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Neutron Activation Analysis (NAA) of *Senna occidentalis* Linn

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Abstract
Senna occidentalis Linn was subjected to neutron activation analysis (NAA) in order to assess its major, minor, trace and ultra-trace contents. The results indicated that Al, Ca, Fe, Na and K have the highest concentration followed by Mn, Zn and Rb while Co, Rb, La, Sc, Sm and Th were in traces. The presence of toxic metals (such as As) in the plant at low levels and absence of others (e.g Cd and Pb) indicates that the plant can be consumed but bearing in mind that long consumption of the plant may lead to their bioaccumulation. The pattern of bioaccumulation of the elements did not follow any particular trend among the different parts of the plant.
Key words: *Senna occidentalis* Linn and NAA.

1. Introduction

Vegetables are the fresh and edible portions of herbaceous plants. They are important food and highly beneficial for the maintenance of good health and prevention of diseases. They contain valuable food ingredients which when successfully utilized, can help to build up and repair the body. They are valued mainly for their high carbohydrate, vitamin, mineral contents and anti-oxidant activities. There are different kinds of vegetables which may be either edible roots, stems, leaves, fruits or seeds. Each group contributes to diet in its own way (Hanif *et al*., 2006; Robinson, 1990).

Some of the vegetables consumed by the rural inhabitants of the third world and developing nations which are believed to have medicinal and nutritive value may contain toxic substances that are detrimental to the health of the consumers (Vashishta *et al.*, 2007). Many workers had been engaged in the extraction of bio-chemicals, structure analysis and antioxidant property of them. One of such plants is *Senna occidentalis* Linn (*Leguminosae, Caesalpinioideae)* which is an annual, woody and ramified plant, with 1.0 to 2.0m height, native to tropical America, which spreads exclusively through seeds (Lorenzi, 2000). The plant is widely distributed throughout the tropical and subtropical regions of the world. It can be found in open pastures and in fields cultivated with cereals such as soybean, corn, sorghum and others; thus during the harvest it is almost impossible to prevent this plant from mixing with the cultivated crops(Lar and Gupta, 1973; Barbisa-Ferreira *et al*., 2005). The antimicrobial, antimalarial, antitrypanosomial and allelophatic activities of the leaves and root bark and nutritional values of this plant have been reported (Tona *et al,* 1999; Samy ad Achimutu, 2000 Tona *et al.,* 2001; Chukwujewu *et al.*, 2006 and Ibrahim *et al*., 2010). Its ethnobotanical studies have revealed the potent cathartic effect of the leaves (Wang *et al*., 2002), used as tea for constipation, treatment of eczema and other skin disorders, treatment for smallpox, measles, potent cure for gonorrhea, pile, control of insects and treatment for fevers (Ogunkunle and Ladejobi, 2006). Also reported is the use of decoction of *S. occidentalis* Linn leaves and flowers as an expectorant in bronchitis and dyspnoea, an astringent and as a mouthwash in stomatis (Ogunkunle and Ladejobi, 2006).

However, information on the elemental composition status of *S. occidentalis* Linn has not been fully documented. Because deficiencies or excesses of minerals especially trace elements leads to various complications and metabolic disorders in human beings. In this article, we report for the first time a comprehensive multi-element analysis of this plant.

For this work, neutron activation analysis (NAA) technique was used, which is capable of determining large number of elements in different matrices (Jonah *et al*., 2006; Achi *et al*., 2012). The Nigeria Research Reactor-1 (NIRR-1) installed and commissioned at the Center for Energy Research and Training (CERT), Ahmadu Bello University, Zaria, Nigeria for training and research was the one used for the analysis. It is a Miniature Neutron Source Reactor (MNSR) and has a tank- in- pool structural configuration with a nominal thermal power rating of 31kW. Like all MNSR facilities, NIRR-1 is specifically designed for neutron activation analysis (NAA) and other functions, therefore its capabilities for the trace, minor and major elements in different sample matrices have been greatly enhanced (Jonah *et al*., 2006; Achi *et al*., 2012). NAA has some unique features, which includes; multi-element nature, rapidity, non-destructibility, sensitivity, accurate, precise, reproducibility of results, complementarity to other methods, freedom from analytical blank and independence of chemical state of elements (Jonah *et al*., 2006).

2. Materials and methods

The plant in this work (*S. occidentalis* Linn) was identified at the Department of Biological Sciences of Ahmadu Bello University (ABU) Zaria and voucher specimen is kept there. Samples of the plant materials (roots, stems, fruits and leaves) were collected from Karkari village, Gwarzo Local Government Area of Western part of Kano State, Nigeria and transported in polythene bags. After collection, the samples were washed twice with tap water and rinsed with deionised water. They were first air dried and then further dried in an oven at 60^{O}C. After drying, the samples were ground using pestle and mortar and passed through 125µm mesh sieve.

