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HEAVY METAL CONTAMINATION IN AGRICULTURAL SOIL OF SOUTH-WESTERN SEASHORE AREA IN BANGLADESH

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ABSTRACT

The investigation of metallic elements in soils collected from different agricultural fields of two regions in Khulna (Dumuria and Dacope), a south-western saline zone of Bangladesh is presented. Particle induced x-ray emission (PIXE) technique has been used to determine the concentration of heavy metals (Cr, Mn, Fe, Ni, Cu and Zn) with the 3MV Tandem Accelerator facility of Bangladesh Atomic Energy Commission (BAEC). Cr and Ni were found at concentrations above potential hazardous levels (PHL) and maximum allowable concentration (MAC) that were also confirmed by their elevated enrichment factor, contamination factor and geoaccumulation index values suggesting contamination of the soils by these metals. The excess of these metals may reduce germination percentage thus decrease crop production which is great problem in saline area of Bangladesh. The pollution load index (PLI) value of Dumuria goes above 1, indicating the deterioration of the site due to metal contamination. Analyzing the data using t-test shows that, among the investigated heavy metals only the Mn concentration level displays significant difference in the two study areas with $p\text{-value} < 0.05$.

Keywords: PIXE, soil, heavy metal, enrichment factor, contamination factor, geoaccumulation index, pollution load index.

1. INTRODUCTION

Soil is important as it roles as a medium for plant growth where it can recycle the nutrient and assets required by plant. Soils are the major sinks for heavy metals released into the environment

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by both natural processes such as weathering of minerals and anthropogenic activities related to industry, agriculture, burning of fossil fuels, vehicular emission, mining and metallurgical processes and their waste disposal. Most metals do not undergo microbial or chemical degradation, and their total concentration in soils persists for a long time after their introduction (Wuana *et al.* 2011) until taken up by plants and become part of food chain. Therefore deposition and accumulation of heavy metal in the environment is of increasing interest because of their persistence, bioaccumulation, toxicity to plants, animals and human health and have negative consequences on the environment (Abrahams 2002; Schroeder *et al.* 2004; Mielke *et al.* 2005; Selinus *et al.* 2005). The accumulation of heavy metal in the soil crop system has been considered as one pathway to human exposed to the heavy metal. It is therefore becomes a great concern because above certain level of concentration, heavy metals have adverse effect on human health which causes many types of diseases.

Bangladesh is likely to be one of the most vulnerable countries in the world to salinity problem. The coastal region of Bangladesh covers about 30% of the cultivable lands of the total area of country which is 147,570 km^2 (Karim *et al.* 1990). Agricultural land use in coastal areas is very poor, which is roughly 50% of the country's average (Petersen *et al.* 2001). The cultivable areas in coastal districts are affected with varying degrees of soil and water salinity. Salinity causes unfavorable environmental and hydrological situation that restrict normal crop production throughout the year. The coastal and offshore area of Bangladesh includes tidal, estuaries and river floodplains in the south along the Bay of Bengal. The coastal areas of Bangladesh 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. The factors which contribute significantly to the development of saline soils include, tidal flooding during wet season (June to October), direct inundation by saline or brackish water and upward or lateral movement of saline ground water during dry season (November to May), sometimes saline waterlogged happened for long time due to tropical storm like Aila, Sidr etc. It affects crops depending on degree of salinity at the critical stages of growth, which reduces yield and in severe cases total yield is lost. Soil reaction values (pH) in coastal regions range from 6.0-8.4 (Rasel *et al.* 2013). The organic matter content of the soils is also pretty low (1.0-1.5%). Nutrient deficiencies of N and P are quite dominant in saline soils. Micronutrients, such as Cu and Zn are widespread. Salinity problem received very little attention in the past. It has become imperative to explore the possibilities of increasing potential of these (saline) lands for increased production of crops. The saline soils are mainly found in Khulna, Barisal, Patuakhali, Noakhali and Chittagong districts of the coastal and offshore lands of the country. The main goal of this research is to achieve the following specific objectives: to observe the heavy elements and their concentration in cultivated soils from two saline zones of Khulna and also to discuss the effect of detected metals on crop production. Particle induced x-ray emission (PIXE) technique has been proved to be a successful method

