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DETERMINATION OF TRACE ELEMENTS IN VARIOUS KINDS OF BEAN BY X-RAY SPECTROMETRIC TECHNIQUES

(1995 - 96)

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DETERMINATION OF TRACE ELEMENTS IN VARIOUS KINDS OF BEAN BY X-RAY SPECTROMETRIC TECHNIQUES

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ABSTRACT

Various kinds of bean such as Peanut, Gram Whole, Black Eye Bean, Small Red Bean, Lab Lab Bean, Green Mung Bean, Filed Pea, Seasame Seed, Sultani, Maize, ButterBean, Dolichos Lab Lab, Toor Whole, Small Yellow Bean, Cow Pea have been collected and analysed by EDXRF analysis for trace elements. The measurement system consists of a Cd-109 annular excitation source, a Si (Li) detector, H V power supply, a Spectroscopy amplifier, a Multichannel Analyser and a personnel computer. The samples were prepared as pressed pellets and measured by Emission Transmission Technique. The accuracy was determined by analysing standard reference material, SOIL-7 form IAEA.

1. INTRODUCTION

Our country needs to produce more agricultural products. The use of fertilizers and improved agricultural management techniques are necessary. Although the use of mineral fertilizers costs money, the benefical effects of them has been proven for many decades. For economic and environmental reasons, however, fertilization should be carried out with great care. Excessive use of fertilizers and agrochemicals give some problems. In fact, determination of trace elements in agriculture products are required in order to improve the efficiency of fertilizer use and to protect the environment.

Energy Dispersive X-ray fluorescence analysis have become a powerful instrument for solving many problems in the field of environmental monitoring, research and management. This analytical technique is very suitable for the typical environmental samples such as soil, ores, minerals, sediments, water and aerosols. It is a very attractive method for multielement analysis.

The main activities of x-ray fluorescence analysis (XRFA) are as follows;

- Application of XRFA to obtain baseline data on the concentration of toxic elements in environmental objects
- Determination of elemental concentration in soil, plants, water, food, etc to support the sustainable development of agriculture and improve the quality of human life.

In the Atomic Energy Department of the Myanma Scientific and Technological Research Department, the research works concerning Energy Dispersive X-ray fluorescence analysis System were as follows;

- Measurement of Elemental Constituents in Various of Myanmar's Rice (1990- 91)
- Measurement of Elemental Constituents in Various Kinds of Myanmar's Rice (1991-92)

- Measurement of Elemental Constitution in Foodstuffs and Chemicals used in it by XRF Analysis (1993-94)
- Determination of elemental Composition in Industrial Products by X-ray spectrometric Technique (1994-95)
- Determination of Trace Element in Cosmetic By X-Ray Spectrometric Techniques (1994-95)

In the present work, composition of trace elements in various kinds of bean are determined by XRF technique applying Emission-Transmission technique. The performance and results are presented in the report.

2. EXPERIMENTAL

The sample preparation for the XRF analysis is very simple and fast mainly in homogenisation is necessary, but it is a critical step. This step give the main source of error and need to be careful for contamination. The sample of approximately 0.4g are grounded and prepared as pressed pellet with 5-10 tons pressure.

The intermediate thick pellet samples of 2.5 cm diameter and surface density of around 0.08 g/cm ^{2} were prepared by pressing the pulverized and homogenized material at 5 Tons of pressure in the SPECAC 25 mm DIE.

Characteristic X - rays in the sample can be excited by radiation from radioactive source and x-ray tube. When a sufficiently energies x-ray photon interacts with a atom from a sample, several physical events happen. One of the interaction is that, the photon energy transfer its energy to one of the electron of the atom (for example a k shell electron), and then this electron eject from the atom. Excitation radiation of energy E(kev)is incident on an atom of the sample. It is transmitted, scattered, or absorbed. The atom then emits an Auger electron or a characteristic K, L, or M x-ray. Efficiency calibration of a Si (Li) detector can be carried out by measuring intensities of fluorescent x-rays generated by source excitation of pure elements or chemical compounds.

The x-ray fluorescence analysis is characterised by the on linear relationship between the measured intensity of characteristic x-rays and concentration of the respective element in the sample. The non linearity is caused mainly by the self-absorption of the excitation as well as the fluorescent radiation in the sample. Namely, deeper layers of the sample are reached by rather attenuated excitation radiation, while fluorescent x-rays emitted away from the surface have also been absorbed on it's way out of the sample towards the detector.

