

Research article

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Geochemistry of sediment archives of agricultural lands with reference to past mining in Goa, West-Coast of India

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ABSTRACT

The impact of iron ore mining on agricultural lands through quantitative determination of major elements (Fe, Mn and Al) and minor elements(Cu, Zn, Pb, Ni and Cr) was assessed. A total of 54 soil core samples were collected from 3 mine affected areasviz., Mayem agriculture field (MF), Pernemagriculture field (PF) and Sanvordem agricultural field (SF) in Goa. Different pollution indices were used to assess the pollution status of the samples. Major elements and trace metal concentrationswere assessed through enrichment factor (EF), Contamination factor (CF), Pollution load index (PLI) and geo-accumulation index (Igeo). Enrichment factors showed significant moderate enrichment for Fe, Pb and Cr in MF and SF cores can be attributed to anthropogenic factors. Contamination factors for Mn and Pb were high in MF core. While, SF core showed considerable contamination for Pb. Pollution load index indicated that MF and SF cores are polluted (PLI >1) and PF core is unpolluted (<1). Igeo values in MF core showed moderately to strongly polluted with Fe and Cr, Whereas SF core was moderately polluted with Pb. While higher values of metals in MF and SF can be attributed to the steep slopes of dumps and unconsolidated nature which gets washed down in the agricultural lands enhancing the metal concentrations.

KEY WORDS: Mayem, Sanvordem, Pernem agricultural land, soil, pollution

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INTRODUCTION

Soil is not only the material on which land organisms live but also play an important role in material cycles and energy exchange in terrestrial ecosystems. Rapid industrialization and urbanization have led to soil pollution becoming a serious environmental problem ¹. Increased heavy metal concentrations are particularly harmful because heavy metals are toxic, persistent ². Physical and chemical processes (leaching and oxidation) accumulated ion of heavy metals in soil, and enter water bodies or cropped areas^{3, 4}. Some heavy metals like Cu, Fe, Mn, Zn are required for growth of plants in trace amounts, but can prove fatal in excess amounts^{5,6}.

Several studies have documented the human activities as a major cause for heavy metal contamination of the soil ecosystem ^{7,8}. Mining no doubt impacts negatively on agriculture because of the complex catena of various interactions that occur as wastes become part of the ecosystem ⁹. There is an increasing need to study heavy metal distribution and accumulation in agricultural soils. Numerous studies on mining and effects on soil, plants and water have been carried out in several countries. ¹⁰reported that mine wastes alterphysico-chemical properties and affect plant growth supportive nutrients. Hence, pose danger mixed with agricultural soils ¹¹.

Iron ore mining in Goa runs by open-cast method of extraction that has serious effects on the environment. In Goa to obtain 1 mnt of iron ore around 2.5 to 3mnts of overburden has to be excavated resulting in problems of storage of dumps. The was tesoccupy more space than allotted to the mining companies for their operations. This accumulated silt enters the fields during rains making the lands unfit for cultivation. The mining industry requires water in huge quantities for backwashing of the ore. The mining dumps have resulted in a permanent damage of the local area. Mining is one of the major concerns causing land degradation as about 12,000 hectares have been rendered wastelands due to mining which is 3% of the total geographical area ¹². ¹³ reported a decline in agriculture due to mining activities in the areas of operation. The four talukas: Bicholim and Sattari in North Goa and Sanguem and Quepem in South Goa largely fall in the mining belt. This accounts for almost 1/5th of the area of Goa i. e, approximately 700 km². The inhabitants in these areas were largely dependent on agriculture for their livelihoods.

MATERIALS AND METHODS

This study was aimed to identify the effect of iron ore mining and processing on quality of sediment cores from agricultural lands through quantitative determination of trace metals. Study area was located at 1. Iron ore affected agricultural lands of northern and southern region of Goa and 2. An agricultural land from non-mining area, in Goa, India (Fig.1).

Two cores one each from Mayem Agricultural Land (North Goa) and Sanvordem Agricultural Land (South Goa) in the vicinity of iron ore mining area and one from pernem agricultural land from a non-mining area were collected.

