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Determination of Trace Elements Concentration in Soil Samples of Fika Agrarian Community of Yobe State, Nigeria, Using MP-AES Analytical Technique

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Abstract:
This study investigated the trace elements concentration in soil samples of Fika agrarian community of Yobe state, Nigeria. Representative soil samples from ten locations were collected from cultivated farmland at a depth of 0 to 20 cm using soil auger in the study area using purposive sampling technique and global Positioning System (GPS) device was used to mark the sampling location and elevation above sea level. The concentrations of Al, Cd, Cr, Co, Fe, Mn, Pb and Zn were determined using Microwave Plasma Atomic Emission Spectroscopy (MP-AES) analytical technique. The data obtained were subjected to statistical analysis to determine the trace elements concentration. Mean, Standard deviation and Pearson Product Moment Correlation coefficient (PPMC) were used to analyse the results. Based on the analysis, the result revealed that the mean elemental concentration was of the order of Pb (81.585) > Fe (53.907) > Al (3.941) > Mn (2.311) > Zn (0.500) > Cr (0.151) > Co (0.002) > Cd (0.001) in ppm respectively. PPMC analysis showed a strong correlation between Zn and Pb. In comparison with others studies against USDE international benchmark we found that all elemental concentrations are below the International benchmark except Pb in the study area which was found to be above the international threshold value. This could be due to the gypsum mining activity being carried out in the study area. Based on the result, it has been concluded that the agrarian soil of Fika is uncontaminated and thus recommended for cereal growth.

Keywords*: Trace element, fika, microwave-plasma atomic emission spectroscopy, soil contamination*

1. Introduction

Soil is conceivably the most endangered component of our environment which is open to potential contamination by a variety of different pollutants arising from majorly human activities such as nuclear, industrial, agriculture, among others (Djingova & Kuleff, 2000; Bermea, *et al*., 2002 & Kalantari, *et al*., 2006). Soil is a composite system which consists of organic and inorganic matter that directly and indirectly supports plant and animal life and is a crucial component of our rural and urban environments (Emanuel, 2015). The accumulation of certain elements, in particular trace elements in agricultural soil is of increasing concern due to food safety issues, potential health risks and its detrimental effects on soil ecosystems (McLaughlin *et al*., 1999).

The main natural sources of elements in soils are weathering of parent material and soil erosion (Emmanuel, 2014). The trace elements are primary content of rocks released due to weathering processes and their moderate concentration levels are usually safe but an increase in concentration of these elements in the environment can be significantly destructive to plants and animal life (Goleakar *et al*., 2013; Macfarlane & Burchett, 2000).

Baseline data on trace element levels in soil is beneficial to all agrarian communities particularly in fertilizer applications, with view to identifying suitable agricultural activities on soil as well as in resource identification, management and land use planning (Wilcke, *et al*., 1998). The association of elements in varying concentrations in soils is indicative of the mineral content, which in turn serves as a measure of soil fertility (Abubakar, 2007). Hence, there is the crucial need to ensure that adequate information on the elemental concentration of soil is available to the agrarian communities so as to identify which part of the soil is best for planting certain crops and the types of fertilizer to be applied to the soil (Emanuel, 2015).

Despite the importance of this data on trace elements to agrarian communities, none of such study was conducted in Fika local government area of Yobe state. Notwithstanding, only few relevant studies were conducted in the Northeast

geopolitical zone, Nigeria. Most of the researches on trace elements concentration in Nigeria were conducted on water system (Asubiojo *et al*., 1997; Mombeshora *et al.,* 1983; Ndiokwere & Cumie, 1983; Nriagu, 1986; Nriagu & Pacyna, 1988) with few studies on soil samples (Akanle *et al.,* 1994; Ogunsola *et al.,* 1994; Onianwa, 2001; Oyedele *et al*., 1995 Abubakar, 2007; Emanuel, 2015). Moreover, most of the studies conducted used either Atomic Absorption Spectrometry (AAS), X-ray Fluorescence (XRF) or Neutron Activation Analysis (NAA) analytical techniques with none of the studies using Microwave Plasma-Atomic Emission Spectrometry (MP-AES) based upon the available literature at the researchers' disposal. It is against this background that the researcher intended to determine the concentration levels of trace element using MP-AES analytical technique in soil samples collected from Fika local government area of Yobe state.