NIRR-1 which is a low-power nuclear reactor with highly enriched uranium as fuel, light water as moderator and beryllium as reflector was used for the analysis. The reactor's associated facility for radioactivity measurements are gamma ray data acquisition system. It consists of a horizontal dip-stick High Purity Germanium (HPGs) detector with a relative efficiency of 10% at 1332.4 keV gamma ray line, MAESTRO emulation software compatible with the ADCAM® Multi-channel analyzer (MCA) card, associated electronic modules all made by EG & ORTEC and a personal computer. The efficiency curves of the detector system at near and far source detector geometries have been determined by standard gamma-ray sources in the energy range of 59.5 – 2254keV and extended o 4000keV. For data processing gamma ray spectrum analysis software WISPAN 2004 developed at CIAE, Beijing, China was used. On the basis of the well known activation equation, the software requires that calibration factors be pre-determined by a multi-element standard reference material for elements of interest using adopted irradiation and counting regimes. The WINSPAN 2004 in addition to NAA calculations, perform peak analysis, remote control of MCA and other auxiliary functions such as efficiency calibration and nuclear data generation.

In this work, the certified reference material IAEA-359 (Cabbage) and IAEA-336 (Lichen) were used to validate the results obtained. For the analysis the sample and the reference material of approximately 150mg were weighed and wrapped in polyethylene (rabbit) capsules. The polyethylene capsules were first cleaned by soaking in 1:1HNO_3 for 3 days and washed with de-ionized water.

For irradiation, four schemes (S1, S2, L1 and L2) were adopted based on the half-life of the product radio nuclide. For elements leading to short-lived activation products (S1and S2) the samples were each packed and sealed in 7cm^3 rabbit capsules and sent for irradiation in turn in an outer irradiation channel B_4 where the neutron spectrum is "soft" the choice of the outer irradiation channel is to eliminate corrections, notable Mg in the presence of Al; Al in the presence of Si; and Na in the presence of P. This is due to the proximity of the inner channels of MNSRs to the core leading to relative higher ratio of fast-to-thermal neutron.

For elements leading to long-lived activation products (L1 and L2), samples wrapped in polyethylene films were then irradiated for 6h in any of the small inner irradiation channels (i.e. A_1, B_1, B_2 and B_3) to take advantage of the maximum value of thermal neutron flux in the inner channels. The neutron flux variability over irradiation volume was determined experimentally to be less than 2% through the measurement of specific activities of irradiated Cu wires arranged axially and radially inside the vial. The stability of the neutron flux throughout the period of

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irradiation for the long irradiation was checked by monitoring the neutron flux reading of a fission chamber connected to the micro computer system. The results indicate a stable neutron flux over irradiation period.

Radioactivity measurements of induced radio nuclides were performed by the PC-based gamma ray spectrometry set up. Following the short irradiation regime the first round of counting was performed for 10min (ie.S1) after a waiting time of 2 – 15min. Samples were placed on a Plexi-glass samples holder designed "H2" which corresponds to source-detector geometry of 5cm. The second round of counting was also carried out for 10min. following the short irradiation regime (ie.S2) after a waiting period of 3 – 4 hrs. Samples were counted on a Plexi-glass holder designated as "H1" corresponding to a source-detector geometry of 1cm. The neutron flux setting for the short irradiation of the materials was raised to 5 x 10^{11}n/cm/s sensitivities for analysis of elements using procedures S1 and S2.

In the case of the long irradiation scheme, the first round of counting was carried out for 30min following the long irradiation (ie. L1) using the holder "H1" after a waiting period counting was performed for 60min (ie.L2) after cooling time of 10 – 15days. The samples were counted using Plexi-glass holder "H1". The choice of cooling time and sample detector geometry was such that detectors' dead time is controlled to be less than 10%.

A description of the irradiation and counting regime adopted for NIRR-1 facilities as well as radio nuclide of interest are given in table 1. Identification of gamma-ray products of radio nuclides through their energies and quantitative analysis of their concentrations were achieved using the gamma-ray spectrum analysis software WINSPAN 2004. For the biological materials the certified reference material IAEA-359 (Cabbage) and IAEA-336 (Lichen) were chosen (Jonah *et al*., 2006).

3. Results and discussion

The irradiation and counting schemes together with the WINSPAN multi-element gamma-ray analysis software have been validated using the standard reference materials.

