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Analysis of some Heavy Metals of Soil Samples in the Bahir Dar Textile Industry, Northern Amhara, Ethiopia

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Abstract

The research investigated the concentration of some heavy metals in a soil sample from the Bahir Dar textile industry by using Flame Atomic Absorption Spectroscopy (FAAS). The results obtained from the present study showed that the overall concentration of seven heavy metals (Cr, Cd, Zn, Fe, Pb, Mn, and Cu) in the range of 65.60-132.3, 10.783-18.967, 174.467-220.267, 3119.366-3147.933, 105.466-234.50, 656.40-709.667 and 55.6-80.6 (mg /Kg) in the soil samples respectively. In general, the levels of metals in soil samples collected from all the sampling sites were found to decrease in the order: Fe >Mn> Zn >Pb> Cr > Cu > Cd. Some of the concentrations of heavy metals (Zn, Mn, Fe, Cu) in studied soil samples found below the maximum limit that proposed for agricultural soil by FAO /WHO and some of the heavy metals (Cr, Cd, Pb) that the concentrations were found above the maximum limit. Results of heavy metal concentrations in the soil samples under-investigated indicate that industrial activities most important sources for some heavy metals in the soil samples of the Bair Dar textile industry.

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Contaminated Soil, Heavy metals, FAAS, Textile Industry

Introduction

Pollution in recent years has increased considerably as a result of increasing human activities such as burning of fossil fuels, industrial and automobile exhaust emissions which were identified as primary sources of atmospheric metallic burden [1]. Soil is composed of mineral constituents, organic matter (humus), living organisms, air and water, and it regulates the natural cycles of these components. Besides the parent material, the sources of contamination in soils are multifarious, and include agricultural and industrial pollution [2]. Soil is mixture of

minerals, organic matter gases, liquids and myriad of micro and macro organisms that can support plant life. Soil as a general term usually denotes the unconsolidated thin, variable layer of mineral and organic material usually biologically active that covers rest of the earth land surface [3]. Soil characteristic parameters have always been used to define quality of soil and often with biological processes influence soil fertility in a variety of ways, each of which can an ameliorating effect on the main soil-based constraints to productivity. Soil texture provides the classes of particle size possessed by a soil which plays a significant role in the development and

stability of soil structure [4]. Soil pollution by heavy metals is a significant environmental problem worldwide [5]. In particular, heavy metal pollution of surface soils due to intense industrialization and urbanization has become a serious concern in many developing countries [6, 7]. The final disposal of industrial sludge in Ethiopia has become a critical issue due to public concern and the limited availability of land. Soil is unconsolidated minerals and organic material found on the immediate earth surface that serves as a natural medium for plant growth and other developmental activities [8]. Pollution of heavy metals, directly and indirectly, affects human health. These substances adversely affect the productivity of soils, plants, animals and the entire environment if exceed certain limits [9]. Contaminations of soil by heavy metals are a global concern and present a serious problem. The quality and health of soil determine agricultural sustainability and environmental quality which jointly determines plant, animal and human health. Therefore, they can affect human and animals' health, and also environmental quality [10]. Metals occur naturally in the earth's crust, and their contents in the environment can vary between different regions resulting in spatial variations of background concentrations [11]. Contamination from industrial activities or byproducts can increase the natural levels of heavy metals in soil and water, creating a health hazard to people, livestock and plants. However, there is no information on the contents of heavy metals in sludge, water and soil around the industry. Literature review revealed that no work has been done concerning the concentration of heavy metals in the soil environment of Bahir Dar Textile Industry. Therefore it is the intention of this project to fill this research gap. The study will provide the baseline data of the levels of cadmium (Cd), Zinc (Zn), copper (Cu), chromium (Cr), lead (Pb), manganese (Mn) in the soil environment. This will help to make a basis for further studies/ monitoring of their concentrations in the soil. The information generated can be used by other scholars doing similar studies as a source of information.

