measuring weight loss. Loss on Ignition (LOI) analysis is used to determine the organic matter content (%SOM) of a soil sample. LOI calculates %SOM by comparing the weight of a sample before and after the soil has been ignited. Before ignition the sample contains SOM, but after ignition all that remains is the mineral portion of the soil. The difference in weight before and after ignition represents the amount of the SOM that was present in the sample.

Total soil organic matter is estimated by Loss-On-Ignition (LOI). The method described is a procedure in which a soil sample is dried at 105°C and then ashed at 500°C. The loss in weight between 105°C and 500°C constitutes the organic matter content (Abella, 2007). Percent organic matter in the soil is determined by the formula:

$$\%SOM = [(W_{105} - W_{500}) \times 100] / W_{105}$$

Where: W_{105} is the weight of soil at 105°C and W_{500} is the weight of soil at 500°C.

The concentration of SOM in soils generally ranges from 1% to 6% of the total topsoil mass for most upland soils. Soils whose upper horizons consist of less than 1% organic matter are mostly limited to desert areas. Plants get nutrient from environmental compartment (soil, water and air). But they are not perfectly selective only to essential nutrients they may take up metals like heavy metals that are toxic even at low level. Soil is considered a critical environment as it accumulates pollutants that can be dispersed in it, both naturally and by various anthropogenic activities metals present in the soil fractions vary in degree of mobility. Their bioavailability is regulated by soil properties (physical, chemical and biological processes) and interactions between them [3].

Changes in the chemical properties of the soils greatly affect concentration of free metals and result in changes in their availability for plants. With increasing pH, contents of organic matter and clay; solubility of most metals are decreased due to their increased tendencies for adsorption [4]. Plants readily assimilate elements through the roots. The direct contact between the plant root and soil allows most metals to enter the plant tissue through uptake of water and nutrients by plants, ion exchange at cell wall and other complicated metabolic mechanisms.

Based on their importance, elements can be essential (such as K, Mg, Ca, Mn, Fe, Co, Cu and Zn) and they are very important for

growth and health, or they may be non-essential (such as Cd, Ag and Pb). Based on the amount needed nutritionally minerals are grouped into macro-minerals and trace. Elements such as Mn, Fe, Co, Cu, Zn, Se, Mo, F and I are essential trace elements, while elements like Ca, Mg and K are grouped under essential macro elements [5].

Macro-nutrients, micronutrients and toxic elements

These metals are required by body in good quantities for proper metabolism and functioning of body organs. These include calcium, magnesium, sodium and potassium. Micronutrients are needed in very small amounts. Their adequate concentrations in plants are generally below the 100 parts per million levels. The essential micronutrients are zinc, iron, manganese, boron, chlorine, copper, molybdenum, cobalt, vanadium, silicon, nickel Heavy metal is the generic term for metallic elements having an atomic weight higher than 40.04 [6]. Plants are sensitive to environmental conditions and they accumulate these heavy metals in their harvestable and intensity of this uptake process can change the overall elemental composition of the plant. Some of the heavy metals namely Pb, Cd, As and Hg are not essential for plants and these are insidiously toxic to mammals [7].

Research Methodology

Equipment

Polyethylene plastic bags were used to pack the soil samples. A drying oven was used to dry soil samples. A digital analytical balance (Mettler Toledo, Model AG204) with ± 0.0001 g precision was used to weigh soil samples. 250 mL round-bottomed flasks fitted with reflux condensers were used in Kjeldahl (England) apparatus to digest the dried and powdered soil samples [8]. A refrigerator (Hitachi, Tokyo, Japan) was used to keep the digested sample until analysis. Agilent model 4200 (USA) Microwave Plasma Atomic Emission Spectroscopy (MPAES) was used for analysis of the metals (K, Mg, Ca, Fe, Mn, Zn, Cu, Pb, Cd). A ceramic mortar and pestle (USA) was used for grinding and homogenizing the soil samples. Conductivity meter and pH meter (Romania) were used for measuring electrical conductivity and pH of the soil samples [9].

Reagents and chemicals

Reagents used in the analysis were all analytical grade. (69%-72%) HNO_3 (Spectrosol, BDH, England) and 70% HClO_4 were used for digestion of soil samples. Strontium nitrate (98%, Aldrich, Milwaukee, USA) was used to avoid refractory interference (for releasing calcium and magnesium from their phosphates). Stock standard solutions containing 1000 mg/L, in 2% HNO_3 , of the metals K, Mg, Ca, Fe, Mn, Zn, Cu, Pb and Cd (Buck Scientific Puro-Graphictm) were used for the preparation of calibration standards and in the spiking experiments. Distilled water was used throughout the experiment for sample preparation, dilution and rinsing apparatus prior to analysis [10].

