

Full Length Research Paper

Energy dispersive X-Ray fluorescence determination of minor and major elements in soils of Mambilla Plateau Northeastern Nigeria

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Received 8 November, 2018; Accepted 17 January, 2019

This study focuses on energy dispersive X-ray fluorescence determination of minor and major elements in selected soils of Mambilla Plateau. The 12 sampled soil composites analysed were done by energy dispersive X-ray fluorescence technique for minor and major elements. The elemental oxides determined were Al₂O₃, CaO, K₂O, TiO₂, SiO₂, BaO, Fe₂O₃, and MnO. The result obtained in the study showed that the general percentage concentration of elemental values were in decreased order of Si>Al>Ti>K>Ca for major elements and Fe>Mn>Ba minor elements accordingly. The major elements category showed silicon as the highest mean concentration at location S 84.09%±6.40 and calcium lower mean concentration with value of 0.32%±0.02. The minor elements concentration percentage revealed Fe with a mean value at location N 23.18%±3.1 being the highest and Ba is the lowest at location S 0.14%±0.1. The soil analysis revealed low concentration of essential major and minor elements, however, high concentration of Al and Fe that may solubilized at low pH and splitting H⁺ from water molecule showed increased soil acidity.

Key words: Energy dispersive X-ray fluorescence (EDXRF), minor elements, major elements, percentage, Mambilla Plateau.

INTRODUCTION

Soil as a natural resource, is a complex body made up of interacting mineral, organic, water and air components with both biotic and abiotic features, which is capable of supporting plant life (Brady, 1990; Carter, 1993; William,

2000; Ayoub et al., 2003). It is the product of weathering of rocks at the earth surface, which composed of four main components: mineral elements 45%, organic matter 5%, water 25%, and air 25% (Boul et al., 1973). Bowen

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(1979) reported soil as a mixture of mineral particles ranging from microscopic to easily visible fragments; adhering to these particles may be nutrients, additional elements, organic matter differing markedly in size, shape, chemical composition, mineralogical nature, chemical and physical properties. Although the knowledge of soil mineral quality plays a vital role in the improvement of the production and the productivity of the agricultural business, there is scarce scientific information available about the magnitude of soil quality changes related to the different land uses (Lal and Stewart, 2010). The soil is composed of wide variety of elements, some of these elements when available in extreme amounts are detrimental to plants and these includes aluminium and lead, others are used by plants in various chemical and metabolic processes to facilitate their growth, development and reproduction. In addition, their insufficiency may result to poor growth and yield problems in agricultural practices (Batista et al., 2017). The availability of chemical elements may vary in all soils according to the conditions acting on their formative processes; this plays a vital role in soil fertility, since mineral surfaces serve as potential sites for nutrient storage (Rahman et al., 2015). Furthermore, the experimental analysis of soil composition is very important in any purpose intended or suitable, especially in the beneficial basic chemical elements classification of the soil. The soil essential mineral elements have been studied into two main groups, categorized as the major or macro elements/nutrients and the minor/micro or trace elements/nutrients (Kabata-Pendias and Pendias, 1992; Neubauer, 2017). The essential elements required by plants in large quantity includes nitrogen (N), phosphorus (P), potassium (K), sodium (Na), calcium (Ca), magnesium (Mg), aluminium (Al), and sulphur (S) (Kabata-Pendias and Pendias, 1992; Adriano, 1986; Adeyeye, 2005; Ngole-Jeme and Ekosse, 2015). While minor elements which are required in small amounts by plants include: boron (B), cobalt (Co), copper (Cu), selenium (Se), molybdenum (Mo), vanadium (V), zinc (Zn), and manganese (Mn) (Adriano, 1986; Kabata-Pendias, 1992). The presence or absence of major or minor elements in soil determined its availability and fertility (McCarthy, 1990; Kumar et al., 2014). The micronutrients occur in very small amounts in both soil and plants, but their significant role is as the need for the major nutrients (Ajibola and Rolawanu, 2000; Aubert and Pinta, 1977). The nutrients natural levels in soil vary, depending largely on the nature of parent materials from which the soils are formed and also on soil-forming processes (Adriano, 1986; Kubota, 1981; Lund et al., 1981; Heil and Mahmoud, 1978). Natural distribution patterns of trace elements in soil have been affected by a variety of anthropogenic activities, including mining of natural resources, smelting, agricultural practices, energy generation, manufacturing industries, waste/effluent disposal, and automobile transportation (Adriano, 1986; Munro, 1983: 1974). By and large, the physical, chemical