The Atomic Emission Spectroscopy (AES) technique involves the measurement of electromagnetic radiation emitted from atoms and the method is used for the multi-element analysis of a wide range of materials. It is the most commonly used procedure for the measurement of trace elements in rocks, water, soil, manufactured goods and biological specimens (Twyman, 2005). In a quantitative analysis, the intensity of the emitted radiation is related to the concentration of each element, with each atomic spectra resulting from the transition of electrons from one discrete electron orbital in an atom to another.These spectra can be understood in terms of the Bohr atomic model.

In the Bohr model, the atom is depicted as a nucleus surrounded by discrete electron orbits, each associated with energy of the order h*v,* where h and *v* are the Planck's constant and the frequency of the radiation respectively. Every atom has a certain number of electron orbitals, and each electron orbital has a particular energy level. When all the electrons are present in the orbitals, the atoms are in the most stable form (the ground state). When energy (either thermal, resulting from collision, or radiational resulting from the absorption of electromagnetic radiation) is applied to an atom and is sufficient to lift an electron from a shell with energy E_i to one with E_j, the atom is said to be in the excited state. The state of excitation is unstable and decays rapidly. The residence time of the unstable excited state is very short, in the order of 10^{-8} s. When electrons return to the stable ground state, energy is emitted and that energy is equal to the difference in the energies between the ground and excited states.The energy is released in the form of electromagnetic radiation and defines the wavelength of the transition (Twyman, 2005). The relationship between the energy and wavelength is described by Planck in equation (1) below:

$$E_j - E_i = h\nu = \frac{hc}{\lambda} \quad (1.0)$$

where $E_j - E_i$ is the energy difference between the two levels (and $E_j > E_i$); h is Planck's constant, 6.624×10^{-34} Js^{-1}; *v* is the frequency of the radiation; c is the velocity of light in a vacuum, 2.9979×10^{8} ms^{-1}, and λ is the wavelength of the radiation in meters. From the above equation,

$$\lambda = \frac{hc}{E} \quad (2.0)$$

where *E* is the energy difference and λ is the wavelength of the emitted radiation.The wavelength of the atomic spectral line gives the identity of the element while the intensity of the emitted light is proportional to the number of atoms of the element.

1.1 Study Area

The study area comprised the whole of Fika local government area of Yobe State, Nigeria. Fika is located between latitude 11°17'16" North and longitude 11°18'28" East (Maplandia, 2017). It has an area of 2208 km² consisting of ten wards. It has a total population of 136,895 at the 2006 census with over 70% of the population involved in agricultural activities. The vegetation of Fika falls under Sudan savannah whose annual rainfall range from 500 mm to 1000 mm. Fika populace experience cool dry (harmattan) season from December to February with a minimum temperature of 22°C; a hot dry season from March to May with a maximum temperature range of 39°C to 42°C; a warm wet season from June to September with average temperature of 40°C and a less marked season after rainfall during the months of October to November with temperature of 28°C (Meteorological Station Potiskum, 2017).

2. Materials and Methods

2.1. Materials

The items used for data collection are as follows: Beaker (500ml), Stirrer, auger, Distilled water, Plastic container (200 ml), Digestion tubes, Hot plate (adjustable), Reagents (HCl and HNO_3), Plastic bottles (50 ml), Marker and masking tape, Ruler, Weighing balance (M. AE240), Plastic mesh sieve, Agilent 4200 MP-AES, GPS device and Hand gloves.

The instrument used for sample measurement and analysis is *Agilent 4200 MP-AES* located in the Multi-user Science Laboratory of Ahmadu Bello University Zaria. The samples locations were sited using Global Positioning System (GPS) device and coded as in Table 1 and Figure 1 below.