Apparatus

Apparatus such as volumetric flasks, measuring cylinder and digestion flasks were washed with detergents and tap water and rinsed with distilled water and soaked in 50% nitric acid for two days. They were then rinsed with distilled water three times and dried in an oven and kept in dust free place until analysis begins.

Description of sampling sites

The soil samples were collected from the most teff productive areas of three different localities of Amhara regional state of Ethiopia. Particularly from Bahir Dar, Bure and Debre Markos, which are located in the north western part of Amhara regional state. The geographical locations (latitude, longitude and elevation) of sampling sites are described as follows. Bahir Dar is located at latitude of 11°35'37.1" N and longitude of 37°23'26.8" E in the northern hemisphere. Bahir Dar is located at the exit of the Abbay from Lake Tana at an altitude of 1,820 meters above sea level. The city is located approximately 578 km north-west of Addis Ababa. DebreMarkos is a city in north-west of Ethiopia. It is located in the Misrak Gojjam Zone of the Amhara administrative region, it is located at a latitude and longitude of 10°20'N 37°43'E coordinates and an elevation of 2,446 meters above sea level. Debre Markos is located approximately 306 km far apart from Addis Ababa [10]. Bure is a town in western Ethiopia located in the Mirab Gojjam Zone of the Amhara region, this town is located at a latitude and longitude of 10°42'N 37°4'E with an elevation of 2091 meters above sea level. Bure is located approximately 414 km far apart from Addis Ababa. The reason for selection of these places was based on the availability of the teff and its popularity in consumption.

Sample collection and preparations

The soil samples were collected from the base of the uprooted plant by auger and properly labeled and packed in polyethylene bags. Each soil sample was air dried at ambient temperature for three days and then ground into powder using acid washed commercial mortar and pestle and sieved to 0.425 mm mesh. The sieved soil samples were stored in the polyethylene bags and placed in desiccators until the time of digestion.

Soil pH determination

Soil pH was measured in a suspension (1:2.5, w/v) of the soil and distilled water. 5 g of air-dried soil (<0.425 mm) was weighed and

transferred to a 100 mL beaker into which 12.5 mL distilled water was added. Then, the mixture was stirred and the pH was measured after allowing the suspension to stand for 10 min at room temperature.

The electrical conductivity determination

The electrical conductivity of the soil samples was measured in suspension (1:2.5 w/v) of the soil distilled water. 5 g of air-dried soil (<0.425 mm) was weighed and transferred to a 100 mL beaker into which 25 mL distilled water was added. The mixture was stirred and allowed to stand for 15 min at room temperature and the electrical conductivity was measured.

Soil organic matter determination

Soil organic matter content was determined using the method of loss on ignition. 5 g of the soil sample, which was dried in an oven at 100°C for 15 min was accurately weighed into a pre-weighed crucible. Then the crucible with soil was placed in a muffle furnace and heated at 500°C for 3.5 h. The sample was then taken from the furnace and placed in desiccators to cool. Then the sample was reweighed and the percentage of organic matter content was calculated.

Optimization of the digestion procedure for soil samples

A 0.5 g of crushed, powdered, sieved and homogenized soil samples were weighed and transferred to a 250 mL round bottom flask. Different digestion procedures were carried out for the teff samples using HNO₃ and HClO₄ acid mixtures by varying volume of the acid mixture, digestion time and digestion temperature. Optimized procedures were selected based on the usage of lesser reagent volume, shorter digestion time and reasonable mild temperature for obtaining clear and colorless solutions of the resulting digests. Based on this fact the optimized digestion conditions for the soil samples in this study were (5 ml HNO₃: 1 mL HClO₄) volume ratio of reagents, 240°C digestion temperature and 2:30 h digestion time and are shown in Table 1.

Trials	Reagent volume (mL)			Temperature (°C)	Time (h)	Results
	HNO ₃	HClO ₄	Total			
1	1	1	2	240	0.104167	Yellow with suspension
2	2	1	3	240	0.104167	Cloudy yellow
3	3	1	4	240	0.104167	Nearly colorless
4	4	1	5	240	0.104167	Slightly colorless
5	5	1	6	240	0.104167	Clear colorless
6	6	1	7	240	0.104167	Clear colorless
7	3	2	5	240	0.104167	Slightly colorless
8	4	2	6	240	0.104167	Nearly colorless
9	4	1	5	240	0.104167	Nearly colorless
10	5	2	7	240	0.104167	Clear colorless

11	5	1	6	240	0.020833	Yellow with suspension
12	5	1	6	240	0.041667	Yellow with suspension
13	5	1	6	240	0.0625	Cloudy light yellow
14	5	1	6	240	0.083333	Light yellow
15	5	1	6	240	0.104167	Clear colorless
16	5	1	6	240	0.125	Clear colorless
17	5	1	6	150	0.104167	Cloudy yellow with suspension
18	5	1	6	180	0.104167	Cloudy yellow with suspension
19	5	1	6	210	0.104167	Slightly yellow
20	5	1	6	240	0.104167	Clear colorless
21	5	1	6	270	0.104167	Clear colorless
22	5	1	6	300	0.104167	Clear colorless

Table 1: Reagent ratios and volumes, temperature and time attempted during optimization of digestion of 0.5 g of the soil sample.