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(Cruvinel *et al.* 1993; Cruvinel *et al.* 1993; Cruvinel *et al.* 1994; Cruvinel *et al.* 1996) for the observing of different elements in soil since it is possible to detect a high number of elements (heavier than Na) with a high sensitivity in a simple and fast way. In the PIXE analysis (Johansson *et al.* 1970), the number of x-ray photons of a given elements provide information on its concentration. In addition, PIXE possesses a number of advantages over some other analytical techniques, namely, its rapid and multi-elemental determination, non-destructive capabilities, small sample size requirement, very low detection limits up to parts per million (ppm) levels.

2. MATERIALS AND METHODS

2.1 Sample collection and preparation: Twenty top soil (0-15cm from the surface) samples have been collected from different agricultural fields of Dumuria (22°48.5' N, 89°25.5' E) and Dacope (22°32' N, 89°30' E) in Khulna. The samples were placed in individual plastic bag, labeled and then bought to the laboratory for analysis. All the samples were oven dried at 80ºC, grounded by agate mortar and then pressed at a pressure of 5 tons per cm^2 using hydraulic press to make a pellet (~ 200 mg weight) of about 13 mm diameter and 1 mm thickness. Several pellets of Standard Reference Material (SRM 2586) provided by the National Institute of Standards and Technology (NIST) (Standard Reference Material 2586, 2008) were prepared by the same method. The prepared samples were thick enough to stop the incident proton beam during the irradiation.

2.2 Irradiation and data acquisition: The prepared samples were irradiated with a proton beam of energy 2.5 MeV generated by 3 MV Tandem Accelerator, Atomic Energy Research Establishment, Dhaka, Bangladesh. A sketch of the experiment chamber is shown in figure 1. The measurements were carried out with a beam spot of 2 mm in diameter and a low beam current of 10-20 nA and the irradiation was performed for 5-10 minutes to get sufficient x-ray counts. An ORTEC high purity germanium (HPGe) detector Model IGLET-X-06135, with a front window of beryllium thickness 25 µm, with full width half maximum (FWHM) 180 eV at 5.9 keV, with the associated pulse processing electronics, and a MCA card interfaced to a PC were used for x-ray data acquisition. The targets were kept at normal to the beam direction and the detector was positioned at 45º to the beam direction. A thin Mylar foil of 150 µm thick was placed between the 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. A – 60V was applied both at the entrance of the chamber and at the entry of the Faraday cup and +60V at the sample wheel and Faraday cup to suppress the secondary electrons. Accuracy and precision of the methods were assured by analyzing certified reference materials in the same experimental conditions.

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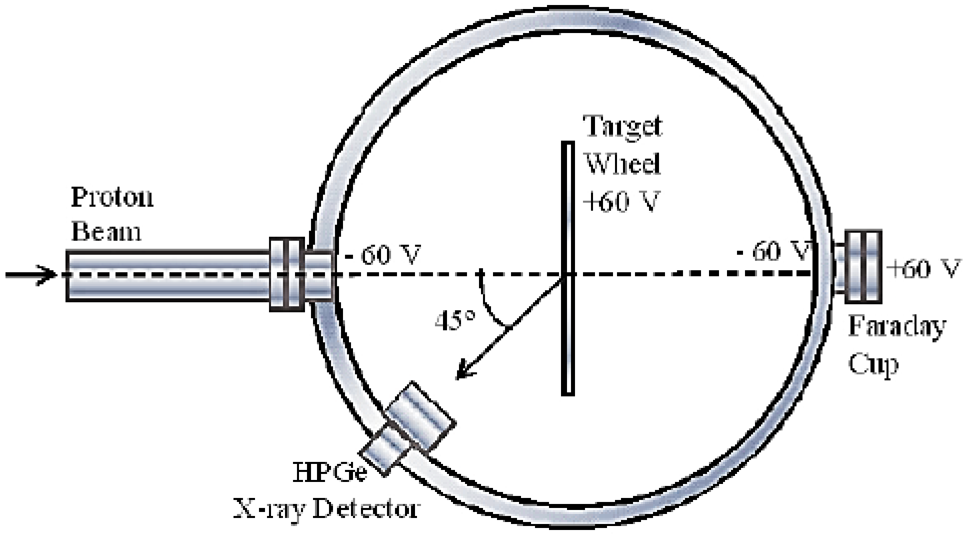

