PIXE Analysis of Some Environmental Samples from Selected Saline Region of Bangladesh

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***Abstract*—In the present study Particle Induced X-ray Emission (PIXE) method has been used to identify the elements present in some environmental samples (soil and vegetable) in order to show the possible influence of soil in the metal absorption by the vegetables grown in saline region of Bangladesh. PIXE can detect a high number of elements with a high sensitivity (ppm range) in a simple and fast way. The samples were prepared for irradiation following conventional methodology and irradiated with 2.5 MeV collimated protons from the 3 MV tandem accelerator of Bangladesh Atomic Energy Commission. The X-rays emitted during irradiation from the samples were measured by HPGe detector and the acquired X-ray spectrum was analyzed by using the software GUPIXWIN. The obtained results were compared with two standard values IAEA-359 (cabbage) and IAEA Soil-7. Concentrations of some heavy metals (Cr, Co and Ni) were found above the potentially risky levels in soils; however, they accumulated in plants to a lesser amount, probably because of the physical and chemical properties of soils that prevent their conduction to vegetables. As, Cd and Pb were not found in any one of the soil and vegetable samples in the present study.**

***Index Terms*—Elemental analysis, GUPIXWIN software, PIXE, salinity, soil and vegetables.**

I. Introduction

Soil contamination by heavy metals has become one of the greatest world environmental problems today; Bangladesh is no exception to it. In many areas contaminated soils are a threat to the groundwater quality, and thereby to the whole biosphere [1]. The main sources of soil contamination are agriculture, industry and urban spreads. In the case of agriculture in technologically advanced countries, industrialization has brought major change to agricultural practices, including the use of chemical fertilizers, animal wastes and subsoil waters. Although these materials are not intrinsically pollutants, inappropriate use can turn them into such [2]. Salinity may also contaminate the soil since it causes unfavorable environment and hydrological situation that restrict the normal crop production throughout the year [3].

Bangladesh is a deltaic country with total area of 147,570 km^2. Coastal area of Bangladesh covers about 20% of the country and over 30% of the net cultivable area. Agricultural land use in these regions is very poor because of salinity which is roughly 50% of the country's average [4]. The soils of coastal areas become saline as it comes in contact with the sea water and continues to be inundated during high tides and ingress of sea water through creeks. It affects crops at the critical stages of growth, which reduces yield and in severe cases total yield is lost. Saline soils are mainly found in Khulna, Barisal, Patuakhali, Noakhali and Chittagong districts of the country. Most of the soils of these regions are moderate to strongly alkaline (pH ranges from 6.0–8.4), organic matter (OM) content ranges from 1.0% – 1.5%, the cation exchange capacity (CEC) ranges from 9.4 – 40.6 m.e.%, the total nitrogen (N) content is very poor, mostly around 0.1% and available phosphorus (P) ranges from 15 – 25 ppm. Widespread Zn and Cu deficiencies have been observed in the coastal regions [5].

The main source of metals in human diet is fruits and vegetables (about 73%). Vegetables can absorb metals from soil and from water used during cultivation and contained in the airborne particles deposited on the leaves. Agricultural techniques especially the use of agricultural products such as fertilizers and pesticides are also an important source of metals for vegetables. Some metals such as K, Ca, Mg, Fe etc. are important for human health; however, others can cause serious damage when ingested above a threshold as Cr, Cd, As, Pb etc. Therefore, in order to understand the effect of metals on human health, it is important to know the contents and concentration of metals in food stuffs as well as in soils where they cultivate. Such types of study are done frequently almost all countries of the world to ensure the quality of health and life of their citizens. But still information of this topic is rare, especially when dealing with developing countries like Bangladesh.

The aim of this study was to identify the elements presents in soils and vegetables collected from different agricultural fields of Dacope, Khulna, a saline region of Bangladesh. In addition to this, the possible impact of soil in the metal involvement by the vegetables was also observed. The study area is located in southwestern part of the country where sometimes saline waterlogged happened for long time due to tropical storm like Aila, Sidr etc. The agro-chemical characteristics of soils in the study area are listed in Table I and Table II. In this work PIXE was applied for determination of elemental mass concentration. Atomic absorption spectrometry (AAS), atomic emission spectrometry (AES) or inductively coupled plasma mass spectrometry (ICP-MS) are also used for elemental analysis of soil [6] but sample preparation steps of these methods are laborious and involve risk of contamination [7]. On the other hand for the

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monitoring of metals in contaminated soils PIXE seem to have several advantages than that of other methods. The outcome of the study will help to understand the environmental pollution by soil salinity and its effect on human health of the locality mentioned above.

