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Elemental Analysis of Soil Samples by Ion Beam Analysis (IBA) Technique.

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Abstract

Proton Induced X-ray Emission (PIXE), an ion beam based analytical technique has been used to analyze the essential and toxic elements in soil at ppm levels. These samples were collected from different residential locations of Rajshahi Bangladesh. The interest, very specifically, was used to investigate whether the toxic elements etc., were present in the samples; and if it were so, to see whether the concentration of these show any significant variation with season and the sampling time. 15 soil samples were prepared and analyzed by PIXE technique. Iron content in the samples studied was found remarkably high, among all of the chemical elements in the soil samples while the mean concentration of Chlorine and Chromium were very low. Significantly increased amount of the elements K, Ca, Mn, Zn, Cu, Co, V, and Rb were observed in the samples as well. Concentrations of K ranged from 5926 to 13167 ppm with a mean of 9706 ppm. The mean concentrations of Ca, V, Mn, Co, Zn, Rb and Ti were 68270, 2760, 12236, 31533 and 11640, 14134 and 28000 ppm, respectively. The concentrations of K, Ca, Zn, Cl were in the same order as in IAEA standard soil-7 sample. But the concentrations of Cr, Fe, Co, Rb and Ti were higher than the standard concentration. Quality control and quality assurance in the measurements were ensured by analyzing the standard samples Coal Fly Ash and IAEA soil A-7 during the experiments and also through participation in the inter comparison study with another laboratory. The level of concentrations and sensitivities of all the element presents in the soil samples were measured; and found in toxic level in some samples and Cl, K and V are found to be in a deficient level in some, whereas is found to be present in optimum level. The presence of the elevated amount of the hazardous heavy metals was primarily due to extremely high concentration of these elements in industry effluent discharging to the ground without proper treatment.

Keywords: IBA, PIXE, MDL, X-rays, Elements and Hazardous.

1. Introduction

Revolutionary change of earth has been done to make the earth most comfortable for human lives since the ancient time. Till now men are trying to change their fate with technological development. Developmental activities e.g. industrial, agricultural, transportation, constructional work, etc. cause degradation and drastic changes in every component of environment such as soil, water and air through pollution. In order to recognize and predict hazardous effects of pollutants on biosphere, the monitoring of these environmental components are essential.

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For this purpose, the quantitative characterization of water, soil, air and vegetation is very essential. Elemental analysis is such a process where a sample of some material (e.g., soil, drinking water, bodily fluids, minerals, chemical compounds) is analyzed for its element and sometimes isotopic composition. Man like all other terrestrial animals, depends on food derived from the soil and the natural composition of the soil is, therefore, of vital important to him. Bangladesh is a country which is mainly dependent of agriculture. Hence the quality of soil plays vital role in agricultural products such as vegetables, crops, food items, etc. Excess or lack of mineral elements in soil may cause various disorders in human health and the people may suffer from various diseases in certain region of the country.

Paulo E. Cruvinel *et al.* carried out an experiment to analyze agricultural soil samples for elemental analysis through particle induced X-ray emission (PIXE) method and the content of essential nutrients in soil samples were determined [1]. Y. Oki *et al.* used a combination method of PIXE and activation analysis, for elementary analysis of soil samples [2]. Proton-Induced X-ray Emission (PIXE) technique has been applied for the determination of major, minor and trace constituents of some multi-elemental samples (environmental, and metallurgical) by Antoaneta Ene *et al.* [3]. Soil, Water and Vegetables were analyzed by using PIGE method and reported that vegetables are not a significant source of F uptake of the population [4]. . Soil samples were found to be rich in Na and Mg, but deficient in P, Cl and B. PIGE method was used for elemental analysis of local vegetables [5]. The elements that were found to be present in those samples measurable quantities are B, Na, Mg, Si, P, Cl, K and Sc. No F in any of the vegetables samples. Na was found in toxic in some samples and Si, B, P, Cl, Mg and K were found to be in a deficient level in some, whereas Sc is found to be present in optimum level. The use of the PIXE technique for elemental analysis avoids the traditational acid digestion procedure common in other techniques. The multielemental characteristic of PIXE allows a simultaneous determination of about 20 elements in the sediment samples such as Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Rb, Sr, Zr, Ba and Pb. The techniques developed for sample preparation and PIXE analysis was proven as advantageous and provided very good reproductivity and accuracy. An important way of determining many elements simultaneously on a typical sample is to use ion beam analysis (IBA), since it is multi elemental, sensitive and virtually nondestructive [6]. Among different IBA techniques, the PIXE method in particular, is suitable for the analysis of heavy elements rather than light ones.