Fig.1: A schematic diagram of experimental chamber for PIXE analysis.

2.3 Data analysis: In the present work, the software GUPIXWIN (Maxwell *et al.* 1989) package has been used to analyze the spectra where all the necessary parameters such as beam type, beam energy, set up geometry, solid angle, beam dose, target details, etc. were used to obtain concentration of the heavy metals in the investigated samples. GUPIXWIN is a program for non-linear least square fitting of PIXE spectra. A calibration factor known as H-parameter (which is the solid angle plus the effect of all other factors which are dependent on the atomic number (Z) of the element or characteristics x-ray energy) was used to obtain accurate value of the concentration of the elements present in the sample. In this work the energy dependent fitted curve of H-parameter, established by comparing the measured values for thick soil standards to the certified reference value, was used. In the X-ray spectrums of Cr, Mn, Fe, Ni, Cu and Zn were identified by their well-defined characteristic peaks at energies of CrK_α: 5.412, MnK_α: 5.895, FeK_α: 6.399, NiK_β: 8.266, CuK_β: 8.905 and ZnK_β: 9.571 keV respectively. The overall data obtained were interpreted using appropriate descriptive and statistical techniques such as charts, enrichment factor (EF), contamination factor (CF), geoaccumulation index (I_{geo}), pollution load index (PLI) and T-test.

2.3.1 Enrichment factor (EF): The behavior of a given element in soil (i.e., the determination of its accumulation or leaching) may be established by comparing concentrations of a trace element with a reference element (Kabata-Pendias *et al.* 1999). The result obtained is described as an enrichment factor (EF) which was calculated using the formula originally introduced by Buat-Menard and Chesselet (Buat-Menard *et al.* 1979) as follows:

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$$EF = \frac{(C_x/C_f)_{sample}}{(B_x/B_f)_{crust}}$$

...(1)

where C_x is the concentration of the examined element in the soil, C_f is the concentration of the examined element in the Earth's Crust, B_x is the concentration of the reference element in the soil and B_f is the concentration of the reference element in the Earth's Crust. A reference element is "conservative" (i.e., the one that content in samples) originates almost exclusively from the earth's crust. The most common reference elements in the literature are Aluminum (Al), Zirconium (Zr), Iron (Fe), Scandium (Sc) and Titanium (Ti) (Blaser *et. al* 2000; Reimann *et. al* 2000; Schiff *et al.* 1999; Schropp *et. al* 1990), although there are also attempts at using other elements e.g., Manganese (Mn) (Loska *et. al* 1997), Chromium (Cr) (McMurtry *et. al* 1995) and Lithium (Li) (Loring 1990). In the present study Fe is taken as reference metal for the calculation of EF since Fe distribution was not associated to other heavy metals (Deely *et. al* 1994). Generally, the enrichment factor is used to assess soil contamination (enrichment) and its interpretation is as EF < 2 – Depletion to minimal enrichment, EF = 2-5 – Moderate enrichment, EF = 5-20 – Significant enrichment, EF = 20-40: – Very high enrichment, EF>40 – Extremely high enrichment (Sutherland 2000).