The digested solutions were allowed to cool and 5 mL of distilled water was added to dissolve the precipitate formed on cooling and gently swirled and filtered into 50 mL volumetric flask through Whatman no. 42 filter paper. The clear solution then was diluted up to 50 mL with distilled water and stored until analysis by microwave plasma atomic emission spectroscopy.

Digestion of the soil samples

0.5 g of crushed, powdered, sieved and homogenized soil samples were weighed and transferred to a 250 mL round bottom flask. To this, 5 mL of HNO₃ and 1 mL HClO₄ were added. The digested solutions

were allowed to cool and 5 mL of distilled water was added to dissolve the precipitate formed on cooling and gently swirled and filtered into 50 mL volumetric flask through Whatman no. 42 filter paper. The clear solution then was diluted up to 50 mL with distilled water. Each soil samples were digested in triplicate. Digestion of a reagent blank was also performed in parallel with the samples. The solutions were used for the analysis of the soil metal concentrations for K, Ca, Mg, Fe, Cu, Zn, Mn, Pb, and Cd by Microwave Plasma Atomic Emission Spectroscopy (MP-AES).

Results and Discussion

The values of soil pH, % SOM and EC of the soils of the three different places were presented in Table 2. The results of each soil's pH, %SOM and EC are discussed below.

Sampling site	pH ± SD	EC ± SD (mS/m)	SOM ± SD (%)
Bahir dar	6.48 ± 0.34	71.4 ± 0.40	17.7 ± 0.37
Bure	6.95 ± 0.07	51.1 ± 0.50	11.9 ± 0.96
Debre Markos	6.92 ± 0.05	18.4 ± 0.97	13.3 ± 0.18

Table 2: The value (mean ± standard deviation (SD), n=3) of pH, electrically conductivity (mS/m) and organic matter (%) of the soil.

The higher the soil organic matter content, the higher the ability of that soil to retain metals within it. The result of the analysis showed that the highest % soil organic matter was obtained in the soil collected from Bahir Dar followed by that of Debre Markos and the lowest was obtained in the soil Bure. So based on the result, the metals are more retained in the soil of Bahir Dar. Therefore, the bioavailability of metals in the soil for the plant species becomes low when the organic content of the soil is high due to the adsorption reaction of metals on it.

Most plants grow best in slightly acidic soils (pH 6.0-7.0). In this pH range, nearly all plant nutrients are available in optimal amounts. Soils with a pH below 6.0 are more likely to be deficient in some available nutrients. Ca, Mg, and K are especially deficient in acid soils. Metal solubility tends to increase at lower pH and most of the mobility of metals is reduced with increasing soil pH because of the precipitation as insoluble hydroxides, carbonates and organic complexes. Usually, the intensity of root uptake of metal by plants decreases with increasing soil pH. Low soil pH value determines the activity of many metal ions in the water contained in the pores of the soil, affecting their bioavailability. The result showed that the soil pH for the three study areas is within the range of 6.48-6.95, which

categorizes the soils under weakly acidic soils. According to most plants grow best in this pH range.

Soil Electrical Conductivity (EC) is a useful indicator in managing agricultural systems. EC directly affects plants growing in the soil or media. EC range of 0-100 mS/m indicates good soil health. Soils that have EC of less than 100 mS/m is considered to be nonsaline. Soil that has EC of more than 100 mS/m is considered to be saline. Important microbial processes such as nitrogen cycling, production of nitrous gases and other N oxide gases, respiration and decomposition of organic matter are affected. Populations of parasitic nematodes and loss of nitrogen can be higher in these soils. The result showed that the soil EC of the three study areas were within the range of 18.4 to 71.4 mS/m, indicating that soil environment is good for the plant growth. EC does not provide a direct measurement of specific ions or salt compounds it has been correlated to concentrations of nitrates, potassium, sodium, chloride, sulfate and ammonia.