Results in Table1give the routine irradiation and measuring regimes developed for NIRR-1 facilities. Table 2 gives comparison of certified values with our results in ppm or as indicated in % for certified biological reference material, IAEA-336 (Lichen) and IAEA-359 (Cabbage). Table 3 gives nuclear data and limits of detection for the elements of interest using adopted experimental conditions. Table 4 gives the results of INAA of samples of *Senna occidentalis* Linn.

Table 1: Routine irradiation and measuring regimes developed for NIRR-1facilities

Neutron flux/irradiation channel	Procedure	T_{irr}	T_d	T_c	Activation products
$1x10^{11}$n/cm^{2}s/outer irradiation channels (B4, A2)	S1	2min	2-15min	10min	^{28}Al,^{27}Mg,^{18}Cl,^{49}Ca,^{66}Cu,^{51}Ti,^{52}V,^{116m}In
	S2	2min	3-4h	10min	^{24}Na,^{42}K,^{165}Dy,^{56}Mn,^{152m}Eu
$5x10^{11}$n/cm^{2}s/inner irradiation channels (B1,B2,B3,L2 and A1)	L1	6h	4-5d	30min	^{239}Np(U),^{72}Ga,^{122}Sb
	L2	6h	10-15d	60min	^{46}Sc,^{141}Ce,^{60}Co,^{51}Cr,,^{134}Cs,^{152}Eu,^{177}Lu,^{131}Ba,^{86}Rb,^{182}Tb,^{175}Yb,^{233}Pa(Th),^{65}Zn,^{59}Fe,^{181}Hf

Table 2: Comparison of certified values (CV) with our results in ppm or as indicated in %for the certified reference materials, IAEA-336 (Lichen) and IAEA-359 (Cabbage).

Element	IAEA-336(Lichen)		IAEA-359 (Cabbage)	
	This work (TW)	(CV)	This work (TW)	(CV)
Al	709±35	570-790	175±13	-
As	BDL	0.55-0.71	BDL	0.096-0.104
Ba	BDL	5.3-7.5	BDL	10.5-11.5
Br	11.0±1.3	11.2-14.6	8.0±3.0	-
Ca (%)	BDL	-	1.97±0.12	1.8-1.9
Ce	BDL	1.11-1.45	BDL	-
Co	BDL	0.24-0.34	BDL	-
Cr	BDL	0.89-1.23	BDL	1.24-1.36
Cs	BDL	0.097-0.123	BDL	-
Cu	BDL	144	BDL	78.6
Eu	BDL	0.019-0.027	BDL	-
Fe(%)	BDL	0.038-0.048	BDL	0.014-0.15
K (%)	BDL	0.16-0.21	3.3±0.4	3.18-3.32
La	0.66±0.04	0.56-0.76	0.27±0.05	-
Lu	BDL	0.004-0.009	BDL	-
Mg (%)	BDL	-	BDL	0.21-0.22
Mn	61±2	56-70	29.4±0.8	31.3-32.5
Na	317±	280-360	676±30	567-601
Rb	BDL	1.54-1.98	BDL	-
Sb	BDL	0.063-0.083	BDL	-
Sc	0.16±0.03	0.15-0.17	BDL	-
Sm	BDL	0.092-0.12	BDL	-
Tb	BDL	0.012-0.016	BDL	-
Th	BDL	0.12-0.16	BDL	-
V	BDL	1.25-1.69	BDL	-
Yb	BDL	0.025-0.049	BDL	-
Zn	BDL	27.0-33.8	BDL	-

BDL: Below detection limit

Table 3: Nuclear data and limits of detection for the elements of interest using adopted experimental conditions