and biological properties of soils are indicators that transmit its usefulness (Shekhovtseva and Mal'tseva, 2015). The desire to examine soil for specific or total chemical elements of soils recent in times has explored different techniques and methods using state of the art technology. Several studies conducted have shown a great deal of analytical methods on soils round the globe such as ion exchange membrane technology (IEM) (Zaher, 2012). The inductive couple plasma-mass spectrometry (ICP-MS) by Smart et al. (2017), Markl et al. (2014), Riedel et al. (2015), and Bzour et al. (2016). The inductive couple plasma atomic emission spectroscopy (ICP-AES) by Xiangdong and Thorton (2001) and Burke et al. (2015) demonstrated analytical electron microscopy (AEM). The inductive neutron activation analysis (INAA) method has been adopted by Tenpe and Parwate (2015), Njing`a et al. (2013), and Ene et al. (2009), X-ray fluorescence (XRF) by Towett et al. (2015), and total X-ray fluorescence (TXRF) by Rahman et al. (2015). The energy dispersive X-ray fluorescence (EDXRF) was determined by Sudhakaran et al. (2018), Thomas et al. (2016) and Idris et al. (2004), wave dispersive X-ray fluorescence (WDXRF) by Grosheva et al. (2007), and proton induced X-ray emission/ion beam analysis (PIXE/IBA) by Mahmud et al. (2015). These are few among many studies that in turn have made available data and significant contributions to the soil literature for proper use and sustainability from the global threatening environmental degradation. In addition, the varying chemical, physical and biological soil properties studied, has specified greatly, recommendations for the future of agricultural soil corrections, sustainability and other industrial applications. The X-ray fluorescence is a multi-elemental analytical technique with broad application in science and industry. The technique is based on the principle that individual atoms, when excited by an external energy source emit X-ray photons of characteristics energy wavelength. Which upon counting the number of photons of each energy emitted from a sample, the elements present may be identified and quantified (James and Jeffery, 2012). Therefore, it is good to know the types of minerals that make up the soil around our environments and also among other things to determine the degree to which the soil chemical compositions, retention and nutrients are absorbed by plant. This study was conducted to evaluate the soils of sampled locations of Mambilla Plateau in Sardauna Local Government Area of Taraba State, Northeastern Nigeria, in order to harness data on the availability of major and minor elements of the soils using energy dispersive X-ray fluorescence spectrometry.