Materials and Methods

Experimental

Description of sampling area

Bahir Dar, the capital of Amhara National Regional State, is situated on the southern shore of Lake Tana, the source of Blue Nile River, approximately 565 km northwest of Addis Ababa at an altitude of 1801 m.a.s.l,

having latitude of 11°03'N and longitude of 37°10'E. The study area experiences average annual rainfall that ranges from 1200 to 1600 mm and it has a mean annual temperature of 26°C. It is a rapidly expanding town with commercial centers, small industries, and residences in all sectors of the Town [12]. Location of the study area is shown in Figure 1.

Apparatus and Instruments

Electronic analytical balance (AA-200DS, Denver Instrument Company) was used for weighing samples of soil. Atomic Absorption Spectrophotometer (AA-500AFG, UK) equipped with deuterium background correctors and Hollow Cathode Lamp of each metal was used for the analysis of heavy metals. Digestive furnace (model: KDN-20c, China), Kjeldahl tubes fitted with a reflux condenser were used in the Kjeldahl digestion block apparatus to digest soil samples.

Chemicals and Reagents

HNO₃ (65.0 %, UNI-CHEM[®] Chemical reagents, India), HClO₄ (70.0-72.0 %, UNI-CHEM[®] Chemical reagents, India) and H₂O₂ (35.0 %, UNI-CHEM[®] Chemical reagents, India) were used for the sample digestions. 1000 ppm stock standard solutions of the heavy metals Fe, Zn, Mn, Cu, Cr, Cd, and Pb were used to prepare calibration standard solutions and ZnSO₄·7H₂O, CuSO₄·5H₂O, K₂CrO₄ (99.5 %, UNI-CHEM[®] Chemical reagents, India), Pb (NO₃)₂ (99.5 %), Cd (NO₃)₂ (97 %, UNI-CHEM[®], NICE, chemicals Pvt. Ltd, India), MnSO₄·H₂O (99 %, UNI-CHEM[®], NICE, chemicals Pvt. Ltd, India) were used to prepare spiking standard solutions.

Soil sampling

The soil samples were collected from topsoil at the depths of 0–20 cm by digging stainless steel knife from inside the industry area after the sludge samples were collected in December, 2018. Three sub-sites were taken due to the closeness to the waste water treatment plant, sedimentation tank of the treatment process and also to the disposal area of sludge. Three soil samples were collected near to the sedimentation tank of the treatment process (T1), waste water treatment plant (T2) and disposal area of sludge (T3) at a distance of 150 (between T1 and T2) and 200 (between T1 and T3) meters from each other. Three soil samples (representative sample) were randomly collected from each of the three sub-sites in the industrial areas and

pooled together to obtain a composite sample. Finally, three soil samples one from each stated areas were transferred in to polyethylene bags and transported to the Ambo University laboratory. The samples were air dried for three days (72 hrs.), ground with porcelain mortar and pestle, passed through 0.5 mm sieve, and then kept in clean polythene bags for further analysis.

Procedures

Preparation of Standard Stock Solutions

The standard stock solutions of Zn, Cu, Cr, Pb, Cd, Fe, and Mn were prepared by dissolving the appropriate amount of the respective metal salt, 4.3987 g of ZnSO₄.7H₂O, 3.9295 g of CuSO₄.5H₂O, 3.7348 g K₂CrO₄, 1.5980 g of Pb (NO₃)₂ and 2.1032 g of Cd (NO₃)₂, 7.162 g of Fe (NH₄)₂(SO₄)₂.6H₂O and 3.118 g of MnSO₄.H₂O in 1000 mL volumetric flask and filled to the mark with distilled water to prepare a 1000 ppm stock solution of respectively.

Working Intermediate Metal Standard Solutions

For the determination of metals in soil samples, 10 mg/L intermediate standard solution in 100 mL volumetric flask was prepared from 1000 mg/L stock solution.

Standard Solutions for calibration

For calibration of the flame atomic absorption spectrophotometer (FAAS) a series of blank and five standard solutions were prepared for each metal from their respective working standard solutions (10 mg/L).