Target isotope	Product isotope by (n,γ) reaction	Half-life	Gamma-energy (keV)	LOD(ppm)
^{23}Na	^{24}Na	14.96h	1368.60	40(L1)
^{26}Mg	^{27}Mg	9.46min	1014.4	7250(S1)
^{27}Al	^{28}Al	2.24min	1778.99	17(S1)
^{37}Cl	^{38}Cl	37.24	1624.7	2900(S1)
^{41}K	^{42}K	12.36h	1524.58	2400(S2)
^{45}Sc	^{46}Sc	83.81d	889.28	0.2(L2)
^{48}Ca	^{49}Ca	8.72min	3084.54	6600(S1)
^{50}Ti	^{51}Ti	5.76min	329.08	2500(S1)
^{50}Cr	^{51}Cr	27.7d	320.98	23(L2)
^{51}V	^{52}V	3.75min	1434.08	15(S1)
^{55}Mn	^{56}Mn	2.58h	846.76	0.9(S2)
^{58}Fe	^{59}Fe	44.5d	1099.25	829(L2)
^{59}Co	^{60}Co	5.27y	1173.2	3.0(L2)
^{65}Cu	^{66}Cu	5.10min	1039.2	172(S1)
^{64}Zn	^{65}Zn	243.9d	1115.55	120(L2)
^{71}Ga	^{72}Ga	14.1h	834.1	1.0(L1)
^{75}As	^{76}As	26.32h	559.10	1.2(L1)
^{81}Br	^{82}Br	35.3h	776.5	3.0(L1)
85Rb	^{86}Rb	18.8d	1076.6	3.0(L2)
^{115}In	^{116m}In	54.15min	1097.3	0.5(S1)
^{121}Sb	^{122}Sb	64.8h	564.24	0.5(L1)
^{133}Cs	^{134}Cs	2.06y	795.85	1.7(L2)
^{130}Ba	^{131}Ba	11.8d	496.3	264(L2)
^{139}La	^{140}La	40.3h	1596.21	0.2(L1)
^{140}Ce	^{141}Ce	32.5d	145.44	14(L2)
^{151}Eu	^{152}Eu	13.3y	1408.5	0.6(L2)
^{152}Sm	^{153}Sm	46.27h	103.18	0.1(L1)
^{159}Tb	^{160}Tb	72.3d	879.38	1.1(L2)
^{164}Dy	^{165}Dy	2.33h	94.70	0.7(S2)
^{174}Yb	^{175}Yb	4.19d	396.33	0.9(L1)
^{176}Lu	^{177}Lu	6.71d	208.36	0.1(L2)
^{180}Hf	^{181}Hf	42.4d	482.2	1.1(L2)
^{181}Ta	^{182}Ta	115d	1221.4	1.0(L2)
^{197}Au	^{198}Au	2.7d	411.8	0.02(L1)
^{232}Th	^{233}Pa	27.00d	312.01	1.2(L2)
^{238}U	^{239}Np	2.36d	277.60	1.5(L1)

S1, S2, L1 and L2 represent irradiation and counting schemes adopted for the respective element.

Table 4: Determined elements and concentrations of *Senna occidentalis* Linn in ppm using NIRR-1.

Element	Roots (SOLR)	Stems (SOLR)	Leaves (SOLL)	Fruits (SOLF)
Al	128±11.0†	380.0±38.0†	380.0±38.0†	53.0±5.0†
As	1.08±0.04	BDL	BDL	BDL
Ba	BDL	BDL	81.0±18.0	BDL
Br	BDL	BDL	4.0±0.8	BDL
Ca	43336.0±659.0‡	8867.0±1293‡	18310.0±2655.0‡	8800.0±1293.0‡
Co	0.41±0.09	BDL	BDL	BDL
Cr	BDL	BDL	BDL	BDL
Cs	BDL	BDL	BDL	NA
Cu	NA	NA	BDL	BDL
Dy	BDL	BDL	BDL	BDL
Eu	BDL	BDL	BDL	BDL
Fe	2215.0±89.0‡	BDL	390.0±55.0‡	BDL
Hf	BDL	BDL	BDL	BDL
K	7949.0±119.0‡	16090±290.0‡	33690.0±438.0‡	15850.0±127.0‡
La	0.74±0.03‡	0.16±0.02‡	1.23±0.02‡	0.14±0.01‡
Lu	BDL	BDL	BDL	BDL
Mn	7.1±0.3‡	7.5±0.3‡	46.0±3.0‡	13.0±1.0‡
Na	278.0±2.0‡	131.0±2.0‡	319.0±3.0‡	36.2±0.5‡
Rb	8.4±1.7†	BDL	24.0±2.0†	5.5±0.5†
Sb	BDL	BDL	0.29±0.05	BDL
Sc	0.03±0.01†	BDL	0.06±0.01†	BDL
Sm	0.12±0.01†	BDL	0.11±0.01†	BDL
Th	BDL	BDL	0.17±0.05	BDL
U	BDL	BDL	BDL	BDL
V	BDL	BDL	BDL	BDL
Yb	BDL	BDL	BDL	BDL
Zn	82.0±5.0‡	BDL	60.0±5.0‡	35.0±3.0‡

BDL:Below detection limit; NA: Not analyzed and †element determined in the part of the plant are not significantly different while ‡ are different at $p<0.5$.

The results of the elemental analysis (Table 4) have indicated that Al, Ca, Fe, Na and K have the highest concentration followed by Mn, Zn and Rb while Co, Rb, La, Sc, Sm and Th are in traces. Macro elements such as calcium and potassium are found in higher concentrations in all the parts of *S. occidentalis* L. In fact, values for Ca conforms to another work elsewhere on the same plant (Odhav *et al*., 2007) and lower values were obtained in *S. siamea* while similar values were obtained for K (Alli-Smith, 2009) and higher values were found in *G. arborea* seeds (Akinjagunla *et al*., 2007).