Mayem Agricultural Land (MF): Mayem Agricultural Land lies in Bicholim Taluka in North Goa which largely fall in the mining belt. The inhabitants in these areas were largely dependent on agriculture for their livelihoods. But with mining industry there was a complete change in the occupational structure. From the point of view of mineral production Bicholim contributes almost 60% of the value of minerals ¹⁴. The average annual rainfall of the order of 3714 mm and 3690 mm has been recorded at Bicholim, which receives mostly from south- west monsoon(Fig.1)¹⁵. Due to the steep slopes of dumps and unconsolidated nature of their constituents, dump materials get washed down the slope either filling up the low-lying agricultural land causing siltation and transforming it into uncultivable land¹⁶.

Sanvordem Agricultural Land (SF): Sanvordem Agricultural Land lies in Sanvordem which is Sanguem Taluka in South Goa District, Goa. Mining is one of the major activities of Sanvordem. Lot of trucks carry the ore from one point to another by road. The spillage of the ore during the transportation on the roads enter the agricultural lands in the adjoining areas during monsoon.

*Pernem Agricultural Land (PF):*Pernem Agricultural Land lies in Pernemtaluka bounded by the Chapora River to the south and Terekhol River to the north. It is the northern most subdivision of Goa that touches the Maharashtra border. Most inhabitants in the area survive on the local land for farming.



Figure 1.Location Map

The core samples were collected from three agricultural lands using acrylic pipes (50cm long and 4.5cm dia) at a depth of 25cm. Inspection on the field revealed that the cores are intact without any disturbance at the surface(Fig.2). The cores were sliced at 2 cm interval and preserved in the deep

freezer until the analyses. The samples were dried at 60° C in a hot air oven and in total, 80 subsamples were analyzed for grain size, organic carbon, major elements and trace metals content. The sediment grain size was analyzed by pipette analysis following ¹⁷. The organic carbon (OC) content in the sample was determined following ¹⁸.

Major elements and trace metals

For elemental analysis, 0.3g of each sample was digested using an acid mixture (HF:HNO₃:HClO₄ in 7:3:1 ratio) ¹⁹ and evaporated almost to dryness using ANA LAB hot plate. Supra pure acids (Merck) were used for digestion and Milli Q water was used for preparation of standard solutions and dilutions. After cooling the residue was dissolved and diluted to 50 ml with 1N HNO₃. The samples were analyzed for major elements (Fe, Mn and Al) on ICP-OES (Model: Agilent 710 series). A multi element standard (23 elements, Merck Germany) was used for calibration. Instrument sensitivity was frequently monitored with respect to mixed standard solutions during the analysis. Trace metals (Cu, Zn, Pb, Ni and Cr) were analyzed on Atomic Absorption Spectrophotometer (AAS Model GBC 932 AA). Standard stock solution of each metal was prepared using Merck standard solutions. The precision and accuracy of the metal analyses were checked against certified reference material (SCO-I) in triplicate. The recoveries of all the metals varied between 88% to 99% except for Cu, for which it was 81%. Pearson's correlation coefficient was used to understand the inter-relationship between metals and other sediment parameters.

Evaluation of statistical parameters

Enrichment factor: Enrichment factor is one of the indices to compute the sedimentary metal source contributed by anthropogenic activities or by natural sources ^{20, 21, 22}. This index was calculated based on a normalization element(Fe or Al) which moderates the variations caused by heterogeneous sedimentation ^{23, 24}. It permits to calculate the heavy metal contamination and it was calculated by,

EF = (Y/X) sample / (Y/X) reference

Y sample - trace element concentration in the sample;

X Reference- trace element concentration in the continental crust ²⁵.

Y sample - Al content in the sample;

X Reference - Al content in the continental crust ²⁶.

Five contamination categories were recognized based on the enrichment factor ²⁷.

EF values< 2 Deficiency to minimal enrichment

EF values 2 <5 Moderate enrichment

EF values 5 <20Significant enrichment

EF values 20<40 Very high enrichment

EF values > 40 Extremely high enrichment

Contamination factor: The level of contamination in the sediment core was expressed by contamination factor (CF), calculated using trace metal data and metal concentration for the world shale average ²⁸ as the background value. The contamination Factor (CF) was calculated by,

 $CF_{metal} = C_{metal} / C_{background}$

 $C_{\mbox{\scriptsize metal}}$ - metal contamination in polluted sediment.

 $C_{\mbox{ background}}$ - background value of that metal.

