

## Influence of Soil Organic Carbon and Ph on Concentrations and Distribution of the Metals, Cu, Fe, Mn, Ni, and Zn in Agricultural Soil

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### Original Research Article

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**Abstract:** A detailed study was carried out with respect to soil organic carbon (SOC), pH and Cu, Fe, Mn, Ni, and Zn on agricultural soil used for paddy rice cultivation for many years. The accumulation of Cu, Fe, Mn, Ni, and Zn in the soil has been correlated with pH and the presence of the five metals. Basic statistics has been elaborately used to explain the results and pollution indices are calculated to estimate the degree of soil degradation. Principal component analysis was carried out to identify the factors responsible for the soil quality degradation. The results throw light on quality of the soil from a well-known agricultural ecosystem.

**Keywords:** organic carbon, soil, paddy rice

### INTRODUCTION

Heavy metal contamination of soils is considered as a global environmental issue in agricultural ecosystem around the world. Over the years, heavy metal accumulation can reduce the quality of agricultural products, crop yield, degrade soil quality and influence the physicochemical properties of the soil. Due to development of industrial activities, urbanization, and agricultural practices, the potentially toxic heavy metals have accumulated in soils [1]. Unlike most of the organic contaminants which lose their toxicity with biodegradation, the metals can result in long lasting toxic effects as these are not degraded [2].

Metal contamination of soil has increased due to mining or industrial activities, improper use of heavy metal-enriched materials in agriculture, including chemical fertilizer and pesticides, industrial effluents, sewage sludge and wastewater irrigation [3].

Besides, environmental conditions such as pH, redox potential, silt, clay and organic matter contents of soil play important role in the availability of the metals [4]. The ability to accumulate heavy metals is associated with the soil type, its physical (i.e., texture, especially the content of clay fraction) and chemical properties as well as the nature of the individual heavy metal [5]. Among the soil properties, soil pH and soil organic carbon (SOC) have played the most important role in determining metal speciation, solubility from mineral surfaces, movement, and eventual bioavailability of the metals both in the soil as a whole and in the soil solution particularly [6].

The majority of metals in soils remains bound in minerals and organic matter (OM, such as passive OM or humus), and is unavailable to plants [7]. It has been shown that heavy metal adsorption onto soil constituents decline with decreased soil organic carbon in soils. Moreover, the dissolved organic matter in soils could increase the mobility and uptake of heavy metals

by plant roots [8]. On the other hand, organic matter is also involved in supplying organic chemicals to the soil solution, which may serve as chelates and increase metal availability to plants [9]. Soil pH influences the trace metal mobility and it is known that acidic soils favour and alkaline soils lower their mobility across the soil matrix. Lack of oxygen in the soil can also trigger mobility of the metal ions [10].

High contents of heavy metals in soils would increase the potential uptake of these metals by plants. Therefore, a detailed risk assessment of heavy metal accumulation in agricultural lands is required for application of inorganic fertilizers, organic wastes and pesticides to soils in order to ensure safe crop production [11].

Although metals like Cu, Fe, Mn, Zn etc., are essential at low levels, other metals, like Cd, Cr, Pb and As, are toxic and may pose a great threat to plants, animals and humans through the food chain [12]. Fe

and Mn are the most abundant metals in the lithosphere, and they generally occur as Fe/Mn oxides and hydroxides, which play an important role in precipitation or solubility of some heavy metals in soils [7]. Cu and Zn are supplied as additives to animal feed for antimicrobial effects and growth promotion, and may be mostly excreted in animal manure [13].

In this work, an intensive study was conducted to profile the accumulation of Cu, Fe, Mn, Ni, and Zn in soil within an agricultural ecosystem with a view to assess the soil pollution index level with respect to the metals and to correlate the metal contamination with pH and soil organic carbon contents. The results may be used to provide the baseline information for the paddy-cultivation soil quality.