2.3.2 Geoaccumulation index (I_{geo}): A common approach to estimating the enrichment of metal concentrations above background or baseline concentrations is to calculate the geoaccumulation index (I_{geo}) as proposed by Müller (1969). The method assesses the degree of metal pollution in terms of seven enrichment classes based on the increasing numerical values of the index. This index is calculated as follows:

$$I_{geo} = log_2 \frac{C_n}{1.5B_n}$$

...(2)

where C_n is the concentration of the element in the enriched samples and the B_n is the background value (earth crust) of the element. The factor 1.5 is introduced to minimize the effect of possible variations in the background values which may be attributed to lithological variations in the sediments (Stoffers *et. al* 1986) [28]. The interpretation of the obtained results is as follows: $I_{geo} \leq 0$ practically uncontaminated, $0 < I_{geo} < 1$ uncontaminated to moderately contaminated, $1 < I_{geo} < 2$ moderately contaminated, $2 < I_{geo} < 3$ moderately to heavily contaminated, $3 < I_{geo} < 4$ heavily contaminated, $4 < I_{geo} < 5$ heavily to very heavily contaminated and $I_{geo} \geq 5$ very heavily contaminated.

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2.3.3 Contamination factor (CF): 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. This can be written as (Hakanson 1980)–

$$CF = \frac{C_m\ sample}{C_m\ background} \qquad ...(3)$$

where C_m sample is the concentration of a given metal at contaminated sites and C_m background is concentration of an element in the background soil sample. According to Hakanson (1980), CF has been classified into four groups: CF < 1 low contamination factor, 1 ≤ CF < 3 moderate contamination factor, 3 ≤ CF < 6 considerable contamination factor and CF > 6 very high contamination factor.

2.3.4 Pollution load index (PLI): The pollution load index (PLI), which basically is a measure of site quality, indicated deterioration due to metal contamination. PLI proposed by Thomlinson *et al.* (1980), was calculated using the following relation:

$$PLI = (CF_1 \times CF_2 \times CF_3 \times \ldots \ldots \ldots \ldots \times CF_N)^{1/N} \qquad ...(4)$$

where N is the number of metals studied and CF is the contamination factor discussed above. The PLI is able to give an estimate of the metal contamination status and the necessary action that should be taken. PLI < 1 denote perfection; PLI = 1 present that only baseline levels of pollutants are present and PLI > 1 would indicate deterioration of site quality (Thomlinson *et al.* 1980).

2.3.5 T-test: The *t*-test is one type of inferential statistics. It is used to determine whether there is a significant difference between the means of two groups. In the present study the obtained data were analyzed using *t*-test to know whether there was significant difference between the concentrations of elements in the soil samples. The *t*-test was calculated using Microsoft Excel. Level of parameter was considered significant if *t*-test value was <0.05.

3. RESULTS AND DISCUSSION

3.1 Quality control (QC) test of measurement:

Six samples of standard reference material SRM 2586 were irradiated with 2.55 MeV proton beam and the emitted X-ray spectra were stored. The results obtained by PIXE technique were

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compared with the certified values i.e. values reported by NIST. The ratios of the measured concentrations of the investigated elements in SRM 2586 to the certified value are shown in figure 2. For most of the elements those ratios are very close to one. Thus, the good consistency between the measured and certified values, confirming the reliability of the results acquired.