MATERIALS AND METHODS

Materials

Analytical balance and MiniPal4 energy-dispersive X-ray

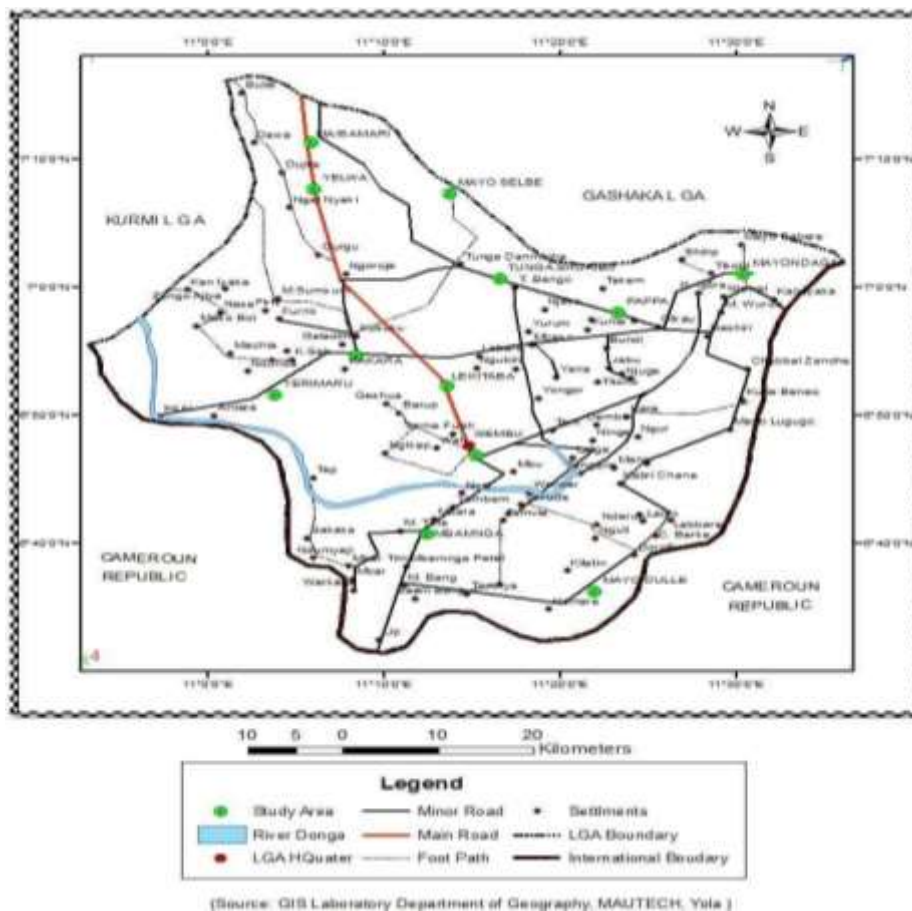


Figure 1. Map of the study area and sampling points, Sardauna Local Government-Taraba, Nigeria.

fluorescence (EDXRF) spectrometer model PW 4052/47B were used. Additionally, the following tools were used: cutlass, hoe, hand trowel, measuring rule, clean dry polyethylene bags and corer, pH meter, and measuring cylinder.

Study area

The study area Mambilla Plateau is located between latitude 6.82120°N and 7.3523°N and longitude 10.7723°E and 11.5345°E in Sardauna Local Government Area of Taraba State, Northeastern Nigeria. Bounded by an escarpment that is about 900 m high in some places. The plateau is a semi temperate highland region, with low grasses and noticeable trees (except for the man made forest) covering an area of over 9,389 km², with daytime temperature that hardly exceeds 25°C (Figure 1). The rainfall is between 1600 and 1800 mm of annual rainfall (Bami, 2003; Adebayo and Umar, 2005).

Sampling and sample preparation

The sampled locations at random were coded as N₁, N₂, N₃, N₄, E₁, E₂, E₃, E₄, S₁, S₂, S₃, and S₄, respectively. The samples were taken at random point and stratified depth of 30 cm each (Radojevic and Bashklin, 2006). A total of 12 representative soil samples were collected from the locations and introduced into a dry clean polyethylene bags then transported to the laboratory. The samples

were air dried and sieved through a 2 mm mesh and later oven dried.

Elemental analysis

The soil elemental oxides were determined using Energy Dispersive X-ray fluorescence (EDXRF) method as adopted by Yu et al. (2002) at Center for Energy Research and Training (CERT) Zaria, Nigeria. Each soil sample Pellets of 19 mm diameter were prepared from 5.0 g powder, mix with 1.0 g cellulose flakes binder and palletized at a pressure of 10 ton/inch in a palletizing machine. The palletized samples were stored in a desiccator for and appropriate programs for the various elements of interest were employed to analyze the sample materials for their presence or absence. The measurement was performed using rhodium as the excitation source. The fluorescent X-ray emitted was sent to the Si (Li) detector, coupled to a computer controlled analog to digital converter (ADC) card. Since each element has a characteristic and identifiable X-ray signature, the presence and concentration of each element of interest within the sample was determined in percentage oxides as reported in this work. The concentration of the elements analyzed has been expressed in elemental percentage shown in Tables 1, 2 and 3.

$$\text{Percentage element} = \% \text{Oxide} / \text{RMM} \times 100 \quad (1)$$

where RMM=relative molecular mass.

Table 1. Percentage (%) oxides of major elements.

Location	Al ₂ O ₃	CaO	K ₂ O	TiO ₂	SiO ₂
E ₁	16.00	0.67	ND	4.47	19.00
E ₂	19.00	0.27	ND	3.14	41.70
E ₃	20.00	0.13	1.50	4.05	48.50
E ₄	14.00	0.01	2.10	4.24	32.00
Mean	17.25±2.2	0.27±0.1	1.80±0.2	3.98±0.4	35.30±6.4
N ₁	11.00	0.27	ND	3.06	51.10
N ₂	12.00	0.18	ND	7.34	10.00
N ₃	17.00	0.14	ND	5.67	26.90
N ₄	9.00	0.20	ND	7.80	11.00
Mean	12.25±1.22	0.20±0.1	ND	5.97±1.34	24.75±0.68
S ₁	27.60	0.37	ND	1.59	54.60
S ₂	12.00	0.10	1.10	2.10	53.00
S ₃	23.80	0.10	ND	2.50	43.30
S ₄	19.00	0.10	1.90	1.97	51.20
Mean	20.60±2.6	0.19±0.02	1.50±0.10	2.04±0.28	50.53±6.6

ND: Not detected.