Four categories of contamination factor were distinguished,

 $1 \leq$ CF Low contamination

 $1 \le CF < 3$ Moderate contamination

 $3 \le CF < 6$ Considerably contaminated

 $6 \ge CF > Highly contaminated$

Pollution load index: The pollution level in trace metal was calculated based on pollution load index ²⁹ This is a simple method to assess the extent of pollution by metals in estuarines.

PLI= n (CF1 × CF2 × CF3X.....CFn)

CF = Contamination factor.

n = Number of metals.

Index of Geo-accumulation: Geo-accumulation Index (Igeo) ³⁰was calculated to ascertain and define metal contamination in sediment samples by comparing current concentrations with pre-industrial levels. The Index of Geoaccumulation (Igeo) was computed using ^{26, 31}.

Igeo = $\log 2 Cn/1.5Bn$

Where Cn is the measured concentration of the element in the core sediment fraction and Bn is the geochemical background value (average shale) in the earth's crust (Wedepohl 1995). The constant 1.5 allows for natural fluctuations in the content of a given substance in the environment with very small anthropogenic influences. Six classes of the geochemical index have been distinguished ³².

Class Value Sediment Quality

0 Igeo values < 0 Practically uncontaminated

1Igeo values <1 Uncontaminated to moderately contaminated

- 2 Igeo values < 2Moderately contaminated
 - 3 Igeo values < 3 Moderately to heavily contaminated
 - 4 Igeo values < 4 Heavily contaminated
- 5 Igeo values < 5Heavily to extremely contaminated
 - 6 Igeo values > 5Extremely contaminated

Field observations: The colour of a mine spoils or weathered mine soil can tell us much about its weathering history, chemical properties, and physical make-up. The sediment core PF showed grey colour (Fig.2). Grey colors in rocks, spoils and soils usually indicate a lack of oxidation and leaching. These materials tend to be higher in pH and fertility ³³. Whereas, sediment core collected from MF and SF showed yellowish brown/ Brown. Bright red and brown colors in spoils and soils generally indicated that the material was oxidized and leached to some degree. These materials tend to be lower in pH and free salts, less fertile, low in pyrites, and more susceptible to physical weathering than dark materials ³³.

RESULTS AND DISCUSSION

Sediment composition and texture:

Table No. : 1 Range and average for sand, silt, clay ,OC, major elements and trace metals in cores collected from Mayem agricultural land (MF), Sanvordem agricultural land (SF) and Pernem agricultural land (PF),

Parameters	MF		SF		PF	
	Range	Average	Range	Average	Range	Average
sand(%)	2.58-32.05	23.75±8.58	51.70-77.59	68.35±6.39	35.15-84.09	61.5±16.99
silt(%)	46.54-84.87	63.49±10.05	17.34-36.14	24.64±5.41	14.05-55.76	32.6±15.72
clay(%)	8.54-23.34	12.76±4.09	2.59-12.16	7.00±2.32	1.86-9.09	5.9±2.1
OC(%)	1.52-2.69	2.15±0.35	0.23-1.01	0.40±0.21	1.09-1.29	1.22±0.07
Fe(%)	20.14-32.79	26.27±3.86	2.35-11.21	8.04±2.31	0.12-0.22	0.17±0.03
Mn(%)	0.52-1.10	0.78±0.19	0.02-0.27	0.09 ± 0.08	0.12-0.20	0.16±0.02
Al(%)	7.05-11.95	9.67±1.47	5.81-9.85	7.77±1.24	7.79-11.18	9.93±0.99
Cu(µg g-1)	18.00-28.00	23.90±3.01	8.33-76.00	27.82±22.97	22.33-124.00	53.78±23.64
Zn(µg g-1)	132.24-189.90	151.87±17.33	30.24-99.90	66.42±26.85	64.67-112.67	93.22±11.60
Pb(µg g-1)	75.33-187.00	136.10±35.11	34.00-79.33	61.97±11.62	17.33-99.33	63.69±21.70
Ni(µg g-1)	51.67-131.00	94.03±28.25	16.00-47.33	33.38±10.42	112.67-181.00	156.75±20.89
Cr(µg g-1)	187.33-321.33	279.10±43.20	111.33-215.00	174.84±34.26	106.67-234.00	172.42±41.52

