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Research Paper

Analysis of essential elements in Ethiopian finger millets (eeleusine coracanda) by instrumental neutron activation analysis (INAA)

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Abstract

The objective of this study was to determine essential elements in white, black and mixed species of Ethiopian finger millets using neutron activation analysis (NAA). The samples were collected from the local market of Bahirdar city, in Northwest Ethiopia. After the samples were dried, milled, heat sealed in a polythene sheet and put in a vial, they were irradiated in the irradiation channels of the Nigeria research reactor. The activities of the samples were measured using HPGe gamma spectrometry and analyzed by Winspan 2004 software. A total of 16 elements (Ca, Mg, K, Cl, V, Al, Ba, Na, Mn Fe, Zn, La, Sc, Sm, Br and Rb) were analyzed in three of the samples. According to the analyzed data Ca, Mg and K as macronutrients were found in range of 592 ± 21 ppm to 4876 ± 36 ppm, elements Fe, Al, and Cl as micronutrient were obtained in the range of 156 ± 18 to 775 ± 42 ppm with all others elements measured in traces. The results, therefore, showed that black and mixed finger millets are significantly enriched in essential elements. In addition, finger millets have also medicinal properties for controlling diabetes, blood pressure, anemia, stomach cancer and Alzheimer because they contained in pharmacological elements: Mg, Fe, Zn, V and Mn.

Keywords: *Finger Millet; INAA; Macro and Macronutrient; Medicinal Plants; SRMS.*

1. Introduction

Cereal crops are the major source of food for the human population of the world. One of those important sources of cereal food is finger millet (Eleusine coracanda). Finger millet is widely cultivated as an important staple food in many parts of eastern and central Africa and India [1].

Finger millet is an excellent source of micronutrients, which could alleviate the wide spread micronutrient malnutrition in the developing countries [2]. As literatures suggest finger millet is a stable food for man and animals due to its better nutritional quality. It contains more percentage of different nutrients like calcium, iron, magnesium, phosphorus, potassium and zinc (3-6). The importance of Eleusine coracana is not only providing essential nutrients but it is also rich in nutritional values to the diet in terms of carbohydrate, protein, fat and sorghum [4], [7], [8]. Shashi et al., (2007)[2] explained in their report that the total carbohydrate content of finger millet is in the range of 72% to 79.5 % and its protein content in nearly 7% [3]. Finger millet, apart from its nutritional values, has also significant medicinal properties for treatment and prevention of cancer initiation. In India, for instance, it is used traditionally as remedy for leprosy, liver disease, measles, pneumonia and small pox [7], [9].

Finger millet also known as Dagussa (in Ethiopian Amharic language) is a native and indigenous food crop to Ethiopia and has been harvesting in different parts of the country for centuries [4]. Usually it is grown almost exclusively once a year. However, in Ethiopia, there is still limited information on what essential elements the finger millet contained in and in what amounts. Thus, the present study was carried out with the objective to find scientific evidences what essential or toxic chemical elements are there in white, black and mixed finger millets. The method employed to study the elemental composition of finger millets was Instrumental Neutron Activation analysis (INAA). Instrumental neutron activation analysis has been proved to be a versatile, sensitive and widely employed non-destructive technique to characterize and determine multi-elements present in geological and biological samples at major, minor and trace levels.

2. Materials and methods

2.1. Irradiation facilities

The research facility used for this study was the Nigerian Research Reactor (NIRR-1) swimming pool-type reactor operating at 30.5 kW, which was installed and commissioned in 20014 at “Center for Energy Research and Training (CERT” in Ahmadu Bellow University, Zaria, Nigeria. According to Yamusa et al., (2013) the research reactor uses high enriched uranium as a fuel and light water as moderator and coolant [10].

2.2. Sample collection and preparation

The three finger millets white, black and mixed as shown in figure 1 were obtained from the local market of Bahirdar city, in Northwest, Ethiopia. Bahirdar city is located at geographic coordinates of 11^{0}35’37” North latitude and 37^{0} 23’26” East longitudes. The samples were washed off using deonized water to clean from dust particles. They were then dried and subjected to milling using clean agate mortar into very fine powder. The powder form of the samples was again dried at 45 ^{0}C in an oven for 24 hrs until each sample deserved a constant weight. Finally, a representative small mass from each sample in the range of 0.250-0.300g was weighed using a four-digit Meter model weighing balance and heat sealed in a cleaned polyethylene sheet. In addition, for the purpose of quality assurance a standard reference material (SRM) of biological origin known as apple leaves (NIST 1515) which was equivalent to the mass of the samples was prepared and packed together with samples in such way suitable for short and long irradiation schemes.