Sample Code	Sample Ward	Sample Coordinates by GPS Latitude Longitude	Collection Village	Elevation in Meters
FKA_1	Fika/Anze	Lat. 11°14'40.9"N, Long. 011°19'37.1"E	Anze	325
FKA_2	Janga/Boza	Lat. 11°34'40.2"N, Long. 011°12'18.3"E	Dogo Abare	463
FKA_3	Ngalda/Dumbulwa	Lat. 11°06'15.8" N, Long. 011°22'02.3"E	Ngalda	260
FKA_4	Turmi/Maluri	Lat. 11°18'19.1"N, Long. 011°22'26.4"E	Turmi	363
FKA_5	Zangaya/Mazawun	Lat. 11°16'59.1"N, Long. 011°22'16.4"E	Gashua	345
FKA_6	Gadaka/Shembire	Lat. 11°16'54.0"N, Long. 011°10'09.0"E	Ngeji	335

Sample Code	Sample Ward	Sample Coordinates by GPS Latitude Longitude	Collection Village	Elevation in Meters
FKA_7	Daya/Chana	Lat. 11°32'36.1"N, Long. 011°02'26.8"E	Daya	449
FKA_8	Mubi/fusami	Lat. 11°15'06.9"N, Long. 011°17'37.0"E	Badawa	340
FKA_9	Gudi/Dozi	Lat. 11°19'19.3"N, Long. 011°03'05.6"E	Gamari	438
FKA_{10}	Shoye/Garin Aba	Lat. 11°16'12.3"N, Long. 011°06'40.4"E	Garin Ada	405