Realizing the importance and gravity of the problem the main objective of the present study is to find the toxicity or deficiency of some trace elements in the soil samples of Rajshahi district. Hence the present study may play an important role in public health and may help to ensure the quality of life by assisting the authorities to take appropriate steps to rectify the problem.

2. Materials & Method

The arable soils were collected from different locations of Rajshahi district and labeled. Then the samples were dried in an oven at a temperature of 60^0 C for about 10 hours to make the samples water free. After cooling the samples to the room temperature in a desiccator, the weights were taken. The process of heating, cooling and weighting was repeated until a constant weight is shown by the balance which is a confirmation of zero water content. The dried weighted samples were then ground in a grinder and made into pellets with a pellet maker. These pellets were used as the targets for irradiation with proton beam.

One of the most attractive ion beam analysis (IBA) techniques is PIXE, which uses characteristics X-rays emitted by the samples after irradiation with proton beams. PIXE provides supports for the reliable measurements of toxic elements in the bioenvironmental samples such as air, soil, etc. in parts per million (ppm) levels, which acts as an essential ingredient in critical decision making for safeguarding the public health and improving the quality of the environment [7-9]. One of the striking examples is then detection of high concentration of lead in the air of Dhaka city using PIXE method [10]. PIXE spectrometry is one of the common and widely used analytical techniques with low energy accelerators. Johansson et al. first developed the technique in the early 1970s [11]. Since then the technique is being used as a powerful tool of elemental analysis. This technique is also used routinely by geologists, archaeologists, art conservators and others to help answer the questions of provenience, dating and accuracy. The PIXE method of elemental analysis is based on the measurement of the characteristic X-ray yields of the elements in a sample bombarded with protons. In this technique, X-rays characteristic are detected using the semiconductor detectors. Energy dispersive analysis of the detector signals can reveal the identity of different elements present in the sample and, more importantly, by measuring the charge, i.e. the number of incoming particles, the concentrations of the elements can be accurately measured. PIXE is truly multi-elemental technique and can identify elements from sodium up to the rest of the periodic table. Applications of this technique are wide-ranging and diverse.

In the present work the Proton Induced X-ray Emission (PIXE) method has been used to determine the concentration of low mass elements in soil of different locations of Rajshahi districts.

3. Results

Environmental pollution by different toxic elements has now become a global issue. The knowledge of elemental contents of soil is very important because it is the root sources upon which all living beings are depended for minerals and food either directly or indirectly. PIXE is a reliable tool for analysis of trace elements. Among all elements in the periodic table at least 36 are known to be associated in one way or another with the fabric of life. Realizing the importance of their roles in human health, elemental analysis of soil samples is very important. In the present study 15 soil samples were analyzed by PIXE technique. The studied elements were K, V, Sc, Ti, Cr, Mn, Fe, Cu, Zn, Co, Rb, Na and Cl.

From the diagram it is found that the mean concentration of Iron is very high among all of the chemical elements in the soil samples while the mean concentration of Chlorine and Chromium are very low in the soil samples. 15 concentrations were measured from 15 samples of each elements and average concentration were recorded in Table 1.