Fig. 1: White, Mixed and Black Types of Finger Millet

2.3. Irradiation and counting

The samples of finger millet and standard reference material NIST 1515 were irradiated simultaneously inside the irradiation channels of the NRR-1 research reactor via pneumatic Rabbit System that operating at a pressure of 0.5Mpa. There were two irradiation schemes designed to determine the varying half-lives of radionuclides present in the sample [11]. These two schemes were short and long irradiations.

For short irradiation scheme, each sample was irradiated for a period of 5 minutes at a neutron flux of $2.5x10^{11} n.m^{-2}s^{-1}$ in the outer irradiation channel B_4 and then allowed to cool for about 2-15 minutes. After such cooling , the samples was then transferred to the Cortec coaxial HPGe detector connected to a desktop PC via the associated electronics and then the first round of counting was done for 10 minutes (1S or 1st short irradiation counting). Similarly, after 3-4 hrs waiting periods for cooling the same patch of samples, a second round counting was performed again for 10 minutes (2S). In this short irradiation radionuclides with short half-lives in the order of minutes were analyzed. In the case of long irradiation scheme, about 6-7 samples including the standard were irradiated simultaneously for 6hrs at neutron flux of $5x10^{11} n.m^{-2}s^{-1}$inside the inner core channel B3 of the same facility. After 3-4 days of waiting period for cooling, each sample was counted for 30 minutes (1L) to find radionuclides having half-lives in the order of hours or few days.

Similarly, to detect and analyze radionuclides with half-lives in the order of days and years, a second round of counting for 1 hr (2L) was done for the same patch of samples after 9-11 days waiting periods for cooling.

2.4. Gamma spectrometry analysis

The counting of the samples and standard reference material (SRM) appeal leaves (NIST 1515) was performed using gamma data acquisition which consist of HPGe detector with relative efficiency of 30% at 1332.5Kev gamma line, Maestro emulation software compatible with the multi-channel analyzer (MCA) and associated with electronics modules all made by EG and G ORTEC personal desktop computer [12]. However, before the start of counting the activities of irradiated samples, efficiency calibration and standardization of the detector was carried out using standard sources as ^{137}Cs, ^{22}Na, ^{60}Co, ^{242}Am, and ^{152}Eu at a positions of 2 cm and 15cm above the detector. Similarly, the energy calibration of the detector system was set at a Full Width Half at Maximum (FWHM) = 2.0 kev at 1332kev resolution using ^{60}Co.

In INAA there are three methods for the multi-elemental analysis of samples. These are known as absolute, relative, K_0 methods. Each method has its own merits and demerits. But in present work, the relative method was chosen for the reason that uncertainties that could appear due to nuclear parameters such as cross sections, neutron flux, decaying scheme and detector efficiency would be eliminated. Since the samples and standards were irradiated and counted under the same geometry, the nuclear parameters get cancelled and thus only measuring parameters such as the activity rates, irradiation time, cooling time and counting time of the irradiated samples were considered. In relative method, the net peak areas for the gamma-rays of radionuclides in the samples were calculated and compared with those of Apple leaves (NIST 1515) and the concentration of each radionuclide in the sample could be determined using the equation (1).

$$C_{sam} = C_{std} \frac{A_{sam}}{A_{std}} \frac{M_{std}}{M_{sam}} \frac{(e^{-\lambda t_d})_{std}}{(e^{-\lambda t_d})_{sam}} \quad (1)$$

Where C_{sam} and C_{std} are the concentration elements in the unknown and standard samples, A_{sam} and A_{std} are activity rates of the elements in unknown and the standard samples, M_{sam} and M_{std} are the masses of the sample and standard, t_d is the decay time for the sample and standard, $(e^{-\lambda t})_{sam}$ and $(e^{-\lambda t})_{std}$ are decay factors of the element in the sample and Standard, $\lambda = \frac{\ln}{T_{1/2}}$ the decay constant and $\tau_{1/2}$ is nuclide half time.

Equation (1) can be reduced into equation (2) if $M_{sam} = M_{std}$

$$C_{sam} = C_{std} \frac{A_{sam}}{A_{std}} \frac{(e^{-\lambda t_d})_{std}}{(e^{-\lambda t_d})_{sam}} \quad (2)$$

Finally, the gamma rays which are unique (or finger print) to each of the elements in the activated samples were identified through their energies qualitatively from the energy spectra in way a similar that shown in figure 2 for black finger millet. The concentrations of those elements were also determined quantitatively as shown in table 2 and 3 using the gamma-ray spectrum analysis software Winspan 2004, software developed at CIAE, Beljing, China.