TABLE I: AGRO-CHEMICAL CHARACTERISTICS OF SOILS OF THE STUDY AREA

District	pH	OM %	Total N %	CEC m.e. %	P ppm
Khulna	6.2-7.9	0.1-0.3	0.1-0.3	18.2-40.6	8-36

Sources: Annual reports of BARI, BRRI, BWDB and DU of the coordinated research project on production potentials of the coastal saline soils of Bangladesh (1987-1989).

II. MATERIALS AND METHODS

A. Sample Collection and Preparation

A total number of 20 soil and vegetable samples were collected from different agricultural fields of Dacope, Khulna, one of the saline affected areas of Bangladesh. The vegetables were cultivated in the same area of soil samples. All the collected samples were oven dried at 60^0C, grounded by agate mortar and then pressed using hydraulic press to make pellet of 13 mm diameter and about 1 mm thickness.

B. Irradiation and Data Acquisition

The present experiment was carried out at the Tandem Accelerator Facilities Division, INST, AERE, Bangladesh Atomic Energy Commission using 3 MV tandem accelerator. A collimated proton beam of energy 2.5 MeV with a diameter of 2 mm made to fall on the sample. The integrated charge irradiation on the sample was kept in the range of 5 to 10 micro coulombs and the irradiation was performed for 5 to 10 minute to get sufficient X-ray counts.

During irradiation, the beam current was in the range of 10 to 20 nA. Individual irradiation was performed for each sample.

The X-rays emitted in the irradiation were counted using solid state detector (HPGe) associated with the necessary electronics. The detector was mounted at 45^0 to the beam direction. A thin Mylar foil was placed between detector and sample for the elimination of unwanted low energy X-rays to reduce noise in counting system and also to improve the spectrum quality. Accuracy and precision of the methods were assured by analyzing certified reference materials in the same experimental conditions.

C. Data Analysis

The PIXE spectral data were analyzed by using GUPIXWIN software [8] package which provides non-linear fitting of the spectra and converts raw spectral data into elemental concentrations. The unknown concentration for a specific element in the analyzed sample is calculated by using the following equation [9]:

$$C_Z = \frac{Y}{Y_t Q \varepsilon T H} \qquad (1)$$

where Y_t &Y are theoretical and experimental intensity of X-ray respectively, Q is the measured proton beam charge, ε is the efficiency of the detector, T is the transmission through any filters or absorbers between the target and the detector and H is the hybrid parameter equivalent to the product of the geometric solid angle of the X-ray detector and any systematic calibration factor present in the PIXE set-up. H values as a function of X-ray energies can be measured experimentally using wide range of pure single-element standards.

III. RESULTS AND DISCUSSIONS

A. Elements in Soil Samples

The relative mass concentrations of ten soil samples were determined by PIXE technique. The results are presented in Fig. 1. Standard values (IAEA Soil-7) are included for comparison. A total number of 21 elements were identified among them K, Ca, Ti, Cr, Mn, Fe, Co and Ni were observed in all samples. Some of them (K, Ti, Mn and Fe) are well agreed with the standard values while Ca found in less amount. On the other hand, heavy elements such as Cr (185.8-298.6 ppm), Co (592.2-882.8 ppm) and Ni (98.03-146.9 ppm) are found with high concentrations considered as potentially hazardous [10], making a more repeated monitoring needed for a future remediation. The values of Cr are very close to second guideline value (250 ppm) [11]. The general level of the pollution is often characterized by a contamination factor defined as a ratio of the element concentration in the samples from the polluted region to the background concentration of the same element. The ratios of concentrations (average) of heavy metals obtained in the investigated soil to IAEA Soil-7 were estimated: Cr 3.12; Mn 0.77; Fe 1.49; Co 85.17; Ni 4.69; Cu 1.45 and Zn 0.56. The highest contamination of Co was found. The contamination factors for the elements Cr and Ni are significantly large than that of Fe. The pH is one of the most important parameters governing different reactions and processes in soils and also controls the behavior and bioavailability of the heavy metals in soils. For instance the motilities of different toxic elements in soils are strongly dependent on pH. For all cationic metals, adsorption increased with pH. In addition to bioavailable or soluble metals in soil, direct ingestion of the metals from livestock and intake in cattle will provide an exposure risk to human health. The toxicity of elements in soils depends also on the oxidation state and species formed by the element. Such studies were not performed in this work.