Spiking Metal Standard Mixture Solution

A mixture of standard solution containing 2 mg/L of each Zn and Mn, 2.023 mg/L Cu, 9 mg/L Pb, 2.25 mg/L Cd, 4.5 mg/L Cr and 3.375 mg/L Fe was prepared. This mixture of standard solution was obtained by taking 0.1 mL of each Mn and Zn, 0.101 mL Cu, 0.225 mL Cr, 0.113 mL Cd, 0.169 mL Fe, 0.45 mL Pb of each metal stock standard solution (1000 mg/L) in to 100 mL volumetric flask and diluting to the mark with double distilled water.

Laboratory sample analysis

Digestion of Soil Samples

For the digestion of soil samples, a subsample of 0.500 g was taken from each sample and placed into Kjeldahl

digestion flasks. To each sample, H₂O₂ (0.500 mL), conc. HNO₃ (0.500 mL) and conc. HClO₄ (6.500 mL) mixture was added for digestion. The sample was swirled gently to homogenize the mixture then it was fitted to a reflux condenser and digested continuously for 2:30 hours on the Kjeldahl digestion block by setting the temperature dial at 250°C. Each soil sample was digested in triplicate and after digestion; the digested sample mixture was left to cool to room temperature. Finally, each mixture was filtered through Whatman filter paper and the filtrate was transferred into a 50 mL volumetric flask and diluted to the mark and the final solution was kept in the refrigerator until analysis after the solution was transferred from the 50 mL volumetric flask into 50 mL polyethylene bottles.

Some Validation of Method (LOD, LOQ, Accuracy and Precision)

The proposed method was validated by evaluating different parameters as limit of detection (LOD), limit of quantitation (LOQ), accuracy (in terms of recovery) and precision (in terms of repeatability) [13].

Limit of detection

Limit of detection (LOD) is the minimum concentration of analyte that can be detected and LOD for each metal was determined from analysis of three replicates of method blanks which were digested in the same digestion procedure as the actual samples [14]. It was calculated as:

$$LOD = 3 \times S_{bl} \dots\dots\dots 1$$

Where S_{bl} is the standard deviation of the method blank

Limit of Quantification

Limit of quantification (LOQ) is the lowest concentration of analyte that can be determined with an acceptable level of uncertainty. LOQ was obtained from the analysis of three replicate of method blanks which were digested in the same digestion procedure as the actual samples. LOQ was calculated as ten times the standard deviation of the blank.

$$LOQ = 10 \times S_{bl} \dots\dots\dots 2$$

Where S_{bl} is the standard deviation of the method blank [14]

Precision and accuracy

Precision is the extent of the consistency of results as they are obtained during repeated applications a specified determination method. It was evaluated regarding repeatability by estimating the relative standard deviation (RSD) of the recovery percentage for each spiked level. Accuracy was evaluated through recovery studies of sample spikes. Triplicate samples were prepared and triplicate readings were obtained. The relative standard deviations of the sample were obtained as:

$$RSD (\%) = \frac{\text{Standard deviation}}{\text{Mean value}} \times 100 \dots\dots\dots 3$$

The percentage recoveries of the analyte were calculated to evaluate the accuracy of the analytical procedure. Recovery was then calculated as:

$$\text{Recovery} (\%) = \frac{\text{Conc.in spiked sample} - \text{Conc.in unspiked sample}}{\text{Amount added}} \times 100 \dots\dots\dots 4$$

Heavy metal analysis of soil sample

The digested soil sample was analyzed for copper (Cu), cadmium (Cd), manganese (Mn), chromium(Cr), lead(Pb), iron(Fe) and zinc (Zn) by atomic absorption spectrometer (AAS) after all parameters (lamp alignment, wavelength and slit width adjustment) were optimized for maximum signal intensity and sensitivity of the instrument. Triplicate determinations were carried out on each sample. The concentrations of samples in mg/L were converted to mg /Kg using the formula [15]:

$$\text{Concentration in mg /kg} = \frac{\text{Concentration in mg/L} \times \text{volume in litre} \dots\dots\dots 5}{\text{Mass of sample in kilogram}}$$

Statistical analysis

One-way analysis of variance (ANOVA) was used to evaluate the significant differences in the mean values of heavy metals among groups of soil sample. A probability level of $P < 0.05$ was considered statistically significant. All statistical analyses were done by Microsoft Office Excel-2007, IBM SPSS Version 20 and Origin 8.1

software packages. Pearson’s product-moment correlation r was used to express the relationship between levels of heavy metal concentrations.