3. Results and discussions

The accuracy of neutron activation analysis (NAA) was evaluated by analyzing appeal Leaves (NIST 1515) of biological origin which was irradiated with the samples. As can be seen from the table 2 the measured concentration of apple leaves was in good agreement with the certified values.

In this work, the concentrations of 16 chemical elements in white, black and mixed finger millets were determined using the neutron activation analysis (NAA). The concentrations of these elements are tabulated in table 3. In short irradiation schemes nuclides with short half-lives such as Mg, Al, Ca, Cl, Na, Mn and K were identified whereas in long irradiation scheme, nuclides with long half-lives such as Br, La, Sm, Sc, Fe, Zn, Rb, and Ba were determined as shown in table 2. The appropriate half-lives and the energies of these elements were taken from nuclear data table of isotopes [13].

Among the 16 elements Mg, K, and Ca were found the highest in three of the finger millets as displayed in figure 2. According to the report of Shobanna et al., (2013)[14] calcium is significantly rich enough in finger millet compared to other cereals and millet species. In this study, Ca concentration in three of the finger millet samples was found the highest in the range of 4430±229 to 4876±36 ppm. Calcium is an essential macro-nutrient for building and giving strength to bones and teeth of human body [15]. Apart from mineralization of bones and teeth, Ca is important for the proper functioning of heart and nerve systems, blood clotting and for the relaxation and contraction of muscular system.

Magnesium like calcium was also found in the category of macro-nutrient in three of the finger millet. The highest concentration of Mg was observed 1907 ±246 ppm in mixed finger millet and lowest 1789±291 ppm in white finger millet. This indicates that the mixed finger is better in Mg content than the black and white finger millet. The recommended daily intake of Mg is 3950mmg/day for men and 300mg/per day for women. As literatures suggest taking Mg rich crops as a diet is very important to human body because Mg has a major role for proper functioning of nerve system, muscle control and blood pressure. However, a human body which is subjected to low level of Mg content is easily exposed to such as hypertension, cardiovascular disease, diabetes, asthma, headache and stress.

Potassium (K) similar to Ca and Mg is the third macronutrient in three of the finger millets as shown in figure 3. The concentration of K was found 2407±19 ppm highest in mixed finger millet and 592±11 ppm lowest in white finger millet. Potassium is an essential nutrient and has a vital role as electrolyte in blood and for the smooth flow of signals from cell to cell in human body. It is also an activator of some enzymes for the normal growth and muscle function [16]. The recommended average intake of K is 2300mg/day for adult women and 3100 mg/for adult men. However, lack of K in human body causes diseases like hear stroke, diabetes and hypertension [17]. This is, therefore; according to the result of this study mixed finger millet is more important to minimize disease like stroke, diabetes and hypertension.

Table 1: Elemental Concentrations of Apple Leaves (NIST-1515) Used for Quality Control Purpose (Mean ± SD)

Element	This work (in ppm)	Certified value(in ppm)
Mg (%)	0.2710±0.005	0.2710±0.008
K (%)	1.526±0.044	1.526±0.015
Ca (%)	1.526±0.044	1.526±0.015
Al	286±5	286±9
Cl	579±20	579±23
V	0.26±0.05	0.26±0.03
Br	1.8 ±	(1.8)
Mn	54±1	54±3
Na	24.4±0.5	24.4±1.2
Ba	49±3	49±2
La	20.00±0.08	(20)
Sm	3.00±0.01	(3.00)
Sc	0.030±0.007	(0.030)
Fe	83±26	83±5
Zn	12.5±0.7	12.5±0.3
Rb	10.2±0.3	10.2±1.5

% = percentage (or x10^4 in ppm), Ppm = parts per million, Values given in parentheses ( ) are information values

Table 2: Radionuclides Measured by (n,γ) Reaction During Short and Long Irradiations, and Their Corresponding Half-Life, Gamma Energies [13], Irradiation Time ($T_{\_Irr}$), Decay Time (T_d) and Counting Time (T_c).