Table 1: Sampling Points and Location of Soil Sample

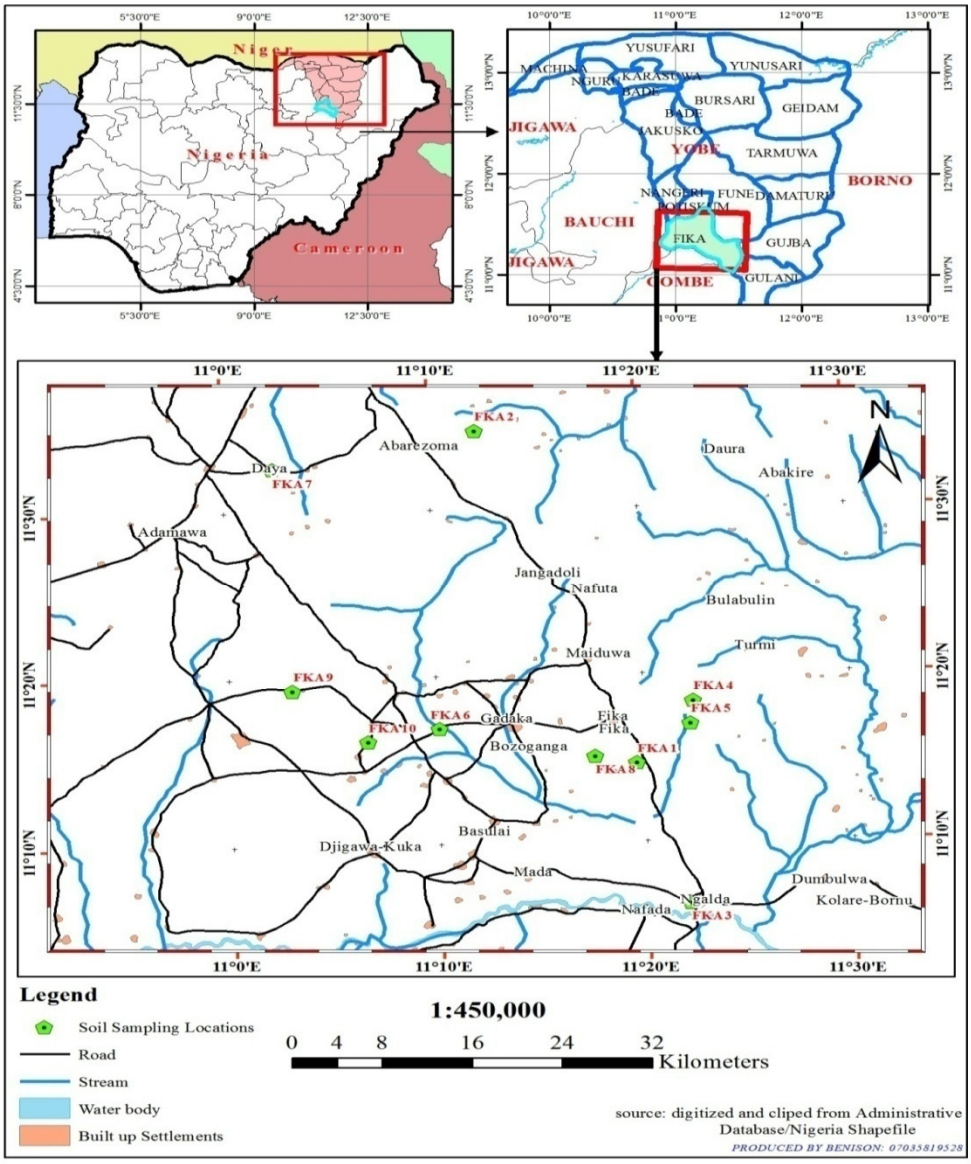

Figure 1: Map of Fika Local Government Yobe State Showing Sample Locations

2.2. Sample Collection, Preparation and Experimental Procedure

Representative soil samples from ten (10) wards were collected from farmland of the study area and GPS device was used to mark the coordinates and elevation above sea level. Three soil samples were collected at a depth of 0 to 20cm using soil auger from each ward. The samples were carefully mixed and put into clean and labelled plastic containers for analyses in the laboratory. Nitric acid ($HN0_3$) and Hydrochloric acid (HCl) were mixed together in the ratio of 3:1 in order to form the wet digestion acid mixture. 0.5g of each of the samples was transferred into digestion tubes, 30ml of the wet digestion acid mixture were added to each. The solutions were heated on hot plate at about 100°C until clear solutions were obtained. Then the digestion process was discontinued and the digest were allowed to cool and transferred into volumetric flasks, they were made up to the mark of 50ml with distilled water. The digest of each sample was transferred into the different 50ml plastic bottles which were made ready for MP-AES analysis. Standard procedure was followed determine the EC of the samples using Agilent 4200 MP-AES machine.

2.3. Quality Control

The validation of MP-AES could be achieved by using standard reference material (SRM) of similar matrix as a control for the sample under investigation. The essence of this validation is to test the calibration of the entire system since the literature values of the standard reference materials (SRM) is re-investigated by the analyst (ATI, 2015). The

calibration of the entire system could be established by observing the variation in the precision between differences with the standard value and the experimental values. Standard stock solution (SRM) was used as quality control in this work. The concentration of each element determined from this work was compared with the standard values and percentage error was computed using equation 3.0 below and the result is shown in Table 5.0.

$$PE = \frac{(Sv - Wv)}{Sv} \times 100 \quad (3.0)$$

where PE is percentage relative error, Sv is standard value and Wv is the work value respectively.

2.4. Data analysis

The parameters of interest to be determined and their associated variables are given below:

2.4.1. Elemental Concentration (EC)

The EC gives the concentration in ppm of each trace element in the study area for the ten composite samples collected. This can be calculated (Abolude, *et al*., 2009) using the following equation (4)

$$EC = \frac{Instrumentreading(ppm) - Blank(ppm) x finalvolumeprepared(l)}{Weightofthesample(g)} \quad (4.0)$$

In this study, the final sample volume prepared after digestion is 50 ml, blank = 0.000 and 0.5 g weight of the prepared samples were used.