The concentration of K in the present soil study in most of the elements is dominant. Thus there is an adequate supply of K from the locating site to their elements. The average K observed in this study is 9706 ppm varying within 5926-13167 ppm. This result lies within the previous experimental values and close to 15000 ppm as mentioned Table. 3. The percentage of error varies between 3.6-10.53% and the MDL range is 486-1235 ppm, thus providing reliability in the K measurement.
The present study shows Ca concentration is 68270 ppm (range 42907- 99124 ppm). Our measured values of concentration 68270 ppm are somewhat greater than the highest value of 24000 ppm as mentioned in the above table. The percentage of error varies between 0.59-0.89 % and the MDL range is 392-940 ppm. In this study, the range of Zn concentration ranges between 5294-21074 ppm with the average of 11640 ppm, which is inconsistent with the previous results as mentioned in Table.3. The percentage of error varies between 15.73-36.45 % and the MDL variation is in between 1074-8178 ppm. The soil samples analyzed in this study were found to contain the average Fe concentration of 957533 ppm within the range of 600624-1431931 ppm, which is approximately twenty times the highest value as enlisted in Table.3. The percentage of error varies between 0.12-0.18% and the MDL varies between 288-1532 ppm. The average Cl content in soil as observed in the present work is 1120 ppm within the range of 201-9416 ppm. Another two studies found that the Cl content in soil are 2521 ppm [15] and 88.5ppm [12] respectively, as shown in Table.4 and our obtained value of Cl concentration also falls within this range. The percentage of error varies between 19.33-28.99 % and the MDL varies between 112-238 ppm.

Rb has been observed to have the average 14134 ppm within the range of 6241-19471 ppm. The percentage of error varies between 14.24-50.98 % and the MDL varies between 743-73070 ppm. It can be observed from Table 4 that a study in U.S. observed the Rb content of 67 ppm [16], which indicates a compatible measurement of Rb in the present study. Ti has been observed to have the average 28000 ppm within the range of 18793- 40305 ppm. The percentage of error varies between 1.16-3.16%. It can be observed from Table.3 that a study in U.S. observed the Ti content of 2900 ppm. V has been observed to have the average 2760 ppm within the range of 1549-4051 ppm. The percentage of error varies between 10.15-16.47%. It can be observed from Table.3 that a study in Brazil observed the V content of 155 ppm.

Cr has been observed to have the average 874 ppm within the range of 655-1055 ppm. The percentage of error varies between 19.43-33.47 % and the MDL varies between 235-610 ppm. It can be observed from Table 3 that a study in Brazil observed the Cr content of 80.7 ppm. The experimental result is more than 11 times greater than the reference value. Co has been observed to have the average 31533 ppm within the range of 20504-42525 ppm. The percentage of error varies between 8.35-11.02 % and the MDL varies between 3364-41543 ppm. It can be observed from Table 3 the experimental result is more than 500 times greater than the reference value. Mn has been observed to have the average 12236 ppm within the range of 7554-17095 ppm. The percentage of error varies between 6.71-11.1 % and the MDL varies between 509-1460 ppm. It can be observed from Table 3 that the experimental result is more than 22 times greater than the reference value.

4. Discussion

Plants and the animals are dependent on the soil for their supply of nitrogen and mineral elements; their internal biochemistry and associated composition is therefore a reflection of the composition of the soil. The imbalance in the supply of mineral elements may cause many diseases. The deficiency of Zn in the soil has already established as a problem in Bangladesh. The present work was conducted to measure the concentration of some essential low atomic mass elements in soil samples. The elemental contents of soil around the Rajshahi Nuclear Medicine Centre have been measured. These elements are essential for both man and animal's health. The samples were bombarded by a proton beam of energy 2.5 MeV from 3 MeV Van de Graff Accelerator.

Large fluctuations in errors ~ 1 to 50% were observed in the measured values of concentrations of the elements. There may be several reasons for these fluctuations in the errors. All the present experiments were done at fixed proton energy of 2.5 MeV, which was perhaps not high enough for exciting all the elements present in the samples with sufficient intensity for concentration determination below percentage level. Proton beam of higher energy (> 2.5 MeV) might be required to excite all the elements present in the target samples. Moreover, if the intensity of the emitted X-ray is weak, then the experiments have to be done over a long period of time to obtain statistically significant number of counts in the X-ray peaks. For very weak peaks, the time required for accumulation of statistically significant counts might at times become absurdly long and expensive. Large errors were observed in the case of Rb where the X-ray peaks were poor.. Important elements which include S, Cl, Ca, K, Fe, Ni, Cu, Zn, Br and Rb were detected using PIXE technique. The roles played by some of these elements in reducing liver, heart and cancer diseases are enormous.

