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APPLICATION OF k_0-INAA TECHNIQUE FOR THE ANALYSIS OF ESSENTIAL AND BENEFICIAL HEAVY ELEMENTS IN SOIL

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Abstract
The researchers analyzed the essential and beneficial heavy elements in soil that improve plant growth, development and other functions in this study using k_0 – INAA technique. The use of $\boldsymbol{k_0}$ **–** Instrumental Neutron Activation Analysis (k_0 – INAA) technique has become a veritable tool for multi-element analysis. This method has been applied in this present study to assess essential mineral elements and beneficial heavy elements that supports plants growth of fifteen (15) samples collected from Safana, Dan Musa and Kankia, LGAs of Katsina State. The samples were properly prepared together with the standard reference material (NIST Coal Fly Ash 1633b) and were irradiated at a thermal power level of 31.0 kW corresponding to a neutron flux setting of 2.5 x 10^{11} cm^{-2} s^{-1} and 5.0 x 10^{11} cm^{-2} s^{-1} for the outer and inner irradiation channels respectively using the Nigeria Research Reactor – 1 (NIRR – 1) at the Centre for Energy Research and Training, Zaria. After irradiation, the samples were counted using a high-purity germanium detection system (GEM 30195 coaxial detector) and the concentrations of eleven elements (Mg, Ca, Ti, V, Mn, Na, K, Cr, Fe, Co and Zn) from the samples and that of NIST Coal Fly Ash 1633b were measured using the k_0 – IAEA program. The result of the NIST Coal Fly Ash 1633b was in agreement with that of the certified values. The concentrations of the elements measured from the samples shows that these elements are sufficient for plant growth, except that some of the essential heavy elements have reached toxic levels.

Keywords: *k_0 – INAA, nuclear reactor, essential mineral elements, beneficial heavy elements, plant growth*

Introduction

Mineral elements which form part of the heterogeneous mixture of soil are recognized to be an integral part of the human body structure as their presence is necessary for the maintenance of certain physicochemical processes which are essential in many activities in the body (Nigel & Sjaan, 2016). These minerals may either be in the form of essential elements, which are responsible for the prevention of deficiency syndromes (Nelson, 1999; CDRI, 2001) or beneficial heavy elements in plant growth (Kabata, 2007). Although these essential elements in soil are very important for the quality of soil and environment, however excessive level of these elements can cause pollution to waters, toxicity in plants, foods and ultimately in animals and humans that depends on these plants for survival (McLaughlin, 2001; Uchida *et al.*, 2007; Rana *et al.*, 2009). Besides, heavy elements in agricultural soil is of great concern, posing potential health risks in food and its detrimental effects on soil ecosystems (Chibuike and Obiora, 2014). Some of these trace elements are Zn, Cu, Mn, Mo, Fe, etc. while Co, Si, Na, etc. are beneficial heavy elements in plant growth. (Nigel & Sjaan, 2016; Lynette, 2016)

To ensure the comprehensive study of these elements, the researchers adopted the k_0-Instrumental Neutron Activation Analysis (k_0-INAA) technique, which is a form of Neutron Activation Analysis (NAA). It should be noted that NAA is a very precise technique mainly used to determine trace concentrations of elements in samples and/or to acquire information on the spatial distribution of a neutron field via neutron activation detectors (Majerle, 2006). Activation analysis therefore is a method for the determination of elements based upon the conversion of stable nuclei to other, mostly radioactive nuclei via nuclear reactions, and measurement of the reaction products (Jonah *et al.*, 2005).

The k_0-based NAA involves the simultaneous irradiation of a sample and a neutron flux monitor, such as gold, and the use of a composite nuclear constant called k_0-factor. The k_0-factor is independent of irradiation and measuring conditions. This technique eliminated the need of using multi-element standards (Simonits *et al.*, 1975; De Corte *et al.*, 1987; Landsberger, 2015).

In the k_0-standardization technique in reactor neutron activation analysis (INAA) the concentration of any element in the sample is determined by a conventional activation analysis formula containing a k_0 - factor – a compound nuclear constant- defined as (De Corte *et al.*, 1993):

$$k_0 = \frac{M_c \theta_a \sigma_a \gamma_a}{M_a \theta_c \sigma_c \gamma_c}$$

where, *a* and *c* denote analyte and comparator, respectively, and
M = atomic mass (g.mol^{-1})
σ_0 = thermal neutron cross-section (m^2)
θ = isotopic abundance, and

γ = absolute gamma-ray abundance

The technique has also been successfully installed and tested for elemental analysis with Nigeria Research Reactor – 1 (NIRR – 1) irradiation and counting facilities at the Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria (Jonah *et al.*, 2008).