2.4.2. Total Metal Content (M_{tot})

This is the difference between all inputs and outputs, where the input sources are weathering of parent material (M_p), atmospheric deposition (M_{ad}), fertilisers (M_f), pesticides (M_{pe}), organic wastes (M_{ow}) and inorganic pollutants (M_{ip}), while the outputs are removal of crops (M_{cr}), leaching (M_l) and volatilization (M_v). The main input and output sources of metals in this study are probably pesticides, mining and waste water (in) and crop up-take and leaching (out) respectively. According to Sanda, (2015), the total metal content in the soil can be expressed by the following equation (5).

$$M_{tot} = (M_p + M_{ad} + M_f + M_{pe} + M_{ow} + M_{ip}) - (M_{cr} + M_l + M_v) \quad (5.0)$$

The equation covers the total concentration of metals (Sanda, 2015).

3. Results and Discussion

The Microwave Plasma Atomic Emission Spectroscopy (MP-AES) analysis results of soil in the study area shows presence of Aluminium (Al), Cadmium (Cd), Cobalt (Co), Chromium (Cr), Iron (Fe) Manganese (Mn), Lead (Pb) and Zinc (Zn) for all the samples at 0 to 20cm soil depths. The average elemental concentrations of trace elements are depicted in Table 2 which indicate that all Cd and Co elemental concentrations were below detection limit except in FKA_4 and FKA_9 respectively. While Zn concentrations in FKA_9 and FKA_{10} were below detection limit; and of all elemental concentration only Pb in FKA_1 was found to be above the International Benchmark Concentration.

Element	FKA_1	FKA_2	FKA_3	LOCATION CODES				FKA_8	FKA_9	FKA_{10}
				FKA_4	FKA_5	FKA_6	FKA_7			
Al	2.793	2.507	7.002	4.815	9.387	1.649	3.607	4.559	1.213	1.877
Cd	BDL	BDL	BDL	0.001	BDL	BDL	BDL	BDL	BDL	BDL
Co	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.002	BDL
Cr	0.008	0.021	0.019	0.053	0.019	0.006	0.008	0.007	0.003	0.007
Fe	4.220	4.868	4.460	15.198	11.858	2.087	3.069	4.989	0.201	2.957
Mn	0.186	0.048	0.260	0.958	0.493	0.047	0.037	0.095	0.083	0.104
Pb	80.990	0.341	0.085	0.042	0.031	0.020	0.034	0.017	0.010	0.012
Zn	0.500	0.020	0.022	0.067	0.024	0.008	0.014	0.056	BDL	BDL

Table 2: Elemental Concentration (EC) of trace element in soil samples values in ppm.
BDL – Below Detection Limit

Table 3 shows the statistical summary of elemental concentration in the study area. The mean elemental concentration obtained is in the order of Pb > Fe > Al > Mn > Zn > Cr > Co > Cd with lowest and highest mean values of 0.001(ppm) and 81.585(ppm) from Cd and Pb, respectively.

Elements	n	Mean	Range	Mean ± SD
Al	10	3.941	8.174	3.941±2.595
Cd	1	0.001	0.000	0.001±0.000
Co	1	0.002	0.000	0.002±0.000
Cr	10	0.151	0.050	0.151±0.015
Fe	10	53.907	14.997	53.907±4.593
Mn	10	2.311	0.921	2.311±0.291
Pb	10	81.585	80.981	81.585±25.591
Zn	8	0.500	0.492	0.500±0.167

Table 3: Statistical Summary Showing Mean, Range and Mean ± SD of Concentrations of Trace Element in Soil Samples

Table4 shows the Pearson Product Moment Correlation (PPMC) coefficients of elemental concentration of the study area. Strong positive correlation coefficients were found between Fe – Cr, Mn – Fe and Zn – Pb at significant level of 0.01. Positive moderate correlation coefficient was obtained between Fe – Al in the study area. However correlation between Cd and Co with themselves and any other trace element cannot be computed since they only have one constant elemental concentration values. On the other hand, weak negative correlation coefficients were obtained between Pb – Al, Zn – Al, Pb –Cr, Zn – Cr Pb – Fe, Zn – Fe, Pb – Mn and Zn – Mn among others.