5. Conclusion

The present study reveals that Iron content in the soil samples of Rajshahi studied was found remarkably high, among all of the chemical elements while the mean concentration of Chlorine and Chromium were very low. The soil and sediment of this area contain significantly high level of trace elements like Cr, Fe, Co, Rb and Ti. The results of the present study indicate that different industries of this region and using more chemicals in agricultural land arc responsible for the significant increase of the heavy metals in the soil samples in Rajshahi area. Further study on the industrial contaminants and related aspects of environment pollution is in progress.

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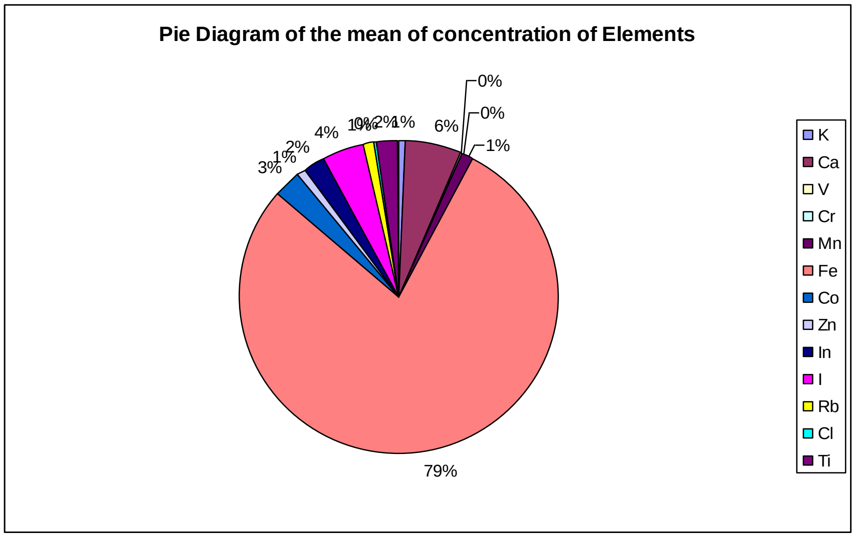

Fig. 1: Pie diagram of the mean of concentration of elements.

Table.1: The mean concentration and comparison between STDEV and relevant error.

Elements	Mean of con (ppm)	Range of con (ppm)	STDEV	Range of error (%)	Range of MDL (ppm)
K	9706	5926-13167	2363	3.6-10.53	486-1235
Ca	68270	42907-99124	22268	.59-.89	392-940
V	2760	1549-4051	870	10.15-16.47	404-982
Cr	874	655-1055	131	19.43-33.47	235-610
Mn	12236	7554-17095	3715	6.71-11.17	509-1460
Fe	957533	600624-1431931	364036	.12-.18	288-1532
Co	31533	20504-42525	9058	8.35-11.02	3364-41543
Zn	11640	5294-21074	5747	15.73-36.45	1074-8178
Rb	14134	6241-19471	4911	14.24-50.98	743-73070
Cl	1120	201-9416	2306	19.33-28.99	112-238
Ti	28000	18793-40305	9513	1.16-3.16	206-851

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Table.2: Comparison of elemental constituents of soil with IAEA Soil-7.

Element	Certified value (ppm)	Measured value Range (ppm)	Mean of con (ppm)
K	12100	5926-13167	9706
Ca	163000	42907-99124	68270
Cr	60	655-1055	874
Zn	25700	5294-21074	11640
Fe	8.9	600624-1431931	957533
Co	104	20504-42525	31533
Rb	51	6241-19471	14134
Ti	3000	18793-40305	28000
Cl	2306	201-9416	1120

Table.3: Comparison of elemental constituents/abundances of soil samples in ppm of different countries with that of the present work.

Elements	Cl	K	Ca	Ti	V	Cr	Mn	Fe	Ni	Cu	Zn	Rb	Co	Reference study
Concentration in ppm	88.5	2976	371	4342	155	80.7	129	32274	21.3	31.3	56.9	58.1	-	[12]
	-	37000	12000	-	-	-	70	46000	-	52	1300	-	-	[13]
	-	-	-	-	-	-	-	18000	2.5	30	62	-	63	[14]
	2521	884	16220	-	-	-	219	21040	-	-	-	-	-	[15]
		15000	24000	2900	80	54	550	26000	19	25	60	67	9.1	[16]
	1120	9706	68270	28000	2760	874	12236	957533	-	-	11640	14134	315 33	Present study