Element	Target	Product nuclide	Half-life ($T_{1/2}$)	Energy (in Kev)	Irradiation time ($t_{\_irr}$), Decay (t_d) time, counting time (t_C). and counting schemes (short and long)
Mg	^{26}Mg	^{27}Mg	9.46 min	1014.4	t_{irr} = 5min , t_d = 3-15 min t_c = 10 min , 1st Short (1S)
Al	^{27}Al	^{28}Al	2.24 min	1779.0	
Cl	^{37}Cl	^{38}Cl	37.2 min	2167.7	
Ca	^{48}Ca	^{49}Ca	8.72 min	3084.5	
Na	^{23}Na	^{24}Na	15.0 hrs	2754.0	t_{irr} = 5 min , t_d = 3-4 hrs t_c = 10min, 2nd short (2S)
K	^{41}K	^{42}Na	12.4 hrs	1524.5	
Mn	^{55}Mn	^{56}Mn	2.58 hrs	1810.7	
La	^{139}La	^{140}La	40.3 hrs	1596	t_{irr} = 6 hrs, t_d = 3-4 days t_c = 30 min, 1st long (1L)
Sm	^{79}Sm	^{80}Sm	46.3 hrs	103.2	
Br	^{81}Br	^{82}Br	35.3 hrs	776.5	
Sc	^{45}Sc	^{46}Sc	83.8 days	889.3	t_{irr} = 6 hrs , t_d = 9-11 days t_c = 1h , 2nd long counting (2L)
Fe	^{58}Fe	^{59}Fe	44.5 days	1291.6	
Zn	^{64}Zn	^{65}Zn	244 days	1115.6	
Rb	^{85}Rb	^{86}Rb	18.7 days	1076.6	
Ba	^{130}Ba	^{131}Ba	11.8 days	496.3	

Aluminum, chlorine and iron were obtained at micronutrient level in the three of the finger millets as depicted in figure 4. However, although Al was categorized as micronutrient its concentration obtained in black finger millet (1469 ±59 ppm) was significantly high. It was found 5.5 times larger than Al concentration in white finger millet (268 ±19 ppm) and 2.7 times larger than concentration of mixed finger (541 ±15ppm). The possible cause for the high concentration of Al in black finger millet could be due to the high absorption of Al by black finger millet from the soil where the finger millet grows.

Although aluminum is the third most abundant metal on the Earth’s crest but it has no known physiological or pharmacological use in human body [18]. Reports also indicate that unnecessary intake of Al in human body could be the cause of many diseases like neurological dementia (or kidney dialysis), Parkinson and Alzheimer. The daily intake of Al by adult is about 7 to 10mg/day [19].

Table 3: Elemental concentration of three types of finger millets in this work (in ppm)

Element	White finger millet (in ppm)	Black finger millet (in ppm)	Mixed finger millet (in ppm)
Mg	1789±229	1886±411	1007±246
K	592±11	1076±20	2407±19
Cl	387±21	219±19	467±20
Ca	4403±299	4876±36	4786±297
Al	268±19	1469±59	541±15
V	BDL	4.3±0.7	0.9±0.2
Na	2.73±0.06	10.73±0.16	10.65±0.12
Br	7.98±0.05	7.58±0.05	17.36±0.02
Mn	54.9±0.3	52.4±0.3	57.3±0.3
La	0.095±0.008	0.34±0.10	0.30±0.01
Sm	0.017±0.002	0.087±0.003	0.061±0.002
Sc	0.07±0.01	0.46±0.01	0.115±0.009
Fe	194±36	775±42	156±18
Zn	15±3	24±3	16±2
Rb	6.2± 0.6	BDL	13.6±1.9
Ba	57±15	BDL	BDL