The composition of each element in the sample can be determined by applying Emission-Transmission technique. This technique is important for environmental samples especially containing low z elements which do not appear in the spectrum. Thus, to correct the absorption in the sample and to measure how much intensities of light element are absorbed in the sample. For this measurement, first we measured the intensities of the sample only, intensities of the sample with pure metal sheet (above the sample) and intensities of pure metal only (the same geometry).

The second factor responsible for the above mentioned non linearity is called enhancement. It is an additional excitation of fluorescent radiation emitted by other elements in the sample.

Energy Dispersive x-ray Fluorescence Analysis System consists of the Si(Li) Canberra Detector Model SL 30175, Serial No. 586408: Cryostat Model 7500: (Detector Area = 30 mm^2, Detector Thickness = 5 mm, Window Thickness = 1.0 mil, Distance form Window = 5 mm) Resolution 170 eV FWHM AT 5.9 KeV; H.V. Power Supply (Canberra Model 3106); Spectroscopy Amplifier (Canberra Model 2020); Canberra Series 35 Plus Multichannel Analyser; IBM/ AT compatible microcomputer- 80386 (TrunkNet) and New Cd-109 annular excitation source (20 mCi).

Calibration of XRF system was carried out by measurement of intensities of fluorescent x-rays generated by excitation of the pure standard metal sheets (foils) Ti, Cr, Fe, Cu, Pb, and Mo and known chemical compounds in thick pellet form $CaCo_3$, As_2O_3 and U_3O_8.

The measurements were carried out under three required conditions, in Emission- Transmission technique, which were sample, sample and target positioned 4 mm above the sample, and target. The time taken for the measurement of each spectrum was 3000 Sec. The spectra of characteristic x-rays produced by exciting sample, sample with target positioned 4 mm above the sample, target (Mo foil) with Cd- 109 radioactive source were analysed.

The characteristic X-ray spectra, produced form the sample as a result of excitation by Cd-109 source are detected by Si (Li) detector and accumulated by Multichannel Analyzer (MAC). The X-ray spectra are transferred to the computer by data transfer programmes included in the QXAS (Quantitative x-ray Analysis System).

Of the QXAS software system the AXIL (the non-linear spectrum evaluation software) pertware) performs the X-ray spectrum analysis, and produces, as the main result, the accurate values of the areas under individual peaks in a spectrum. Indetification of all peaks in the spectrum and their careful analysis were performed. AES under Quantitative Analysis was employed for analysis. In the program the analysis based on Total Matrix absorption was selected and measured intersities of fluorescent k X-rays from target of Mo positioned 4 mm above the sample were inserted to obtain quantitative results.

3. RESULTS AND DISCUSSION

The results of measured samples; peanut, Gram whole, Black Eye Bean, Small Red Bean, Lab Lab Bean, Green Mung Bean, Field Pea, Seasame Seed, Sultani, Maize, Butter Bean, Dolichos Lab Lab, Toor Whole, Small Yellow Bean, Cow pea are given in Annex. Trace elements mainly composed in the samples in the samples were potassium,Calcium, Manganese, Iron, Copper, Zince, Rubidium and Strontium. It can be deduced from the results that the heavy toxic elements such as Arsenic (As), and Mercury (Hg) were not present in the samples measured. The lowest Detection limit (LDL) was calculated from three times the background counts (3 * B.G).

4. CONCLUSIONS

X- ray fluorescence spectrometry is very suitable for the trace element analysis of heavy metals in the environmental samples. Not only the major and minor elements but also the trace elements can be accurately determined by XRF , when correct anc proper sample preparation is used ,the spectrometer is on good condition and the data reduction employed is adequate.

5. ACKNOWLEDGMENT

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6. REFERENCES

1. " QXAS Experiences with Programmes for Quantitative " Programme manual.
2. P.V Espen, K. Janssens, I Swenters, " AXIL X-Ray Analysis software " , Users Manual.
3. " Radioisotope X-Ray Fluorescence Spectrometry, IAEA, Vienna, 1970.
4. C.T. Yap, P. Kump, S.M. Tang, and L. Wijedinghe, "Applied Spectroscopy " , Vol 41, No.1, P 80.
5. A. A. Markowicz and R.E. Van Grieken," Analytical Chemistry Fundamental Reviews " , 1990, 62 P 101 R.