Other researchers (Adeleye *et al.*, 2012; Njinga *et al.*, 2012; Joseph *et al.*, 2015b) has successfully applied this technique combined with the k_0-IAEA software using NIRR-1 for multi-elemental analysis. This study seeks to apply this technique to assess some essential elements and beneficial heavy elements in soil from Safana, Dan Musa and Kankia LGAs of Katsina State. This study is important because no research work had been carried-out to the best of our knowledge on the trace, minor and heavy elements concentrations on soil from these LGAs in the Central Area of Katsina State, apart from the one reported by Joseph *et al.,* (2015b) which focuses on the use of k_0 – NAA Standardization technique in evaluating elements of significance for plant growth in the cultivated areas of Dutsin-Ma Local Government, Katsina State-Nigeria. More so, the information derived from this current work will guide the government and the populace within the study areas who are predominantly farmers the best method on the utilization of the soil.

Materials and Methods

Sample Collection and Preparation

The soil samples were collected from five (5) different locations each from Safana (SFN), Dan Musa (DMS), and Kankia (KKA) LGAs of Katsina State. The soil hand auger was used in collecting these samples between the distances of 5 to 10 *km* from the depth of 0 – 30 *cm*, and each were immediately transferred into a clean labelled plastic container at the sampling point for further analysis in the laboratory. The samples were at first dried naturally at room temperature, then oven dried and later grounded in an agate mill to a grain size below 20μm plastic mesh and made homogeneous (Inuwa *et al.,* 2007; IITA 1979) at the NAA laboratory of CERT. The samples and the reference material NIST Coal Fly Ash 1633b with certified values supplied by International Atomic Energy Agency (IAEA), Vienna, Austria used for quality control (150 mg of powder) were wrapped in a polyethylene sample vials.

Irradiation and Measurements

The samples and the reference material were irradiated in the irradiation channels A1, B2, B3 and B4 of NIRR-1. Further details of NIRR-1 can be found in (Jonah *et al*., 2005; 2006; Ahmed *et al*., 2006; Sadiq *et al*., 2010; Njinga *et al*., 2011).

After the irradiation, the samples and reference material were measured using a high-purity coaxial germanium detector (HPGe) 30195 following the measurement protocols described by Jonah *et al*., (2005, 2006).

To measure the concentrations of the elements present using k_0-INAA technique via k_0-IAEA software (Rossbach & Blaaw, 2006), the detector used for measurement is first calibrated as described by Joseph *et al*., (2015a). The details of the k_0-IAEA software used for calculation of the concentration of this present study has equally been described (Blaaw *et al*., 1991; Blaaw, 1993; Ewa, 2004; Kolotov & De Corte, 2004; Rossbach *et al*., 2007). In the use of the k_0-IAEA software, the samples which are usually regarded as ordinary samples and the reference material spectra were all interpreted simultaneously using the k_0 – IAEA software program.

Results

The result of the reference material evaluated and used as quality control to validate the accuracy of the technique is in good agreement with the certified values for the elements evaluated. This result is presented on Table 1. We have also presented the result of the elemental concentration of the soil from the three (3) LGAs on Table 2.

Table 1: Result of NIST Coal Fly Ash 1633b (mg/kg)

ELEMENT	CERTIFIED VALUE	*k*o-IAEA
Al	150500 ± 2700	149820±512
Br	2.9*	3.2±0.3
Ca	15100 ± 600	15200±801
Dy	17*	18.9±4.7
Eu	4.1*	3.2±0.3
Fe	77800 ± 2300	77820±300
K	19500 ± 300	19400±600
La	94*	94±0.43
Lu	1.2*	2.0±0.40
Mg	4820 ± 80	4910±200
Mn	131.8 ± 1.7	133.6±3.1
Na	2010 ± 30	2180±28.3
Ni	120.6 ± 1.8	120.8±7.4
Rb	140*	139.6±2.1
Sb	6*	6.8±0.5
Sc	41*	42.7±6
Sm	20*	19±3
Sr	1041 ± 14	1041±26
Ta	1.8*	1.9±0.9
Tb	2.6*	2.5±0.32
Th	25.7 ± 1.3	24.9±3
Tm	2.1*	2.7±0.13
U	8.79 ± 0.36	8.9±0.14
V	295.7 ± 3.6	301.1±4.1
Yb	7.6*	7.4±0.7
Zn	210*	201±6