The sediment core collected at Mayem agricultural land (MF) recorded the highest silt content (63.49%); moderate content of sand (23.75%) and lower clay content (12.76%) (Table 1).Thus exhibiting sandy silt type sediment texture. Higher concentration of silt may be due to runoff from the mine dumps on the steep hills in the adjoining areas. The downcore variation of sand showed a decreasing trend from bottom up to 8cm depth and thereafter showed an increasing trend up to the surface of the core. Distribution of silt compensates the variation of sand throughout the length of the core. Where as clay showed an irregular trend. Organic carbon showed an increasing trend from bottom up to 14cm depth and thereafter it showed a decreasing trend up to the surface of the core with a range of 1.52- 2.69(2.15%)(Fig.2a).

Sediment core collected from the Sanvordem agricultural land (SF) recorded (Table 1) higher values of sand (68.35%); moderate silt (24.64%) and lower values of clay (7.00 %) thus indicating silty sand texture type unlike in SF core. Vertical profile of sand showed an increasing trend from bottom

up to the surface of the core. Whereas distribution of silt compensates the variation of sand throughout the length of the core and clay showed an irregular trend. Organic carbon content(%) of core ranges from 0.23-1.01(0.40%), the down core variation showed a decreasing trend from bottom upto the surface of the core (Fig 2b).

The sediment core collected from Pernem agricultural land, sand recorded higher values(61.5%); moderate silt(32.6%) and lower values by clay (5.9%)(Table 1). Vertical profile of sand showed an increasing trend from bottom up to the surface of the core, whereas silt compensates sand throughout the length of the core, clay showed a decreasing trend from bottom up to the surface of the core. Organic carbon content of the core ranges from1.09-1.29(1.22%).the down core variation showed an increasing trend from bottom up to the surface of the core (Fig 2c).



b.

a.



c.



Figure 2. Variation of sand, silt, clay and OC (%) in sediment cores of a. Mayem agricultural land (MF), b. Sanvordem agricultural land (SF) and c. Pernem agricultural land (PF)

Metal geo-chemistry

It is found that the minimum average concentration of Fe in the soil is 26.27 %, the variation in the concentration over the core depth is from 20.14-32.79 % in MF core, whereas in the PF the average concentration varies between 0.12-0.22 (0.17%) (Table 1). It is estimated that this variation is randomly distributed over the core depth with varying concentrations. It is estimated that the varying concentration is correlated with the quantity of runoff and the deposition of soil layer yearly. Similar patterns of random distribution was recorded in SF core(Fig. 3a) with an average of 8.04% and the variation is from 2.35-11.21%, which confirms that the concentration build up in soil relates to proportional runoff during the monsoon season. MF contained more Fe indicating the influence of mining dumps at the vicinity of it.

Mn concentration(%) in MF core was randomly distributed with minimum(0.52) at 4cm depth and maximum(1.10) at 6cm depth of the core(Fig. 3b).So, the similar pattern of runoff was recorded in Fe concentration in MF core at 8cm depth. The SF core also appeared to follow similar random concentration build-up with a range of 0.02-0.27(0.09%).It was observed that maximum concentration build-up coincided with 6cm depth similar to Fe found. However, the concentration in PF varied from 0.12-0.20(0.16%).

Depth variation of Al in MF, SF and PF was 7.05-11.95(9.67), 5.81-9.85(7.77) and 7.79-11.18(9.93), respectively. PF and MF core showed minimum variation in Al concentration close to the world's shale value, the Al percentage variation at 14cm depth showed higher values than the normal variation (Fig. 3c). This particular phenomenon of Al building up at specific depth of 14cm needs to be further studied.

It is evident from the table that the concentration of Cu in MF, SF and PF varied from 18.00-28.00(23.90), 8.33-76.00(27.82) and 22.33-124.00(53.78). In MF and SF, core concentration of Cu had more or less remained same (Fig.3d).However, a significant rise in Cu concentration at 8cm

depth in PF core was from the non-mining region may probably be due to excessive runoff and soil deposition for the buildup of Cu percentage.

Depth variation of Zn in MF, SF and PF was 132.24-189.90(151.87), 30.24-99.90(66.42) and 64.67-112.67(93.22).Zn concentration in PF core was from non-mining region showed much less variation. While MF core showed wider variation with a peak at 8cm depth(Fig. 3e). However, in SF core there was sudden drop of Zn concentration. In MF core Zn concentration was much higher than in PF and SF core with a peak at 8cm depth indicating that average Zn content in MF core was uniformly higher compared to other cores.