K is essential to all organisms with the possible exception of blue green algae. It is a major cation and is important in nerve action. This cation is present in intracellular fluid. Within the cell it functions as sodium in extracellular fluid by influencing acid base equilibrium, osmotic pressure and water retention. When present in extracellular fluid it influences muscle activities. However, it is moderately to mammals when injected intravenously (Singh *et al.*, 2010). The samples have relatively low amount of Na compared to that of K. A K/Na ratio in diet is an important factor in the prevention of hypertension and arterosclerosis, since K depresses and Na enhances blood pressure (Yoshimura *et al.*, 1991). It has been indicated that a K/Na ratio of 3 – 4 is considered the most adequate for the normal retention of protein during growth stage (Guil-Guerrero *et al*., 1998). The estimated K/Na ratio in *S. occidetalis* L. are above the range, but addition of NaCl however, in the diet prepared with this plant leaves is expected to bring the ratio within the range. Ca is essential for all organisms and used in cell wall, bones and some shells as structural component; important electrochemically and by coordination.

The amount of Al in S. *occidentalis* L. is and ranged from 52.0 – 380.0ppm and until recently, Al was considered harmless for the human organism as it is readily excreted through urine. However, studies of environmental toxicology conducted in recent years indicated that Al could be a cause of many diseases in humans, animals and plants (Barabasz *et al*., 2002) attributed to its strong influence on the activity of many enzymes.

Zn found in appreciable concentration in S. *occidentalis* L. is essential to all organisms and an important trace element having definite role in metabolism, growth and development (Singh and Garg, 1997). It is an essential component of over 200 enzymes having both catalytic and structural roles. Zn deficiency is characterized by recurrent infections, lack of immunity and poor growth (Singh *et al.*, 1997). Low intake of zinc my cause coronary disease. Clinical materials prove that Zn can have good effect on eliminating ulcer and promoting

healing of wounds (Singh *et al.*, 2010).

Iron which ranged from 390.0ppm in the leaves to 2215.0ppm in the roots of *S. occidentalis* L. is essential for human body in the production of hemoglobin, in the oxygenation of red blood cells. It is needed for a healthy immune system and for energy production. Severe iron deficiency results in anemia and red blood cells that have a low hemoglobin concentration. In young children, iron deficiency can manifest in behavioral abnormalities (including reduced attention) reduced cognitive performance and slow growth. In adults, severe iron deficiency anemia impairs physical work capacity (Singh *et al.*, 2010).

The functions and uses of Mn, Co, Br, As and Rb have been reported in the literature (Frausto da Silva and Williams, 2006; Fank *et al.*, 1976). The functional values of Mn are as a Lewis acid and catalyst for oxidation. It is essential to all organisms, activates numerous enzymes. Cobalt is essential in trace amounts for human life. It is part of vitamin B-12, and plays a key role in the body's synthesis of this essential vitamin. Cobalt has also been used as a treatment for anemia, because it causes red blood cells to be produced. The toxicity of cobalt is quite low compared to many other metals in soil. Exposure to very high levels of cobalt can cause health effects. Effects on the lungs, including asthma, pneumonia, and wheezing, have been found in workers who breathed high levels of cobalt in the air. Arsenic takes a role in the metabolism of methyl compounds and the deficiency of it will lead to impairment of growth, reproduction and heart's function. Br may be essential in mammals. It is non toxic except in oxidizing forms. The function is not clear so far. In the present study, the concentrations of K and Ca were found to be high followed by the remaining elements in trace levels.

For toxicity, the presence of arsenic in S. *occidentalis* L. will not to any undesirable effect because its concentration is very low as per World Health Organization, Maximum Tolerable Daily Intake (WHO-MTDI) value (2µg/day/kg body weight). The non detection of Pb and Cd leads to the fact that the plants is grown in pollution free areas because metal content of the plant depend largely on regional soil characteristics and climate condition (Singh *et al*., 2010).

4. Conclusion

The plant *S. occidentalis* Linn from the data reveals that it contains appreciable amount of major, minor, trace, and ultra-elements which were detected and determined by NAA technique as it is one of the most powerful techniques for its multi-elemental trace analysis capability and high sensitivity. These elements take definite and specific roles during metabolism in the human body. So, it will be very useful if the molecular structure of the compounds containing the trace elements in the plants be elucidated. The different concentration of the elements in different plants leads to the conclusion that the plants will have different specific roles in the treatment of different diseases.

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