Results and Discussions

Some Validation of Method (LOD, LOQ, Accuracy and Precision)

Limit of Detection (LOD) and Limit of Quantification (LOQ)

LOD and LOQ for each metal were determined from the analysis of triplicates of method blanks which were digested in the same digestion procedure as the actual samples. For the present study, three reagents blank solutions were digested for soil sample and each of the samples was analyzed for metal concentrations of Mn, Fe, Cu, Zn, Cr, Pb and Cd by FAAS. The standard deviations for each element were calculated from blank measurements.

From Table 1, The limit of detection (LOD) values for all the metals analyzed in the soil samples ranged from 0.036 mg /L for Cu to 0.174 mg /L for Cd and the limit of quantification (LOQ) values for all the metals analyzed in this samples also ranged from 0.12 mg /L for Cu to 0.58 mg /L for Cd.

Accuracy and precision

In this study, the recovery test was done by spiking a suitable known quantity of metal standard solution into a test portion of the sample. For doing so, each sample was spiked in triplicates and the spiked and non-spiked samples were digested and analyzed using the same analytical procedure [16]. The percentage recoveries of soil samples are given in Table 2.

As can be seen from Table 2, the percentage recovery of the metal analysis in the soil ranged between 91.857–95.926 % and the RSD values ranged between 0.368 – 2.730 %. The matrix spike recovery obtained in this study falls within the normal acceptable range of 90–110 % for a good recovery study. The RSD values of the samples were < 10 %, indicating that the proposed method was precise.

Concentration of heavy metals in soil samples

The mean concentrations of heavy metals in the soil samples are shown in Table 3 and Figure 2. In the

present study, cadmium was the metal present in the lowest concentrations, being in the range from 10.783 to 29.667 mg /kg in the soil samples analyzed and iron was the metal present in the highest concentrations ranging from 3119.366 to 3147.933 mg /kg in the soil samples.

The high concentration of Cd obtained in this study was 29.667 mg /kg which was at site2, and the lower level was found in the site3 (10.783 mg /kg). The natural background level of Cd in agricultural soil in China is \leq 0.20 mg/kg [17]. The values of Cd found in the present investigation were higher than values 0.01 to 2 mg /kg set by [18].

The concentration of copper (Table 3) in soil samples ranged from 55.6 to 88.8 mg /kg. The maximum permissible level of copper concentration in agricultural soil in some European countries is 100 mg /kg [19]. So, the concentration of copper in this study found to be below the permissible limit set by [19]. Other studies of soils near textile industries have reported that the levels of copper in the range of 200 to 250 mg /kg[20].

The concentrations of zinc were in the range of 174.467 to 220.267 mg /kg. The lowest zinc content was obtained in soil collected from site 2 and the highest in soil collected from site 1. Zinc is widely used in industries to make paint and dyes [17]. The concentration of Zn in this study was lower than the standard limit set by other

literatures [21,19]. The concentration levels of chromium were range from 65.6 to 132.3 mg /kg; the target value recommended by FAO/WHO standard is 50 mg /kg [22]. So, the concentration of Cr in this study is higher than the standard limit set by FAO/WHO. This data showed that the chemicals such as dyes (Cr pigment) and other finishes used on the fabrics can lead to an increase in the concentration of chromium metals in the soils.

The concentration of manganese found in this study was ranging from the 656.4 to 709.667 mg /kg and which were lower than the FAO /WHO, 2000 mg /kg. The value of the concentration of manganese in the present study was below in the maximum limit set FAO /WHO [22, 19]. This indicated that the chemical used in the industry was not significantly contributed to increase the concentrations of Mn heavy metals in the soil.