BDL = Below Detection Limit, ppm = parts per million

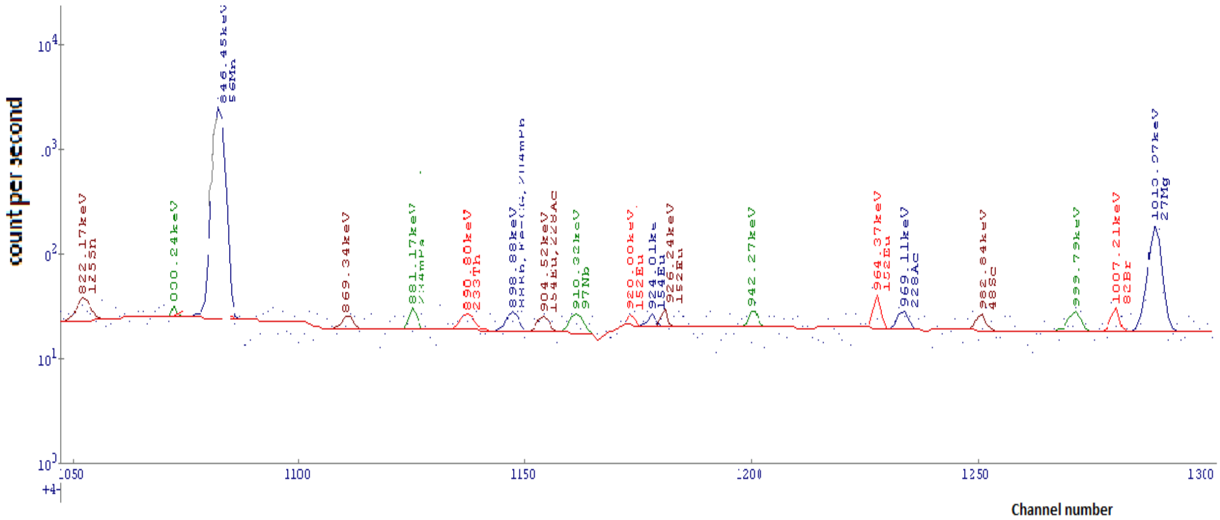

Fig. 2: A Typical Gamma Ray Spectrum of Black Finger Millet Viewed Partially At T_{irr} = 5min, T_d = 4 Min and T_c = 10min

Chlorine (Cl) and iron (Fe) are the most essential components of nutrients that are required by human body. The concentration of chlorine obtained in this work ranged from 219 ±19 ppm in black finger millet to 467 ±20 ppm in mixed finger millet as micronutrient. With regard to the biological importance, the role of chlorine in human body is to act as an anion in the extracellular fluid in plasma, lymph, connective tissue [20]. However, the excess doses of Cl intake could be the cause for malfunction of nervous system and organs on such as liver and lungs [21]. Similarly Iron concentration in three of the finger was recorded in the range from 156±18 ppm in white millet to 775 ±42 ppm in black millet. Iron is important for the formation of hemoglobin in red blood cells and also for facilitating the oxidation process of carbohydrates, protein, and fat to control body weight. The permissible limit of iron for human body as set by FAO/WHO (1984) is 20 ppm [22]. But in this study the concentration of Fe was found above the permissible limit. Studies, however; suggest that excess intake of Fe becomes hazardous to human health rather benefits.

The concentrations of Na, Mn, V and Zn in this work were measured in traces in all samples except vanadium (V) in white millet and rubidium (Rb) in black finger millet which were obtained below detection limit. The concentration of sodium was found in the range 2.73±0.06 ppm to 10.73 ±0.16 ppm at trace level. Biologically, sodium is important for maintaining acid-base equilibrium in human body and also acts as electrolyte in transmitting nerve impulse and in relaxing muscles of human body [23, 24]. The excess or deficiency of Na intake in human body could be subjected to water retention, high blood pressure, stomach ulcer and cancer, confusion, weakness, seizures, coma and even death.

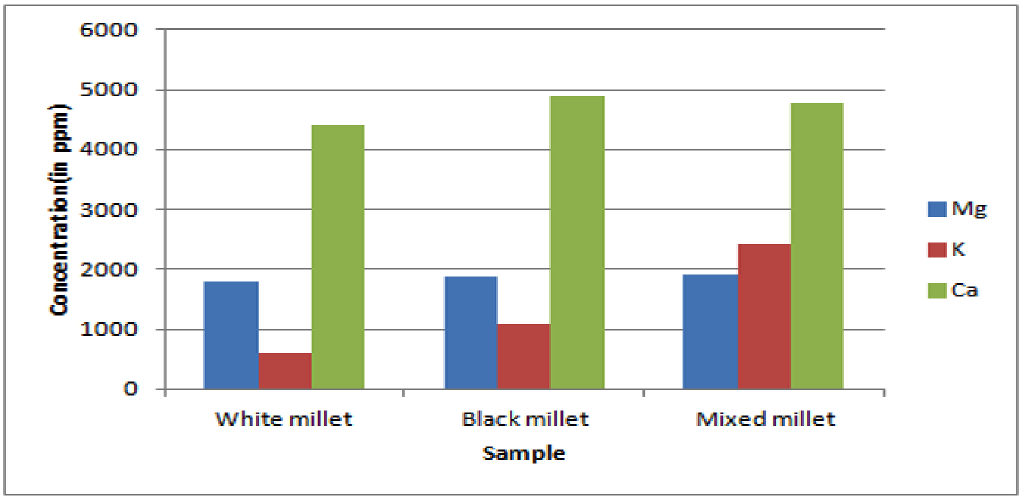

Fig. 3: Macronutrients in Three Finger Millet Sample

Similarly, Zinc (Zn) concentration like Na was found in trace amount. It ranges from 15±3 ppm ppm to 24±3 ppm in three of the millets. Zinc is found important in wound healing, nervous system, reproductive and immune systems, metabolic function, malaria treatment and as well as the treatment of diabetes Mellituses) [25]. The permissible limit of Zn set by FAO/WHO (1984) is 27.4 ppm [26].