In general, loading, and accumulation of metals in the soil depend on different factors such as the chemical form of elements, pH, organic matter content, texture and Cation Exchange Capacity (CEC) of the soil. With increasing pH, organic matter content, CEC and clay, the percentage and availability of the metals are reduced. In addition, the existence of carbonate, sulfate and phosphate and sulfide in the soil creates an increase in the metal precipitation and consequently decrease their availability to the plants.

Calibration of the instrument

Calibration curves were prepared to determine the concentration of metals in the sample solution. The instrument was calibrated using

four series of working standards. The calibration graphs and correlation coefficients of each of the elements were determined by plotting working standards concentration of metals versus their corresponding emission intensity.

Evaluation of analytical method

Recovery is one of the most commonly used techniques utilized for validation of the analytical results and evaluating how far the method is acceptable for its intended purpose. The validity of the digestion procedures were assured by spiking the samples with a standard solution of known concentration of the target analytes and the percentage recoveries lies from 92%-104%, which were within the acceptable range. The spiked soil samples were digested in triplicate following the same procedure used for digestion of the fruit and the soil samples. The resulting digest of the spiked samples was analyzed for their respective metal contents using MP-AES and percent recoveries were calculated for the soil samples.

Level of metals concentration in the soil samples

Metals may enter the human body through inhalation of dust, direct ingestion of soil and consumption of food plants grown on metal contaminated soil. The most important pathway through which human exposure to metals takes place is soil-plant-human (food chain) and soil human (incidental soil ingestion) relationship. Out of the two soil-to-plants transfer are the key components of human exposure to metals. Therefore, analyses of the level of metals in soil are important. The results of the analysis for the soil samples are given in Table 3.

Sampling sites	Mean concentrations of the metals (mg/kg)								
	K	Mg	Ca	Mn	Fe	Cu	Zn	Pb	Cd
Bahir Dar	3701 ± 3	1202 ± 2	6201 ± 2	1449 ± 1	69714 ± 3	28.1 ± 3	132 ± 1	3.10 ± 0.4	1.09 ± 0.1
Bure	3635 ± 2	1109 ± 1	2189 ± 3	1023 ± 6	75823 ± 3	56.9 ± 2	155 ± 3	4.33 ± 1.0	2.15 ± 0.3
Debre Markos	3366 ± 4	1408 ± 1	5062 ± 4	1115 ± 2	76731 ± 2	30.5 ± 1	168 ± 2	5.25 ± 0.3	3.14 ± 0.2
Overall mean	3567 ± 3	1239 ± 1	4484 ± 3	1196 ± 3	74089 ± 3	39.0 ± 2	152 ± 2	4.20 ± 0.6	2.10 ± 0.2

Table 3: Mean concentration of metals (mg/kg), (mean ± SD, n=3) in the soil samples.

As shown in Table 3, the results showed that the mean concentration of the Fe content of the soils is the highest of all the other studied metals in all of the three sampling areas. The overall mean concentrations of the metals collected from the three sampling areas in mg/kg can be ordered as Fe (74089 ± 3) > Ca (4484 ± 3) > K (3567 ± 3) > Mg (1239 ± 1) > Mn (1196 ± 3) > Zn (152 ± 2) > Cu (39.0 ± 2) > Pb (4.20 ± 0.6) > Cd (2.10 ± 0.2). On the other hand, the soil samples were not free from the level of the toxic heavy metal Pb and Cd ranges from 3.10-5.25 mg/kg of Pb and from 1.09-3.14 mg/kg of Cd collected from the three sampling areas. This may be caused due to different agricultural practices like the usage of fertilizers.

Statistical analysis

Statistical method was used to check whether there is a contribution from the random errors for the difference in results of analysis or not. If there are differences, statistical analysis will tell us whether the differences are significant or not at a specified confidence level. One-Way Analysis Of Variance (ANOVA) was used to perform the statistical analysis soil samples as independent and concentration of the metals as a dependent variable to test whether there are significant differences between means of each soil samples collected from the three sampling areas. There was a significant difference between the metal concentrations of the soils collected from the three sampling areas at a confidence level of 95%.

Conclusion

The levels of essential and trace metals (K, Ca, Mg, Fe, Zn, Cu, Mn, Pb and Cd) in the soil samples collected from the three sampling areas were determined by using MP-AES. The study showed that the metals were present at different concentrations in the samples from different sites. Comparable results were found with some of the values reported in the literature and for Cd and Pb metals; the concentrations slightly exceeded the permissible levels by WHO/FAO, which could be attributed to the agricultural practices employed such as the use of fertilizers and herbicides. In this study soil pH, soil organic matter and soil electrical conductivity were also determined. Therefore, this study will give brief information about the essential and trace metals of the soil samples collected from the three different sampling areas.

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