The result of lead (Pb) concentration was in the range of 105.466 to 234.5 mg /kg. The permissible limit for lead set by FAO /WHO in soil is 100 mg /kg [22]. The average concentration (152.622 mg /kg) of lead obtained in this study was higher than FAO /WHO recommended maximum limit for soil (100 mg /kg). However, other studies reported very high levels of lead in soils ranging between 3500-6860 mg /kg [23]. This also due to the use of chemicals such as dyes and other incoming fibers in the industry.

Table.1 Limit of detection (LOD) and limit of quantification (LOQ) of the soil matrix spike sample for the determination of metals

Elements	LOD (mg/L)	LOQ (mg/L)
	Soil	Soil
Cu	0.036	0.12
Cr	0.087	0.13
Zn	0.039	0.29
Mn	0.087	0.27
Pb	0.063	0.21
Cd	0.174	0.58
Fe	0.093	0.-31

Table.2 Percent recovery of metals in soil samples (mean \pm SD, n = 3)

Elements	Conc. in unspiked sample (mg/L)	Amount added (mg/L)	Conc. in spiked sample (mg/L)	Recovery (%)	RSD (%)
Cd	0.290 \pm 0.174	0.18	0.462 \pm 0.169	95.556 \pm 0.352	0.368
Cu	0.887 \pm 0.033	0.16	1.037 \pm 0.127	92.593 \pm 0.854	0.922
Zn	1.745 \pm 0.034	0.16	1.892 \pm 0.054	91.857 \pm 2.120	2.308
Cr	0.902 \pm 0.041	0.36	1.243 \pm 0.167	94.722 \pm 1.900	2.006
Pb	2.345 \pm 0.182	0.72	3.024 \pm 0.293	94.306 \pm 1.928	2.044
Mn	7.094 \pm 0.014	0.16	7.247 \pm 0.134	95.625 \pm 2.611	2.730
Fe	31.194 \pm 0.059	0.27	31.482 \pm 0.101	95.926 \pm 2.200	2.293

Table.1 Heavy metal concentrations (mean \pm SD, n = 3, mg /kg dry weight) in soil samples

Heavy Metals	Site 1	Site 2	Site 3
Cd	18.967 \pm 3.166	29.667 \pm 17.402	10.783 \pm 2.532
Cu	88.800 \pm 3.251	80.600 \pm 5.212	55.600 \pm 3.923
Zn	220.267 \pm 3.252	174.467 \pm 3.430	213.467 \pm 2.060
Cr	65.600 \pm 0.173	88.933 \pm 4.648	132.300 \pm 4.677
Mn	691.700 \pm 0.700	709.667 \pm 1.026	656.400 \pm 3.799
Pb	105.466 \pm 2.419	117.900 \pm 0.520	234.500 \pm 8.248
Fe	3119.366 \pm 5.972	3147.933 \pm 20.935	3138.100 \pm 20.785

Table.2 Comparison of heavy metal concentrations in soil with the standard of FAO /WHO (2001) and Kabata-Pendias and Pendias (2001) (mg /kg)

Metals	Present study	Kabata-Pendias and Pendias [20]	FAO /WHO [22]
Cr	95.611	100	50
Cd	19.806	5	3
Zn	202.734	300	300
Fe	3135.133	NA	5000
Pb	152.622	100	100
Cu	75.000	100	100
Mn	685.922	NA	2000

Table.5 Metal to metal correlation coefficient matrix (r) of soil samples

	Mn	Cr	Zn	Cd	Pb	Cu	Fe
Mn	1						
Cr	-0.564	1					
Zn	-0.053	0.032	1				
Cd	0.558	-0.038	-0.051	1			
Pb	-0.738	0.953	0.296	-0.515	1		
Cu	0.556	-0.976*	-0.135	0.408	-0.939*	1	
Fe	-0.046	0.368	-0.501	0.468	0.240	-0.256	1