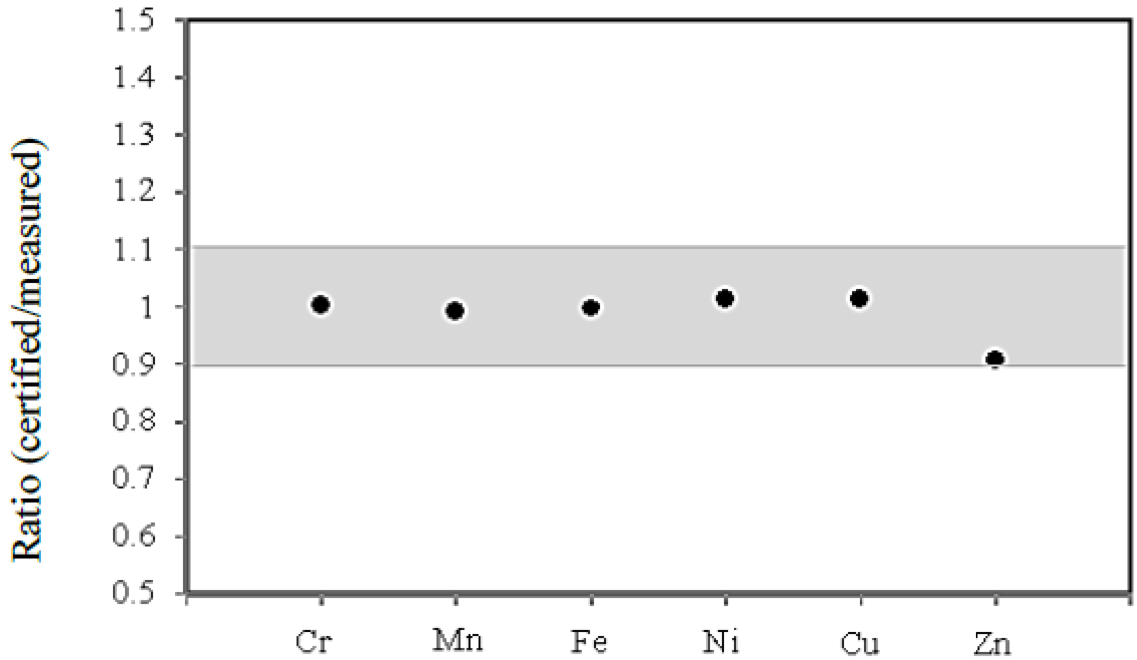

Fig. 2: Quality control (QC) tests of the present measurements.

3.2 Elemental content in soil samples:

Mean concentrations of Cr, Mn, Fe, Ni, Cu and Zn in soils of the studied locations with their range, maximum allowable concentration (MAC) and potential hazardous level (PHL) are presented in table 1. The concentrations are decreased in the order as Fe > Mn > Cr > Ni > Zn > Cu. In this study the mean concentration of Cr and Ni are exceed both MAC and PHL value among six observed elements. In Dumuria soil the average value of Cr exceeds second guideline value (250 ppm; under observation) whereas in Dacope samples which are lies between second and lowest guideline value (100 ppm) (Field *et. al* 1983). These required more attention because Cr is a toxic metal. The toxicity of Cr depends upon its oxidation state. Hexavalent Cr is more toxic than the trivalent form. Cr (VI) compounds penetrate biological membranes much more readily than do Cr (III) compounds. Extra Cr in soil reduces seed germination percentage (Rout *et. al* 2000; Peralta *et al.* 2001), root length (Prasad *et. al* 2001; Iqbal *et. al* 2001), plant height

(Joseph *et. al* 1995; Barton *et. al* 2000) and leaf number (Shanker 2003). Ni is an element that occurs in the environment only at very low levels and is essential in small doses, but it can be dangerous when the maximum tolerable amounts are exceeded. Additional accumulation of Ni seriously affects the yield of plants, significantly decreasing the numbers of seeds and seed yield per plant (Tripathy *et. al* 1981). This approves with literature that crop production rate decreases in the study area (Rasel *et. al* 2013). The excess of Cr and Ni may due to the application of biosolids (e.g., livestock manures, composts, and municipal sewage sludge) to land. Heavy metals most commonly found in biosolids are Pb, Ni, Cd, Cr etc. and the metal concentrations are governed by the nature and the intensity of the industrial activity, as well as the type of process employed during the biosolids treatment (Mattigod *et. al* 1983). The application of numerous biosolids to land inadvertently leads to the accumulation of heavy metals in the soil (Basta *et. al* 2005). It is estimated that in the United States, in the European community and in Australia most biosolids applied to agricultural land are used in arable cropping situations where they can be incorporated into the soil (Silveira *et. al* 2003; McLaughlin *et. al* 2000). Cu and Zn were found at less concentration among the investigated elements. Wet and cold soil conditions can cause Zn deficiency because of slow root growth and slow release of Zn from organic matter (Manjula 2009). In Bangladesh soils Cu and Zn are the most widely deficient in some areas (Islam 1992). Zinc deficiency was also observed in the studies by the workers of Bangladesh Rice Research Institute and Department of Agricultural Extension (BRRI Annual Report, 1980; Mukhopadhayay *et. al* 1986). Fe and Mn are two most important soil elements. Fe serves as an activator for biochemical processes such as respiration, photosynthesis and symbiotic nitrogen fixation (Manjula 2009) and Mn serves as an activator for enzymes in plant growth processes, and it assists iron in chlorophyll formation. Plants obtain this nutrient from the soil in the form of manganous ion (Mn^{+2}). It is key element to many plant functions, including photosynthesis, respiration and nitrogen metabolism, because it forms bridges between enzymes and their substrates (Manjula 2009). In this study Fe and Mn were traced in all soil samples with adequate amount.