Table 2. Percentage (%) element of major elements.

Location	Al	Ca	K	Ti	Si
E ₁	15.69	1.19	ND	5.57	31.62
E ₂	18.63	0.48	ND	3.93	69.40
E ₃	19.61	0.23	1.59	5.07	80.72
E ₄	13.70	0.02	1.17	5.56	53.26
Mean	16.91±2.22	0.48±0.04	1.38±0.02	5.05±0.47	58.75±3.86
N ₁	10.79	0.39	ND	3.83	86.05
N ₂	11.77	0.32	ND	9.22	16.64
N ₃	16.67	0.25	ND	7.10	44.77
N ₄	8.83	0.35	ND	9.88	18.31
Mean	12.02±2.67	0.33±1.33	ND	7.50±3.12	41.44±1.23
S ₁	26.58	0.66	ND	2.00	90.87
S ₂	11.77	0.18	1.17	2.63	88.21
S ₃	22.56	0.30	ND	3.13	72.07
S ₄	18.63	0.18	2.02	2.47	85.21
Mean	19.89±2.45	0.32±0.02	0.80±0.14	2.56±0.02	84.09±6.40

ND: Not detected.

RESULTS AND DISCUSSION

The major and minor elements detected in samples were Al₂O₃, K₂O, CaO, SiO₂, TiO₂ and BaO, Fe₂O₃, and MnO. The results of major elements in Tables 1 and 2 showed decreasing order of concentration as follow: SiO₂>Al₂O₃>

TiO₂>CaO>K₂O. In Table 2, the elemental percentage concentration of Al analyzed in the samples ranged from 8.83 to 26.58% across E, N, and S locations with means of 16.91% ± 2.22, 12.02% ± 2.67 and 19.89% ± 2.45, respectively. The highest concentration was at location S₁ (26.58%) and low concentration revealed at location

Table 3. Percentage (%) oxides and element of minor elements.

Location	BaO	Fe ₂ O ₃	MnO	Ba	Fe	Mn
E ₁	0.34	37.10	0.22	0.22	23.23	0.31
E ₂	0.40	30.28	0.26	0.26	18.96	0.37
E ₃	ND	24.22	0.22	ND	15.17	0.31
E ₄	0.80	29.80	0.23	0.11	18.66	0.32
Mean	0.51±0.1	30.35±4.1	0.23±0.02	0.20±0.02	19.00±2.7	0.33±0.03
N ₁	ND	17.50	0.22	ND	10.96	0.31
N ₂	ND	46.54	0.38	ND	29.14	0.54
N ₃	ND	44.17	0.53	ND	27.66	0.75
N ₄	ND	39.70	0.44	ND	24.86	0.62
Mean	ND	36.98±3.4	0.39±0.01	ND	23.16±3.1	0.56±0.14
S ₁	0.02	10.06	0.07	0.01	6.30	0.10
S ₂	0.04	21.00	0.04	0.02	13.15	0.06
S ₃	0.34	18.17	0.05	0.22	11.38	0.07
S ₄	0.48	13.40	0.03	0.31	8.39	0.04
Mean	0.22±0.01	15.66±2.4	0.05±0.01	0.14±0.1	9.81±1.4	0.07±0.1

ND: Not detected.