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

Figure.1 Map of the study area

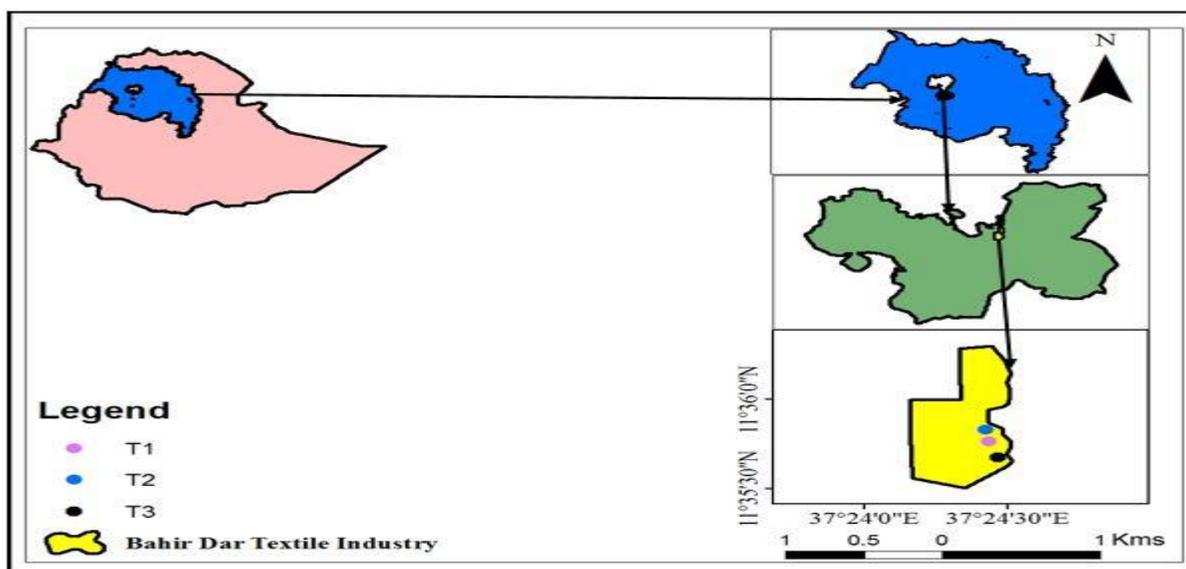
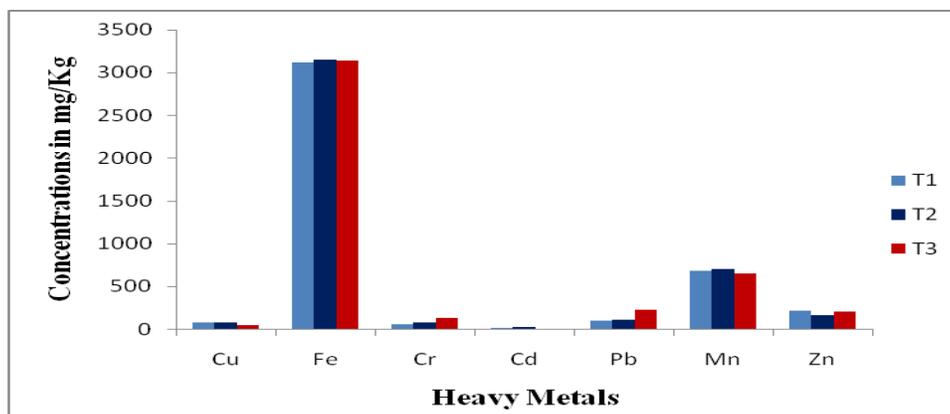


Figure.2 Mean concentrations (mean \pm SD, $n = 3$) of heavy metal in soil samples



The results in Table 3 and Figure 2 revealed that the concentration of iron (Fe) was highest among the heavy metals analyzed from all the sampling sites and the level obtained was found in the range of 3119.366 to 3147.933 mg /kg. At high concentrations, iron is responsible for anemia and neurodegenerative conditions in human beings [24]. Table 4, the concentration of iron obtained in this study was lower than FAO/WHO recommended maximum limit for soil (5000 mg /kg). So, the concentration of iron was found below the limit that was recommended by FAO/WHO [22]. Comparison of heavy metal concentrations in soil sample with the standard of FAO /WHO (2001) and Kabata-Pendias and Pendias (2001) showed in Table 4 below.