Concentration of Pb in MF, SF and PF varied from 75.33-187.00(136.10), 34.00-79.33(61.97%) and 17.33-99.33(63.69%). PF and SF core did not show much variation in Pb concentration (Fig.3f). The concentration of Pb in PF core was consistently less. It was consistently more in SF core. However, in MF core it registered higher concentration at 8cm depth. Depth variation of Ni in MF, SF and PF ranged from 51.67-131.00(94.03), 16.00-47.33(33.38) and 112.67-181.00(156.75) respectively. MF and SF did not show much variation. The concentration of Ni in PF core was consistently high. In SF core it was consistently low (Fig. 3g).

Concentration of Cr varied from 187.33-321.33(279.10), 111.33-215.00(174.84) and 106.67-234.00(172.42). MF, SF and PF did not show much variation upto 4cm depth and thereafter MF showed high values compared to SF and PF core (Fig. 3h).In general MF core showed high concentration of Fe, Mn, Zn, Pb and Cr, whereas SF core showed high concentration of Pb and Cr. However, PF core was enriched with Pb, Ni and Cr compared to shale values.

Relatively high values of elemental concentrations of Fe and Mn obtained in MF core must be directly reflecting large input of these elements from old mine dumps which stabilized later. Higher elemental concentration of Fe, Mn, and Zn indicated an increase of input of these elements in recent years³⁴. About 60% of the total iron-ore reserves of Goa came from the Northern zone which included BicholimTaluka¹⁴. Heavy metals from mining sites may reach agricultural soils through leaching. Also during the rainy season large quantities of tailing and waste containing heavy metals are carried by runoff to the agricultural fields near the mining sites which lead to the elevated levels of heavy metals in the soils ⁹. Iron ore mines act as an important source of major metals; mainly Fe and Mn, and also, contributes for trace metals into the environment ^{35, 36, 37}.



Figure 3.Variation, Major elements (Fe, Mn and Al) and Trace metals (Cu, Zn, Pb, Ni and Cr) in sediment cores of a. Mayem agricultural land (MF), b. Sanvordem agricultural land (SF) and c. Pernem agricultural land (PF)

Evaluation Indices

Enrichment factor: The enrichment factor (EF) was used to assess metal contamination in the sediments of MF, SF and PF (Fig. 4a,b,c). In MF core, Mn and Pb showed significant enrichment, Fe and Cr showed moderate enrichment suggesting significant anthropogenic inputs, whereas Cu, Zn and Ni showed deficiency to minimal enrichment (Fig. 4a). Enrichment factor for SF core (Fig.4b). Fe, Pb and Cr showed moderate enrichment, whereas Mn, Cu, Zn and Ni showed deficiency to minimal enrichment (Fig. 4b). However in PF core Fe, Mn, Cu, Z, Pb, Ni and Cr showed deficiency

to minimal enrichment (Fig. 4c). Significant moderate enrichment of Fe, Pb and Cr in MF and SF cores can be attributed to anthropogenic inputs while other elements (Mn, Cu, Zn and Ni) may be of crustal origin. According to ³⁸, EF values between 0.05 and 1.5 indicated that the metal was entirely from crustal materials or natural processes. EF values higher than 1.5 suggest that the sources are more likely to be anthropogenic.



Figure 4.Enrichment factor for major elements and trace metals in cores collected from a. Mayem agricultural land (MF), b. Sanvordem agricultural land (SF) and c. Pernem agricultural land (PF)

Contamination factor: Contamination factor for MF core indicated that Mn and Pb were highly contaminated; Fe and Cr are considerably contaminated; Al, Zn and Ni were moderately contaminated and Cu showed low contamination (Fig.5a).In SF core Pb showed considerable contamination; Fe and Cr were moderately contaminated and Mn, Al, Cu; Zn and Ni showed low contamination (Fig. 5b). However, In PF core, Mn, Cu, Pb and Ni showed moderate contamination and Fe, Al, Zn and Cr showed low contamination (Fig. 5c).