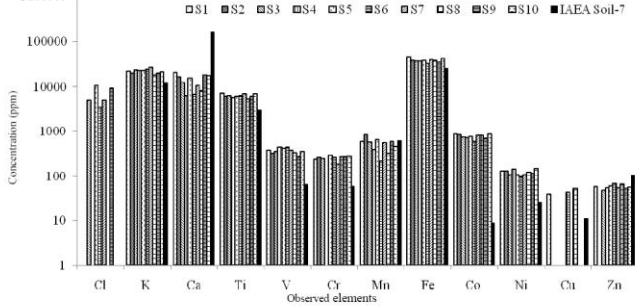

Fig. 1. Concentrations of different elements identified in soil samples.

V observed in soils could be from the ore or from anthropogenic source (burning of crude oil) [12]. Cl was found in 5 samples (S3, S5, S6, S7 and S9) with high concentrations but not present in standard value. This is due to the soils of the study area become saline as it comes in contact with the sea water and continues to be inundated during high tides and ingress of sea water through creeks and sometime saline waterlogged happened for long time. Zn was found in 9 samples with concentration less than IAEA value. Cu is found only in 3 samples with high levels than standard value but below potentially harmful, indicating that the soils are Cu deficient. For the Cu content there might be an influence of the larger peak of Zn.

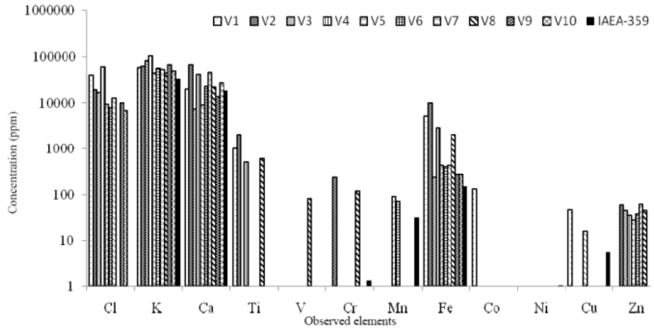

Fig. 2. Concentrations of different elements identified in vegetable samples.