N₄ (8.83%). Aluminium was reported to dissociates water molecule which increases the hydrogen ion [H⁺] concentration in the soil and in arid soils, toxic Al³⁺ cations increase disturbs root growth and functions (Horst, 1995). Calcium concentration in all the samples ranged from 0.18 to 0.48%, with elemental mean values of 0.48% ± 0.04, 0.33% ± 1.33 and 0.32% ± 0.02 at E, N, and S locations appropriately. However, Idris et al. (2004) revealed 48.08, 18.90, 1.26, 7.71 and 1.93% for Al₂O₃, SiO₂, Ti₂O, and K₂O in bauxite analysis of Mambilla samples. The Spectrum Analytical (2016) reported acidic soils to have less calcium. In addition, whereas the additional "free" calcium is not absorbed into the soil but much of the free calcium forms nearly insoluble compounds with other elements such as phosphorus (Spectrum Analytica, 2016). Table 2 shows potassium detected in five out of the twelve representative samples collected between 0.80 and 1.38% with percentage means of locations E 1.38% ± 0.02 and S 0.80% ± 0.14, while location N was below detection limit in the study. The absence or loss of soil potassium may be due to leaching, erosion and potassium fixation. It has been shown that parent materials composition has a direct impact on soil properties; especially soils developed over sand stone are low in soluble bases and coarse in texture which facilitates leaching (Michael, 2011). The result for silicon indicates percentage availability of silicon which was satisfactory as the second most abundant element on the earth crust as reported by Havlin et al. (2003). It ranged from 11.00 to 61.20% with means of E (58.75% ± 3.06), N (41.44% ± 1.23) and S (84.09% ± 6.40) sample locations. Titanium oxide was detected with elemental

range of 2.56 to 7.50% and a mean values of locations E (5.05 ± 0.47), N (7.50% ± 3.12) and S (2.56% ± 0.02). Table 3 shows the oxides of minor element analyzed were BaO, Fe₂O₃ and MnO. The result indicated elemental Fe concentration as the highest among the minor elements determined, then manganese and barium (Fe>Mn>Ba). In the concentration results of barium element in the samples locations ranged from 0.01 to 0.31%, the highest concentration was at S₄ 0.31%. While N₁-N₄ showed no results, perhaps were beyond detection limit. The locations generally showed elemental means of Ba at E (0.20% ± 0.02) and S (0.14% ± 0.1). Ponge et al. (2002) reported barium to be soluble at low pH but leached from water shed soil by acidic deposition. However, in a related study, Isaac et al. (2018) reported high acidity of Mambilla soils, which showed low detection of element concentration. The iron (Fe) content in the soil samples analyzed was high; it ranged from 6.30 to 29.14%. The location N₂ gave the highest concentration of Fe 29.14% and mean values of Fe at locations E (19.00% ± 2.70), N (23.16% ± 3.10) and S (9.81% ± 1.40), respectively. While Idris et al. (2004) reported 7.71% Fe₂O₃. This study result agrees with iron being the fourth most abundant element on earth crust present in form of silicate minerals or iron oxides and hydroxides (Schulte, 2004); in addition, resulting in red and yellow colours due to iron concentration of the soil (Isaac et al., 2018; Schulte, 2004; Havlin et al., 2003). Ajala and Onwukeme (2012) similarly reported higher percentages of iron 67.00% and manganese 65.10% in metropolitan soils. Also, Nderi et al. (2015) reported a low Fe at 6.77%; Idris et al. (2004) obtained 7.71%.

Iron oxide in soil is an important sorbents as it ascribed the role of binding heavy metals present in the soil solution as cations and anions (Dong, 2000; Kunaljeet et al., 2008). The manganese (Mn) concentration values in the samples range from 0.04 to 0.75%. The highest concentration of Mn was at N₃ (0.75%), while mean values were E (0.33% ± 0.03); N (0.56% ± 0.14) and S (0.07% ± 0.1). It had been established, acidic soils showed higher oxides of Al and Fe (Chand et al., 2011), manganese deficiency because reducing conditions precipitated and occluded oxides and hydroxides were solubilized to complex form (McBride, 1994). This agrees with the acidic profile of Mambilla soil reported at pH 3.96 to 4.30 (Isaac et al., 2018) in relation to this study. Tigist et al. (2014) reported Mn 0.03% and Chand et al. (2011) reported 0.04% Mn at pH 4.6 to 5.3. These results showed low concentrations of manganese in the soils studied.

Conclusion

In view of this study, the elemental composition of a given soil is basic to its usefulness, especially in crop production, mineral exploration and other industrial applications. This study showed the soil locations are strongly acidic with very low essential elements for potential agricultural production. Perhaps, further study can be undertaken on total assessment of essential elements, especially nitrogen, phosphorus, and the toxicity and biochemical compositions of the soil, exploring other instrumental methods that will provide new insights or affirm previous works.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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