**: Non certified/recommended value*

Table 2a: Concentrations of elements in soils investigated by k_0-INAA (in mg/kg)

ELEMENT	KKA 01	KKA 02	KKA 03	KKA 04	KKA 05
Mg	ND	ND	ND	ND	ND
Ca	ND	ND	ND	ND	ND
Ti	3332 ± 312	3124 ± 2658	35214 ± 300	1949 ± 242	2555 ± 229
V	18.6 ± 2.9	21 ± 3	21 ± 4	18 ± 3	13.99 ± 2.70
Mn	105 ± 1	91 ± 2	154 ± 1	92 ± 1	116 ± 2
Na	2112 ± 6	1215 ± 4	1162 ± 6	2113 ± 15	1180 ± 7
K	13130 ± 223	27820 ± 302	15230 ± 247	16360 ± 447	11620 ± 369
Cr	15 ± 1	12.1 ± 1.4	21.7 ± 1.9	12.9 ± 1.6	10 ± 1.9
Fe	9575 ± 229	8439 ± 193	7769 ± 159	6723 ± 194	5348 ± 145
Co	3.8 ± 0.3	1.87 ± 0.18	2.6 ± 0.4	2.1 ± 0.1	1.37 ± 0.16
Zn	17 ± 6	ND	ND	ND	ND

Table 2b: Concentrations of elements in soils investigated by k_0-INAA (in mg/kg)

ELEMENT	DMS 01	DMS 02	DMS 03	DMS 04	DMS 05
Mg	ND	ND	1477 ± 363	ND	1352 ± 292
Ca	ND	3416 ± 92	60105 ± 1062	ND	ND
Ti	3026 ± 272	3004 ± 285	4752 ± 371	2575 ± 371	2180 ± 187
V	30 ± 2	22 ± 25	62± 3	31 ± 2	15 ± 2
Mn	170 ± 1	137 ± 1	427 ± 2	159 ± 1	162 ± 1
Na	1152 ± 5	3173 ± 6	2362 ± 7	1208 ± 5	980 ± 4
K	11160 ± 203	7638 ± 201	118730 ± 229	6979 ± 200	5498 ± 159
Cr	33 ± 25	41 ± 2	8.0 ± 0.3	36 ± 2	14 ± 2
Fe	10110 ± 190	7300 ± 168	23340 ± 280	9921 ± 198	682 ± 157
Co	7.3 ± 0.3	3.4 ± 0.2	8.9 ± 0.3	3.9 ± 0.2	2.8 ± 0.2
Zn	10 ± 4	ND	53 ± 6	50 ± 5	14 ± 4

Table 2c: Concentrations of elements in soils investigated by k_0-INAA (in mg/kg)

ELEMENT	SFN 01	SFN 02	SFN 03	SFN 04	SFN 05
Mg	ND	2179 ± 414	ND	ND	1487 ± 394
Ca	ND	3562 ± 794	3412 ± 836	ND	2321 ± 713
Ti	2005 ± 209	2972 ± 303	3209 ± 279	3890 ± 16	3929 ± 330
V	24 ± 25	46 ± 4	34 ± 3	23 ± 2	43 ± 3
Mn	158.1 ± 1.3	249 ± 2	203 ± 2	183 ± 2	127 ± 11
Na	19 ± 6	3659 ± 11	2330 ± 9	1923 ± 10	1620 ± 8
K	1287 ± 243	1854 ± 59	14900 ± 450	9422 ± 424	12130 ± 550
Cr	18 ± 2	54 ± 2	32 ± 2	22 ± 2	36 ± 2
Fe	8580 ± 172	15010 ± 225	17810 ± 249	10320 ± 196	15440 ± 247
Co	4.3 ±0.2	5.2 ± 0.2	4.1 ±0.2	3.1 ± 0.2	6.6 ± 0.3
Zn	ND	42 ± 5	50 ± 5	11 ± 3	36 ± 6

Discussion of Findings

The results on Table 2a, 2b and 2c shows that Magnesium (Mg) was measured in only four samples two each from DanMusa and Safana LGAs with range of 1352±292 to 2179±414 mg/kg. Calcium (Ca) was only measured from DanMusa and Safana LGAs in five samples ranging from 2321±713 to 60105±1062 mg/kg, while Zinc (Zn) was measured in nine samples, with one from Kankia LGA and four each from DanMusa and Safana LGAs also ranging from 10±4 to 53±6 mg/kg. Also measured in all the samples are Titanium (Ti), Vanadium (V), Manganese (Mn), Sodium (Na), Potassium (K), Chromium (Cr), Iron (Fe) and Cobolt (Co) with concentrations ranging from 1949±242 to 4752±371 mg/kg, 13.99±2.70 to 62±3 mg/kg, 91±2 to 427±2 mg/kg, 19±6 to 3659±11 mg/kg, 1287±243 to 118730±229 mg/kg, 8.0±0.3 to 54±2 mg/kg, 6723±194 to 23340±280 mg/kg and 1.37±0.16 to 8.9±0.3 mg/kg respectively.