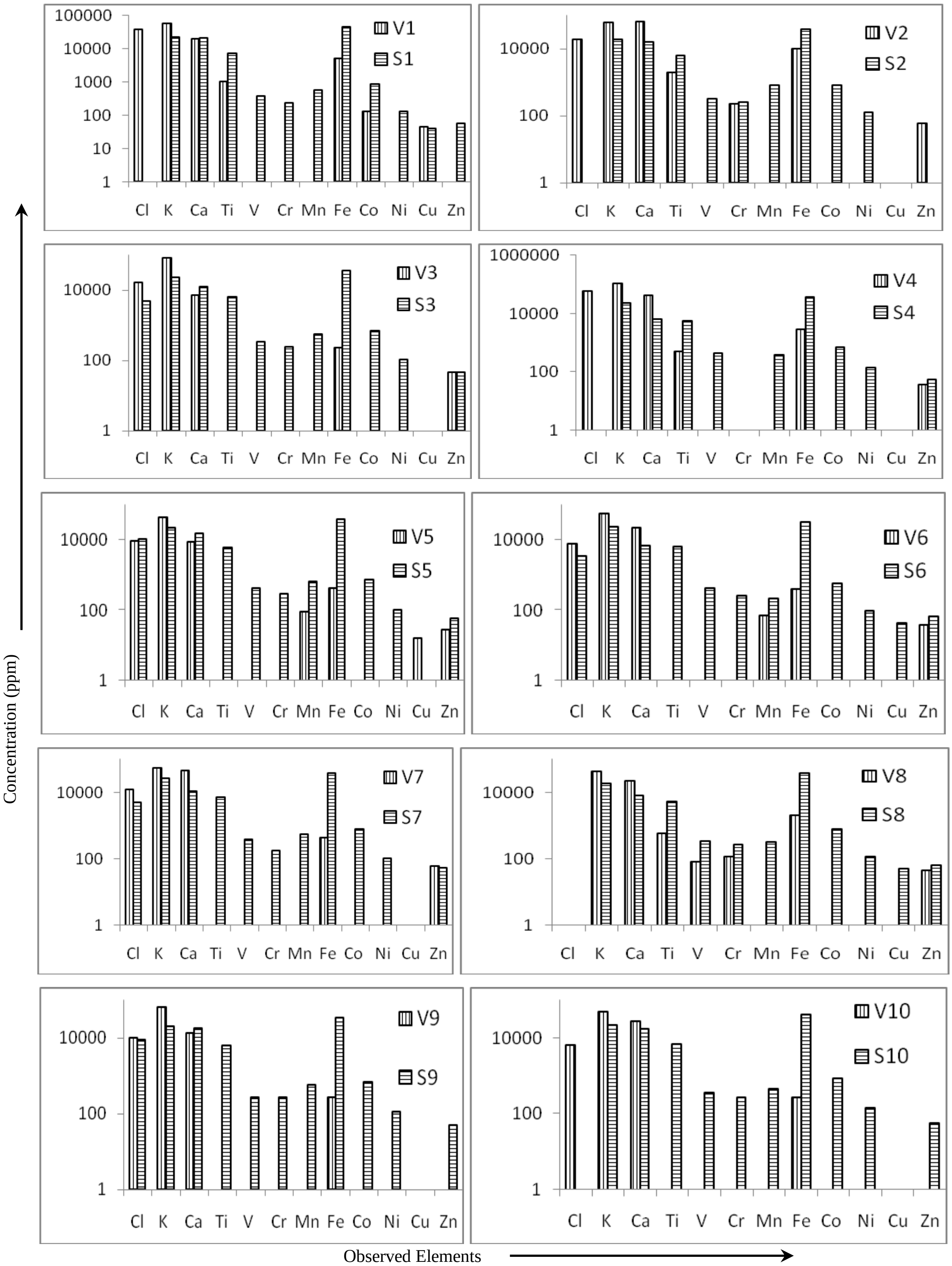

Fig. 3. Concentrations of different elements identified in soil and in the vegetable samples.

TABLE II: CONCENTRATIONS (MG/KG) AND METAL/SOIL RATIOS IN DIFFERENT VEGETABLES

Leafy vegetable	Part	Cl	K	Ca	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
V1- Water spinach	Soil	ND	22247	20990	7261	386.7	240	591.1	45820	860.4	130.1	40.25	57.9
	Leaf	39470	57889	20016	1042	ND	ND	ND	5204	132.5	ND	46.66	ND
	Leaf/Soil (%)	**IV**	**260.2**	**95.4**	**14.4**	**0**	**0**	**0**	**11.4**	**15.4**	**0**	**115.9**	**0**
V2- Red Spinach	Soil	ND	20123	16506	6289	322.6	259.2	849.8	38554	842.9	128.4	ND	ND
	Leaf	19396	63557	68365	2048	ND	238.8	ND	10171	ND	ND	ND	59.87
	Leaf/Soil (%)	**IV**	**315.8**	**414.2**	**32.6**	**0**	**92.1**	**0**	**26.4**	**0**	**0**	**IV**	**IV**
Fruit vegetable													
V3- Lady's finger	Soil	5224	26694	10741	7107	383.3	185.8	553.8	39353	826.2	106.4	ND	54.96
	Fruit	12647	53730	46473	ND	ND	ND	ND	433.1	ND	ND	ND	62.03
	Fruit/Soil (%)	**242.1**	**201.3**	**432.7**	**0**	**0**	**0**	**0**	**1.1**	**0**	**0**	**IV**	**112.9**
V4- Snake Gourd	Soil	ND	18406	8201	5423	338.7	269.7	325.7	37922	823.3	120.2	52.75	66.9
	Fruit	ND	44063	22316	609.4	80.93	122.1	ND	2067	ND	ND	ND	46.3
	Fruit/Soil (%)	**IV**	**239.4**	**272.1**	**11.2**	**23.9**	**45.3**	**0**	**5.5**	**0**	**0**	**0**	**69.1**
V5- Eggplant	Soil	9270	20317	18149	6274	270.8	272.5	586.1	34568	693.5	115.1	0	52.62
	Fruit	10209	66346	13943	ND	ND	ND	ND	274.9	0	0	0	0
	Fruit/Soil (%)	**110.1**	**326.6**	**76.8**	**0**	**0**	**0**	**0**	**0.8**	**0**	**0**	**IV**	**0**
V6- Papaya	Soil	ND	21735	17634	7102	356.7	277.8	467.3	41408	882.8	146.9	0	56.27
	Fruit	6757	48601	27266	ND	ND	ND	ND	274.1	ND	ND	ND	ND
	Fruit/Soil (%)	**IV**	**223.6**	**154.6**	**0**	**0**	**0**	**0**	**0.7**	**0**	**0**	**IV**	**0**
Root vegetable													
V7- Potato	Soil	5090	23636	12523	6515	355.3	244.4	585	36536	734.9	108.3	0	48.1
	Root	17097	82848	7383	ND	ND	ND	ND	237.2	ND	ND	ND	46.21
	Root/Soil (%)	**335.9**	**350.5**	**58.9**	**0**	**0**	**0**	**0**	**0.65**	**0**	**0**	**IV**	**96.1**
V8- Radish	Soil	ND	22865	6349	5757	441.7	ND	393.9	36742	728.1	141.1	ND	55.49
	Root	60251	105964	41557	516.5	ND	ND	ND	2894	ND	ND	ND	36.2
	Root/Soil (%)	**IV**	**463.4**	**654.5**	**8.9**	**0**	**IV**	**0**	**7.9**	**0**	**0**	**IV**	**65.2**
V9- Red turnip	Soil	10729	22935	15801	6323	423	298.6	652.9	38387	759.6	105.7	ND	60.3
	Root	9335	45076	8994	ND	ND	ND	90.96	431.5	ND	ND	16.04	28.53
	Root/Soil (%)	**87.0**	**196.5**	**56.9**	**0**	**0**	**0**	**13.9**	**1.1**	**0**	**0**	**IV**	**47.3**
V10- Root turnip	Soil	3560	24453	6873	6419	442.7	264.4	215.7	32167	592.2	98.03	43.36	69.54
	Root	8013	57521	23260	ND	ND	ND	70.45	399.7	ND	ND	ND	38.4
	Root/Soil (%)	**225.1**	**235.2**	**338.4**	**0**	**0**	**0**	**32.6**	**1.2**	**0**	**0**	**0**	**55.2**