6. C. T. Yap, kump, S.M. Tang and M.G. Bilal, " X-ray Spectrometry " , Vol. 16, 1986.

7. A. Markowicz, A. A. Abdumnabi, " X-ray Spectrometry ", Vol 20, 97-101 (1991).

8. P. Kump, " Quantitative Analysis of Environmental Samples - QAES ", March 1991.

9. " Study of the Effect of Sample loading Heterogeneity in the Energy - Dispersive X- ray Fluorescence Analysis of Specimens of Intermediate Thickness ", Andrzej Markowicz and Ashur A. Abdunnabi, X-Ray Spectrometry, Vol. 20. 97-101 (1991).

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NO	NAME	Element (Concentration mg/kg)							
		K	Ca	Mn	Fe	Cu	Zn	Rb	Sr
1	Peanut	7920.0 ± 1321	1430.00 ± 256.3	14.63± 3.93	106.33±19.41	52.55± 9.45	44.37± 7.82	22.26± 3.85	1.18 ±0.38
2	Gram Whole	9509.6 ± 1541.3	1622.55 ±458	-	154.63±29.72	49.46±9.53	55.09±10.17	18.14± 3.44	4.19 ± 1.22
3	Black Eye Bean	15000 ±4100	2805.00 ± 623	34.6 ± 12	141.75±18.3	45.9 ± 6.29	53.6 ± 6.77	72.95± 8.63	9.07 ± 1.53
4	Small Red Bean	6029.0 ± 608.3	493.80 ±169.7	-	233.91±17.3	84.74±13.53	63.53± 9.07	24.04± 3.28	5.15 ± 0.78
5	Lab Lab Bean	12950 ± 1699	114.90 ±19.2	-	106.13±13.63	78 ± 8.22	34.1 ± 4.36	28.13± 2.67	3.74 ± 0.79
6	Green Mung Bean	11100 ± 814	922.00 ± 157	38.5 ± 9.72	83.90 ± 9.45	92.6 ± 7.40	56.9 ± 4.85	21.7 ± 1.87	<LDL
7	Field Pea	5892.3 ± 614.2	200.60 ±113.6	30.95± 10.1	473.17±36.63	72.5 ± 6.59	58.2 ± 5.17	14.67± 1.62	8.56 ± 1.24
8	Sesame Seed	3753.9 ± 413.0	10065.00±826.5	44.6 ±10.67	162.00±14.2	74.27± 6.55	59.7 ± 5.19	4.55 ± 1.18	142.23±10.33
9	Sultani	6732.8 ± 1281.5	606.20 ±263.7	57.01±19.89	126.01±22.22	95.52±16.35	45.59± 7.78	57.75± 8.04	3.31 ± 1.46
10	Maize	1492.9 ± 246.0	227.00 ± 22.4	25.30±9.55	66.57±8.48	36.9 ± 3.9	36.9 ± 3.9	<LDL	<LDL
11	Butter Bean	7840.8 ± 763.7	294.00 ± 28.5	30.05± 10.3	103.3±11.55	90.15 ± 7.9	40.4 ± 4.23	32.95± 2.92	2.50 ± 1.3
12	Dolichos Lab Lab	14100 ± 1020	751.00 ± 179	30.1 ± 10.5	326 ± 24.1	105 ± 8.21	41.3 ± 4.09	25.1 ± 2.11	3.72 ± 0.94
13	Toor Whole	10800 ± 823	55.2 ± 163	33.3 ± 10.7	62.3 ± 9.29	26.7 ± 3.63	12.4 ± 1.95	4.41 ± 1.31	<LDL
14	Small Yellow Bean	10200 ± 833	<LDL	52.4 ± 13.7	131 ± 14	105 ± 8.99	37.2 ± 4.50	20.00± 2.07	<LDL
15	Cow Pea	9250.0 ± 755	753.00 ± 181	<LDL	103 ± 11.9	84.8 ± 7.65	46.04± 4.80	3.96 ± 1.44	31.7 ± 2.67
16	Flour(smooth)	5430.0 ± 974	1090.00 ± 343	-	125 ± 17.1	86.5 ±8.48	19.00± 4.39	31.1 ± 4.98	<LDL
17	Flour(rough)	4920.0 ± 782	682.00 ± 263	-	136 ± 17	78.5 ±8.29	40.9 ± 5.47	7.39 ± 2.00	<LDL

LDL = Lower Detection Limit
LDL of Ca = 100 mg/kg LDL of Mn = 12.5 mg/kg LDL of Rb = 3.77 mg/kg LDL of Sr = 1.04 mg/kg