Elements	Al	Cd	Co	Cr	Fe	Mn	Pb	Zn
Al	1.000							
Cd	CC	CC						
Co	CC	CC	CC					
Cr	0.388	CC	CC	1.000				
Fe	0.655*	CC	CC	0.870**	1.000			
Mn	0.520	CC	CC	0.903**	0.926**	1.000		
Pb	-0.156	CC	CC	-0.168	-0.090	-0.055	1.000	
Zn	-0.253	CC	CC	-0.172	-0.104	-0.018	0.992**	1.000

Table4: The PPMC Correlation Coefficients of Elemental Concentrations (EC) Of Trace Elements in the Study Area
***Correlation Is Significant At The 0.01 Level. *Correlation Is Significant At The 0.05 Level.*
Cc= Cannot Be Computed Because At Least One Of The Variables Is Constant

Table 5 shows theaverage elemental concentration of the study compared with Iranian Bekhtegan Lake surface soil, Amhara region Ethiopia, Nigerian Ameka Mining Area Abakalaki and Egyptian Lake Edku Sediments against the United State Department of Energy (USDE) International Benchmark.

Elements	FKA	IRN	ETP	EGP	ABK	BMK
Al	3.941	-	-	-	-	50.000
Cd	0.001	0.520	2.300	-	0.08	4.000
Co	0.002	14.430	-	98.9	-	20.000
Cr	0.151	74.950	29630.000	113.1	12.28	1.000
Fe	53.907	79307.15	499.800	38822.8	206.00	-
Mn	2.311	278.32	499.800	1923.6	-	500.000
Pb	81.585	12.710	13.800	44.6	54.47	50.000
Zn	0.500	34.170	213.400	82.5	43.03	50.000

Table 5: Comparison of Mean Elemental Concentrations (EC) of Trace Elements in Nigeria-Fika (FKA), Iran (IRN), Ethiopia (ETP), Egypt (EGP), Abakalaki (ABK) Against the U.S. Department Of Energy International Benchmark (BMK) For Soil in (PPM)
Source: Efroymson Et Al. (1997), Shekeri Et Al. (2014), Mathias & Stephen (2016), Yousef Et Al., (2017) and Addis & Abebaw (2018)

The concentration of trace element in the three regions are of order of: - for Al: BM >FKA, for Cd: BM > ETP > IRN > ABK > FKA. For Co : EGP > BM > IRN > FKA, for Cr : ETP > EGP > IRN > ABK > BM > FKA, for Fe : IRN > EGP > ETP > ABK > FKA, for Mn : EGP > BM > ETP > IRN >FKA, for Pb : FKA > ABK > BM > EGP > ETP > IRN, and for Zn : ETP > EGP > BM > ABK > IRN > FKA. These imply that all the elemental concentrations are below the International Benchmark except Pb in FKA was found to be above the international threshold. This is due to the gypsum mining activity being carried out in the study area. The finding of this study is supported by Buba and Aboyeji (2015) studies which found high concentration of Lead (Pb) in Zamfara mining sites.

4. Conclusion

Based on the MP-AES result of this study, the elemental concentration of Al, Cd, Cr, Co, Fe, Mn, Pb and Zn were determined correlated and compared with other relevant studies and international benchmarks. The maximum and minimum elemental concentration ranges obtained are: Al (1.213 –7.002)ppm, Cd (BDL – 0.001)ppm, Co (BDL – 0.002)ppm, Cr (0.003 – 0.053)ppm, Fe (0.201 – 15.198)ppm, Mn (0.037 – 0.0958)ppm, Pb (0.010 – 80.990)ppm and Zn (0.008 – 0.500)ppm with the order of Pb (81.585) > Fe (53.907) > Al (3.941) > Mn (2.311) > Zn (0.500) > Cr (0.151) > Co (0.002) > Cd (0.001) in ppm respectively. The presence of plants' micro nutrients such as Fe and Zn and Mn below the USDE benchmark is of agricultural advantage. It can therefore be concluded that Fika is an uncontaminated agrarian area suitable for cereal growth.

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