ND = not detected, IV = incalculable value

The interesting is the Cr/Fe ratios in all soil samples are about 0.007, which indicates that the mobilization of Cr is dependent on Fe-content in soil. Although Cr is an essential element for humans, the hexavalent form of Cr is toxic. The wide spread of Cr in the environment poses a serious threat to human and animal welfare [13]. The toxicity of Cr, however, is a function of oxidation state. Chromium exists in the environment in two stable oxidation states, namely Cr^{3+} and Cr^{6+}. Cr^{6+}exists as chromate (CrO^{4-}) is easily soluble in soils and ground water, and trends to be mobile in the environment. On the other hand, Cr^{3+} forms complex with soil minerals, which are insoluble [14]. Within soils, Fe^{2+} and sulfide (S^{2-}) control the redox status of chromium [15]. In the homogeneous system, Fe^{2+} serves as a catalyst for the reaction, i.e., Cr^{6+} is reduced by Fe^{2+} to form Fe^{3+}, which is then reduced by sulfide to regenerate Fe^{2+}, enhancing the overall rate of Cr^{6+} reduction. The ultimate way of Cr^{6+} reduction is of considerable pragmatic importance because it dictates the reactivity of reduced Cr^{3+} in the environment. The solubility of Cr^{3+} is proportional to the ratio of Cr^{3+} to Fe^{3+}, with increased quantities of Fe^{3+} stabilizing the solid [14]. Mechanisms under which Cr^{3+} could be remobilized are dependent upon the solid phase speciation and co-association of Cr with other soil constituents.

Nickel does not form insoluble precipitates in unpolluted soils and retention for Ni is, therefore, exclusively through absorption mechanisms. Nickel will absorb to clays, iron, manganese oxides and organic matter and is thus removed from the soil solution. The formation of complexes of Ni with both inorganic and organic ligands will increase Ni mobility in soils. As and Pb are a matter of serious concern because of they are carcinogenic, toxic at very low concentrations and bioaccumulative in organisms. Automobile emissions are considerable source of heavy metals, especially Pb in the soil. In the present study As and Pb were not found in any samples signifies that the soils are not contaminated by these metal.