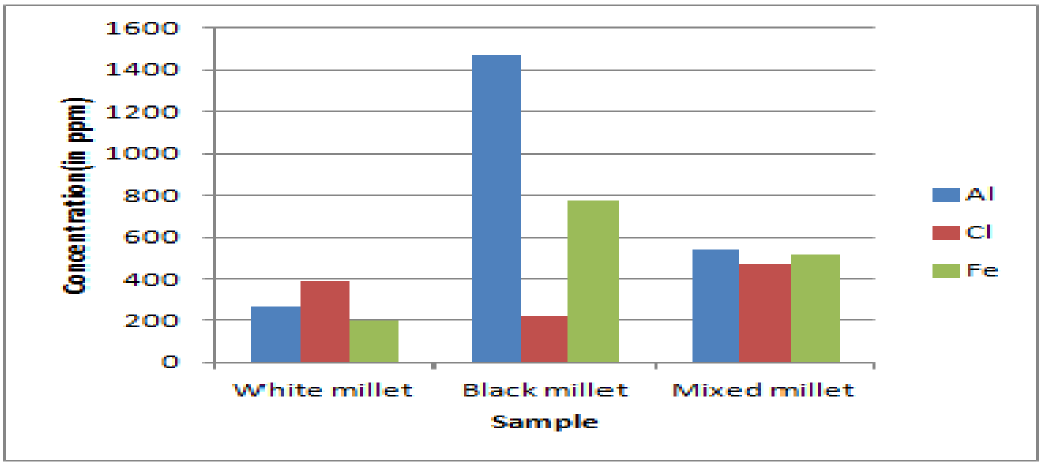

Fig. 4: Micronutrients in Three Finger Millet Sample

The concentration of manganese in this study was ranged from 52±3 ppm to 75.3 ± 0.3 ppm above the permissible limit. Mn is an essential element required for various bio-chemical and enzymatic processes. It is also important for eliminating tiredness, fatigue and nervous irritability in human body [27]. The permissible limit of Mn as estimated by FAO/WHO (1984) is 2ppm [22]. The other trace element found in this study was vanadium with the concentration of 4.3 ±0.7 ppm in black millet and 0.9±0.02 ppm in mixed millet. Vanadium is an essential nutrients found in many food [28]. It is important for bone formation, cellular replication and for treatment of diabetes Mellituses. The fifth trace element obtained in study was rubidium (Rb). Its concentration ranges from 6.2 ±0.6 ppm to 13.6±0.9 ppm in white and mixed millets. Rubidium is required for the production of hormones and various enzymes in human body [28].
Although the concentration of other nonessential elements such as Ba, Br, La, Sm and Sc was obtained in the range of 0.095 ± 0.008 ppm to 57 ±15pm in the finger millet samples many reports suggest that their biological and physiological importance have not yet known.

4. Conclusion

Applying the relative method of instrument neutron activation analysis A total of 16 elements (Mg, Cal, Cl, K, Al, V, Na, Mn, Fe, Zn, Ba, Br, La, Sm, Sc and Rb) were determined in three of the finger millet samples using instrumental neutron activation analysis (INAA). Among the 16 elements Ca, Mg and K were found as micronutrients; Al, Cl and Fe were obtained as micronutrients; Na, Mn, Rb, Zn, and V measured in traces with Ba, Br, La, Sc and Sm as nonessential elements measured at trace levels.

The data set in this work showed that the white, black and mixed Ethiopian finger millets are enriched in essential nutrients like Ca, Mg, K, Fe, Na, Al, Cl, Zn, Rb and V. In particular, the concentrations of essential nutrients in black and mixed finger millets were found to be higher than that of white finger millet. This implies black and mixed finger millets were found significantly enriched in nutritional values. What's more, in this study apart from their quality of nutritional values, they contained in essential elements like Mg, Zn, Mn, and V that have medicinal properties for treatment and prevention of malaria, diabetes Mellituses, anemia, Alzheimer, stomach ulcer and cancer, and for wound healing and controlling blood pressure.

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