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Table 1: Measured concentration of heavy metals in the studied soil with their MAC and PHL value (all values are in ppm range).

Metal (Z)	Dumuria soil		Dacope soil		MAC	PHL
	Min-Max	Mean±SD (*n*=12)	Min-Max	Mean±SD (*n*=12)		
Cr (24)	156-363	279±65	94-298	229±87	50-200	95
Mn (25)	475-855	639±151	211-653	419±134	NA	3900
Fe (26)	26162-49265	41397±5673	34605-48708	41694±4703	NA	NA
Ni (28)	58-173	132±30	95-180	135±26	20-60	100
Cu (29)	11-55	36±13	16-55	35±10	60-150	65
Zn (30)	51-93	72±13	45-94	66±15	100-300	300

SD=Standard deviation; MAC=Maximum allowable concentration; PHL=Potential hazardous level; NA=Not available.

3.3 Enrichment factors (EF):

The EF values of the investigated elements were calculated using Fe as reference material and presented in table 2. Fe usually has a relatively high natural concentration, and is therefore not expected to be substantially enriched from anthropogenic sources in estuarine sediments (Niencheski *et. al* 1994). The EF method normalizes the measured heavy metal content with respect to a sample reference metal such as Fe, Al, Ti etc. Based on the calculated EF values, all the studied soils were moderately enriched with Cr and Ni; depletion to minimal enriched with Mn, Fe, Cu and Zn.

3.4 Contamination factors (CF) and pollution load index (PLI):

Soils of the study areas were also assessed for CF and PLI, and the CF values are given in table 2. The assessment of soil contamination was carried out using the CF based on four classification categories recognized by Hakanson (1980) whereas the PLI was carried out to measure the site quality proposed by Thomlinson *et al.* (1980). The overall contamination of soils at the site, based on the CF values indicate that the soils were moderately contaminated with Ni but showed signs of low contamination with Cr, Mn, Fe, Cu and Zn. In this study, the PLI values of Dumuria and Dacope sampling site were found 1.09 and 0.98 respectively which indicate deterioration of

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site quality and perfection respectively. Thus, an immediate mediation to ameliorate pollution at Dumuria site is needed.

Table 2: Enrichment factors, contamination factors and geoaccumulation index of heavy metals.

Metal (Z)	Enrichment factors (EF)		Contamination factors (CF)		Geoaccumulation index (Igeo)	
	Dumuria Soil	Dacope Soil	Dumuria Soil	Dacope Soil	Dumuria Soil	Dacope Soil
Cr (24)	3.04	3.72	0.76	0.93	0.58	0.87
Mn (25)	0.59	0.92	0.42	0.64	-1.77	-1.16
Fe (26)	1	1	0.81	0.8	-1.02	-1.03
Ni (28)	2.17	2.14	1.8	1.76	0.1	0.07
Cu (29)	0.79	0.83	0.43	0.45	-1.36	-1.3
Zn (30)	1.27	1.4	0.19	0.21	-0.67	-0.54