B. *Elements in Vegetable Samples*

The selected vegetables were cultivated in the same area of soil samples. The vegetable samples were also analyzed by PIXE technique. The relative mass concentrations identified in vegetable samples are shown in Fig. 2. Wide ranges of elemental contents were observed among the studied specimens. Most of the elements in vegetables are found in less concentration than soil indicating their lesser transmission from soil to plant. K and Ca are found the most abundant detected elements (major) in the samples. K is crucial to heart function [16] and Ca is an essential element for maintaining healthy bones and teeth. The Fe content is also fairly good in the entire samples. Fe deficiency can causes anemia. Cl was identified almost in all vegetables samples; this could be due to saline water used for irrigation and the vegetables are grown in saline soil. NaCl and $Na(NaCl)_n$ characterize the marine salt. Thus NaCl from marine aerosol was deposited on leaves and absorbed by vegetables. V observed only in V8 could be from the ore or from anthropogenic source (burning of crude oil) [12].

Heavy metals such as Cr, Mn, Co, Cu and Zn were found in some different samples. Cr was detected in V2 and V8 at high amount 238.8 ppm and 122.1 ppm respectively that exceed the toxic level (5-30 ppm) [10]. Co was found only in V1 with concentration 132.5 ppm which also more than toxic level (15-50 ppm) [10]. These required some attention. Ni and Pb were not found in any vegetable samples under study. Mn and Zn contents are found in some samples with good amount but below toxic levels [10]. Ga, Ge, As and Br were not identified in vegetable samples indicating that any agro chemical product had not been used. Vegetables are one of the key sources of mineral elements. Excess or lack of mineral elements in vegetables may cause serious disorder in human health and people may suffer from various diseases. The result shows that most of the investigated vegetables contain elements of vital importance in man's metabolism and that are needed for growth and developments, prevention and healing of diseases.

C. *Elements Uptake from Soil to Vegetable*

When growing edible vegetables or crops in contaminated soils, it is important to know the extent of accumulation in plants and their different parts. In order to study the metal uptake by vegetables from the study area, we selected species which are most frequently grown and commonly consumed by population in the region. The elemental mass concentrations of elements identified in soil and vegetable are shown in Fig. 3. The analysis of the vegetable and soil samples show that the K, Ca and Fe present in all soils were absorbed by all the vegetables. The Cl present in soil S3, S5, S6, S7 and S9 were also absorbed by the vegetables. Additional Cl was observed in vegetables V1, V2, V4 and V10 were not present in the soil. Heavy metals Cr, Co and Ni present in 6 soils (S3, S5, S6, S7, S9 and S10) were not absorbed by the vegetables. Mn was present in all soils but only absorbed by the vegetables V5 and V6. The elements not identified in vegetables could not be biologically available in soils or they could be absorbed in very low concentration (less than 1 ng/g). On the other hand the elements not identified in soils but present in vegetables could be absorbed from water or fertilizer or pesticides used during cultivation or contained in the airborne particles deposited on the leaves [12].

Table II represents the uptake of metals from soil to vegetables. For low Z elements the uptake is very high (Cl from 87.0-335.9%, K from 196.5-463.4%, Ca from 76.8-654.5%) than heavy metals, which specifying their easy mobilization. For Fe, the uptake variation is small (0.65-26.4%). Although Cr, Co and Ni are found in high concentrations in soils but their uptake by vegetables is very low. Cr uptake was found only in two vegetable samples, V2 and V4, but with great amount (92.1% and 45.3% respectively). Co was uptake only by V1 with 11.4% whereas Ni was not uptake by any vegetables in the present study. Zn is readily absorbed showing the highest (112.9%) uptake than all heavy metals ranging from 47.3-112.9%.

IV. CONCLUSION

In the last few years, nuclear techniques have made an important contribution in the field of elemental analysis of environmental samples. Elemental contents in soils and vegetables are an important characteristic in environmental monitoring essential for human health. In this study, the application of PIXE technique allows the identification of metals present in soil and in the vegetables collected from selected saline region of Bangladesh. Vegetables can absorb metals from different sources, but this absorption depends on the metal chemical form. The results from the study show that low Z elements are easily accumulate to vegetables with high amount. Cl was observed in almost all vegetables at significant amount accrued from the soil as well as from irrigation water and marine aerosol deposited on leaves, representing the samples were collected from sea shore area. Some heavy elements such as Cr, Co and Ni are present at levels considered potentially hazardous in soils. Among them Cr and Co are transfer to some vegetables at levels that exceed those considered as toxic. This needed further examined because of long term effect of these metals on human health. Finally this methodology can be considered convenient for identifying the possible sources of metals in environmental soil and vegetable samples.

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