High contamination for Mn and Pb and considerable contamination for Fe and Cr in MF sediment core can be attributed to enormous mining activities and discharge of wastes. Moderate contamination of Al may bedue to use of aluminum salts in treating wastes from the mining pits. Considerable contamination of Pb in SF sediment core may be due to run off from mining waste which in cludedtrace elements and minerals often associated with iron deposits ³⁹. On the other hand, low contamination of Fe in PF core revealed that this area is not affected by mining activities.Soil contained various functional groups which are effective agents for heavy metal sorption. Their

interactions affects the properties and processes in the soil which are determined by the level of Fe, Al, and Mn oxides and hydroxides, rainfall distribution, erosion, soil drainage, redox potential, texture and organic matter and clay content. The dominant process at any specific time determines the heavy metal retention capacity ^{40, 41}. Pb production and operation facilities without a waste-gas treatment system, battery production and scrap battery recovery facilities, thermal power plants, and iron–steel industries are the other lead sources. Moreover, among the heavy metals, Pbwas the most immobile element and its content in soil was closely associated with clay minerals, Mn-oxides, Al and Fe hydroxides, and organic material ⁴². Cr behavior in soil was regulated by soil pH and redox potential⁴³.





Figure 5.Contamination factor for major elements and trace metals in cores collected from a. Mayem agricultural land (MF), b. Sanvordem agricultural land (SF) and c. Pernem agricultural land (PF)

Pollution load index: PLI values in MF, SF and PF cores varied from 2.02 to 2.83 (2.47); 0.75to 1.25(1.00) and 0.66to 0.87(0.76), respectively. These results indicated that MF and SF cores are polluted (PLI >1) and PF core is unpolluted (<1). Higher PLI values of MF and SF cores can be attributed to enrichment of pollutants due to the steep slopes of dumps and unconsolidated nature of their constituents, dump materials get washed down the slope either filling up the low-lying agricultural land of Mayem area and due to transportation of ore by roads in the Sanvordem area. The downcore variation of PLI (Fig.6) in MF core showed a decreasing trend from bottom up to 4cm depth and thereafter it showed an increasing trend upto the surface of the core, whereas SF and PF

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core showed constant low values from bottom upto 10cm depth and thereafter it showed an increase in values upto the surface of the core.



Figure 6.Downcore variation of PLI in sediment cores of Mayem agricultural land (MF), Sanvordem agricultural land (SF) and Pernem agricultural land (PF)

Geo-accumulation Index (Igeo): The I-geo grades for the study area sediments varied from metal to metal and site to site (across metals and sites) ⁴⁴. Geo-accumulation Index is presented in Fig. 7a,b,c. Igeo values in MF core is moderately to strongly polluted with Mn; moderately polluted with Fe and Cr; unpolluted to moderately polluted with Zn and unpolluted with Al, Cu, Pb and Ni, Whereas, SF core is moderately polluted with Pb; unpolluted to moderately polluted with Fe and Cr and unpolluted with Mn, Al, Cu, Zn and Ni. However, PF core was not polluted to moderately polluted with Fe, Al, Cu, Zn, Ni and Cr. The moderate contamination of Cr might be associated with the presence of liquid manure, composed materials and agrochemicals such as fertilizers and pesticides ⁴⁵. The increase of Pb in sediments is caused by the use of combustion of petroleum and diesel and other traffic activities ⁴⁶. Dispersal of Fe mining wastes rich in Pb, Zn, and Cd is the likely source of these metals in soils ⁴⁷.







Figure 7.Geoaccumulation (Igeo) for major elements and trace metals in cores collected from a.Mayem agricultural land (MF), b. Sanvordem agricultural land (SF) and c. Pernem agricultural land (PF)

CONCLUSION

Of the three agricultural lands, Mayem agricultural land is more enriched with Fe, Mn, Pb and Cr due to large scale extraction of minerals from Fe-Mn ores and dumping of mine wastes. Contamination factors revealed that Mayem agricultural land is highly contaminated with Mn and Pband considerable contamination of Pb in SF core due to the runoff from the mining-related activities. Geo accumulation index (Igeo) of metals indicated that MF core was highly polluted by Mn due to anthropogenic factor; SF core was moderately polluted by Pb. Pollution load index indicated that MF and SF sediment cores were polluted (PLI > 1). PF core did not show any pollution. This study revealed that mining has an adverse impact on the sediment quality of Mayem agricultural land and Sanvordem agricultural land.

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