From the Table 4, some of the concentrations of studied heavy metals (Zn, Fe, Mn and Cu) were found below the maximum limit that was proposed by FAO/WHO and some of the heavy metals (Cr, Cd and Pb), that their concentrations were found above the maximum limit

Pearson's correlation analysis

Pearson's correlation coefficient was used to examine the relationship between the various heavy metals in the soil and sludge samples from all the sample sites. Table 5 showed that the correlation matrix of the relationship between heavy metals concentration of soil samples. Other study reported that high correlation coefficient (near +1 or -1) means a good relationship between two variables, and its concentration around zero means no relationship between them at a significant level of 0.05% level, it can be strongly correlated, if $r > 0.7$, whereas r values between 0.5 and 0.7 shows moderate correlation between two different parameters [25].

Correlation of heavy metals in soil samples

The results of the correlation coefficients (Table 5) showed that there was a strong positive correlation between Pb with Cr ($r = 0.953$). This strong positive correlation shows that the elements are closely associated, thus suggesting their common origin, but there was also a strong negative correlation between Cu with Cr ($r = -0.976$) and Pb with Mn ($r = -0.738$).

There were also moderate positive correlation Cd and Mn ($r = 0.558$) and Cu with Mn ($r = 0.556$), Fe with Cd ($r = 0.468$). Moderate negative correlations were found between Cr with Mn ($r = -0.564$), Pb with Cd ($r = -0.515$), Fe with Zn ($r = -0.501$). The other elements have

a weak negative or positive correlation indicating that the presence or absence of one element affects a lesser extent to the other.

Conclusions of the study are as follows:

All the metals investigated were found to be present in all the soil samples. The results obtained from the present study showed that the overall concentration of heavy metals (Cr, Cd, Zn, Fe, Pb, Mn, and Cu) in the range of 65.60-132.3, 10.783-18.967, 174.467-220.267, 3119.366-3147.933, 105.466-234.50, 656.40-709.667 and 55.6-80.6 (mg /Kg) in the soil samples respectively. The result from this study showed that the heavy metal concentration of soil in the studied area were in the order Fe > Mn > Zn > Pb > Cr > Cu > Cd. The concentration of Fe was several times higher than other metals. The concentrations of the metals in all the samples analyzed were near and above to the limit set by FAO/WHO and Kabata-Pendias and Pendias and some of the heavy metals (Cr, Cd, Pb) that the concentrations were found above the maximum limit. The level of cadmium was high in the soil samples; due to the sources of Cd in the soil include farmyard manure and atmospheric deposition and the chemicals such as dyes (Cr pigment) and other finishes used on the fabrics can lead to an increase in the concentration of chromium metals in the soils. The use of chemicals such as dyes and other incoming fibers in the industry, that causes to increase the natural levels of lead in the soil samples.

The analysis of soil also revealed that the chemical use of the industry could contribute to heavy metal contamination of the soil. Results of heavy metal concentrations in the soil samples under-investigated indicate that industrial activities most important sources for some heavy metals in the soil. One-way analysis of variance (ANOVA) was made at a 95% confidence level on soil samples. The results showed that there were significant differences ($p < 0.05$) in the concentrations of the heavy metals Cr, Zn, Cu and Mn among the analyzed soil samples while there was no significant difference ($p > 0.05$) in the concentrations of Cd, Fe, and Mn. From the present study, one can observe that there is a possibility of a contaminant in the soil of industrial areas where the industrial contribution is a major effect. This study also recommends further investigations on the contamination of the water by heavy metals. It is recommended that further studies should focus on the determination of heavy metals on different types of plants grown around Bahir Dar textile factory.

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