3.5 Geoaccumulation index (I_{geo}):

The geoaccumulation index was assessed based on the seven descriptive classes for increasing I_{geo} values proposed by Müller (1969) stated previously. The calculated values of the I_{geo} in the studied soils are discussed in conjunction with the EF and the CF results, presented in table 2 and figure 3. Data reveal uncontaminated to moderate contamination of soils of both region by Cr and Ni. The negative values in the Mn, Fe, Cu and Zn indexes of geoaccumulation are a result of deficient to minimal enrichment and/or relative low level of contamination (table 2). Although the nature of I_{geo} calculation, which involves the logarithmic function and a background multiplication factor of 1.5, is somewhat different from other pollution calculation methods discussed in this study, the I_{geo} factors are in general comparable to results reported for EFs and CFs.

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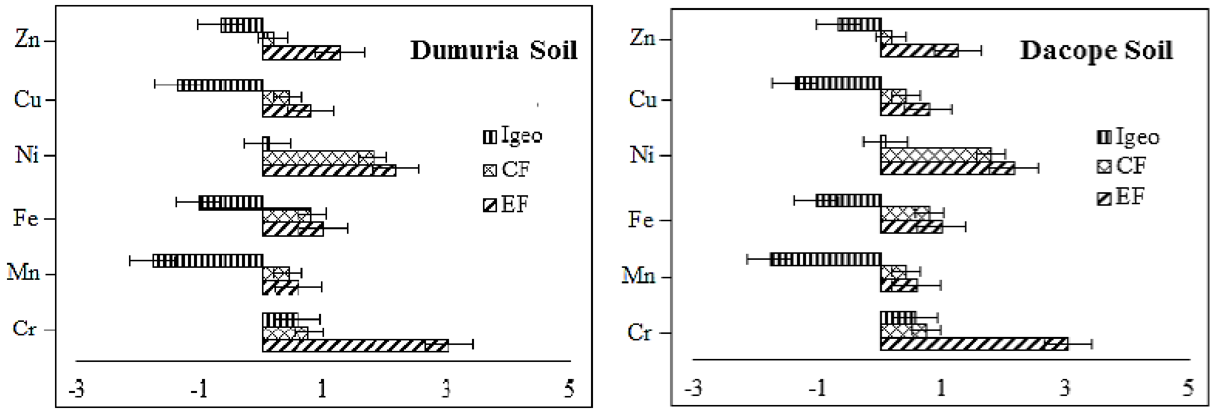

Fig. 3: The enrichment factors, contamination factors and geoaccumulation index.

3.6 T-test:

The *t*-test has been employed to identify the comparison of mean concentration of heavy metals between two sampling sites. Statistical test reveals that among the observed heavy metals only Mn shows considerable difference between two areas with significant value (< 0.05).

4. CONCLUSIONS

In the present study the assessment of pollution in cultivated soils by heavy metals (Cr, Mn, Fe, Ni, Cu and Zn) in two regions of south-western part of Bangladesh was analyzed with PIXE technique. Pollution by these metals was examined using several calculation methods: enrichment factors (EF), contamination factors (CF), pollution load index (PLI) and geoaccumulation index (I_{geo}). The study shows that among the detected heavy metals Cr and Ni were found above their PHL and MAC values which decreases germination percentage as well as growth rate in some cases. This regards with literature that crop production rate decreases in the study area. This was corroborated by their raised EF, CF and I_{geo} values, suggesting significant anthropogenic contribution to the elemental concentration of the soils. The elevated levels of these elements reported may result in the accumulation of potential toxic elements by the food stuff which may cause ill health. The PLI value of Dumuria goes above 1, indicating deterioration of the site quality. The *t*-test value displays that among the observed heavy metals only Mn shows considerable difference between the two areas. This study revealed the need for further detailed metal enrichment assessment in the study area and the country as well.

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