Apart from Ti which is not essential to plants vis-à-vis human health (Welch & Graham, 2004; White & Broadley, 2005a; Graham *et al*., 2007), all other elements measured are essential and beneficial for plant growth, including Cr and Zn which are heavy elements. Though, some beneficial elements such as Mg and Ca are less available for plant use, and this could be due to the sandy nature of the soil with reduced microbial activities (Preeti *et al.*, 2016). It is also noticed that V, Mn and Fe more available and are above the recommended value allowed for plant growth. The elevated nature of some of these elements could be due to agricultural practices such as the application of fertilizers, bush burning and harmattan dust.

The importance of these elements in human, animal and plant nutrition has been well recognized (Nigel & Sjaan, 2016; Lynette, 2016). Deficiencies or disturbances in the nutrition of an animal causes a variety of diseases and can arise in several ways (Preeti *et al*., 2016)). Mg plays a major role in the production of chlorophyll, on which photosynthesis depends and also activates many enzymes (Williams, 1992). More so, Mg is a co-factor in several enzymatic reactions that activate the phosphorylation processes, and it is required to stabilize ribosome particles and also helps stabilize the structure of nucleic acids, as well as assistance in the movement of sugars within a plant. Ca concentration range required by plants is between 100 to 200 mg/kg (Filby, 1995), as such those measured in this research are adequate, however our result shows that probably due to high pH value in some of the locations, Ca is less available to plant.
Beside, trace elements such as Mn, Fe, Co and Zn measured are also in adequate amount for plant growth, except that Co is gradually becoming elevated, however, Co essentiality has been shown but it deficiency inhibit some elements in plant growth. Fe is above the recommended but within the tolerable level in all the samples because the WHO recommended level of iron in plants is 20 ppm (or mg/kg) (Afzal Shah et al 2013).

V is toxic to humans and other animals (Perez-Benito, 2006). Though, low concentrate-on of V is beneficial to microorganisms, animals, and higher plants (Tisdale *et al.*, 1993). A normal concentration of V required for plant growth is 2.0 parts per billion (ppb), whereas normal V concentration in plant material averages about 1.0 ppm (or mg/kg) (Epstein, 1972). As such the concentration of V in all our samples are extremely high. Though, Ti is not an essential element for plant nutrition and no clear evidence of a biochemical role of Ti has been reported, but according to Preeti *et al*., (2016) it is a physically promotive trace elements, apart from what Chapman (1972) opined earlier that it has catalytic function in nitrogen fixation by symbiotic microorganisms and in photo-oxidation of nitrogen compounds by higher plants and particular processes of photosynthesis. Cr measured in all the samples exceeded the permissible limit of Cr for plants of 1.30 ppm recommended by WHO, though within the world background report (7 – 221 mg/kg), while some are above the range of the US permissible limit of 20 to 85 mg/kg (Sandia Corporation, 2000).

Conclusion

In this present study, we have assessed some essential and beneficial heavy elements in soil of fifteen samples from DanMusa, Kankia and Safana LGAs of Katsina State using k_0-INAA technique via the k_0–IAEA program which involves the simultaneous irradiation of a sample and a neutron flux monitor and the use of a composite nuclear constant called k_0-factor. The facilities used are NIRR – 1 for irradiation and HpGe detector for counting of the samples all located at CERT, Ahmadu Bello University, Zaria. A total of eleven elements (Mg, Ca, Ti, V, Mn, Na, K, Cr, Fe, Co and Zn) were measured with the result showing that most of these elements are adequate and are as essential to productive as well as profitable crop plants just as Nitrogen and phosphorus, except that Mg and Ca are less available for the plants. More so, the cereal plants grown within the study areas have developed the ability to make use of these elements as they are available in the soil.
Cr however, in all our samples were above the permissible limit set by WHO though within the world range. Hence in order to ensure proper use of the soil and to avoid loss of most needed trace elements, we recommend that the farmers desist from bush burning and also practice rotational farming system.

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