**MATERIALS AND METHODS**

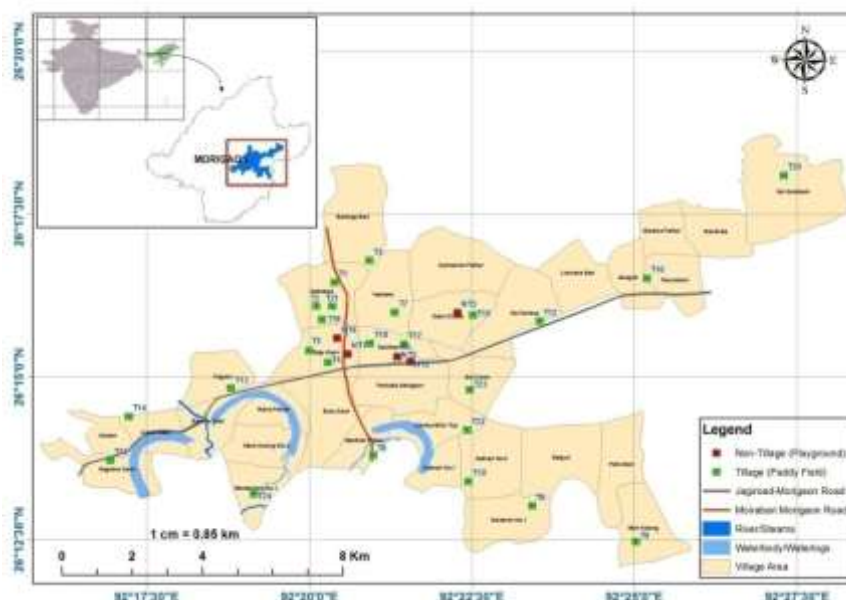
**Site Description**

An area known for paddy rice production, with a nearly plain topography and a subtropical

climate of warm and humid summer followed by a cool winter was selected. The average annual rainfall for the last three years has been about 1500 to 2600 mm. The pre and post monsoon months have unpredictable and erratic rainfall. The mean temperature varies from 37 to 39 °C and 6 to 7 °C respectively during the summer and the winter.

**Soil Sampling and Processing**

Soil samples were collected from 25 sites of tilled rice growing areas (T1 to T25). The locations of the sampling sites are shown in Fig. 1 and the GPS coordinates are presented in Table 1. The study area is humid, alluvial and flooded with traditional rice cultivation practiced for several generations. Farmers have been using urea, sodium superphosphate, single super phosphate (SSP) monoammonium phosphate, murate of potash as fertilizer supplements on a regular basis with every batch of cultivation.

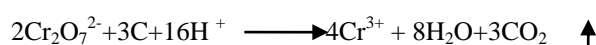


**Fig-1: Location Map of sampling sites**

**Table-1: Locations of the soil sampling sites, T1 – T25, from agricultural areas**

Sample Identity	Latitude (N)	Longitude(E)	Sample Identity	Latitude (N)	Longitude(E)
T1	26°16'27.49"	92°20'23.36"	T14	26°14'23.71"	92°17'13.51"
T2	26°16'05.81"	92°20'05.87"	T15	26°15'57.43"	92°22'31.03"
T3	26°16'47.60"	92°20'55.52"	T16	26°16'31.22"	92°25'11.48"
T4	26°15'13.74"	92°20'17.30"	T17	26°15'30.56"	92°21'27.66"
T5	26°15'24.52"	92°19'59.78"	T18	26°15'31.22"	92°20'56.23"
T6	26°13'01.52"	92°23'25.57"	T19	26°15'51.68"	92°20'11.67"
T7	26°15'59.87"	92°21'18.86"	T20	26°18'06.02"	92°27'17.88"
T8	26°13'48.19"	92°20'59.01"	T21	26°16'05.48"	92°20'21.26"
T9	26°12'28.51"	92°25'01.45"	T22	26°14'11.39"	92°22'25.70"
T10	26°13'24.04"	92°22'27.07"	T23	26°14'48.37"	92°22'27.86"
T11	26°13'43.64"	92°16'56.50"	T24	26°13'12.52"	92°19'08.78"
T12	26°15'50.31"	92°23'33.43"	T25	26°15'24.51"	92°19'59.77"
T13	26°14'49.74"	92°18'47.54"			

Soil samples were collected from 0 – 15 cm depth (average conventional tillage depth) twice a year during the hot, humid summer (June-July, S1, S2 and S3) and the cold, dry winter (December-January, W1, W2 and W3) for 3 years. From each site, three soil samples were collected from an area of 1 m<sup>2</sup> (one from the centre and two from two diagonally opposite corners) using a 5 cm corer. Each of the three samples was air dried in a shade, pebbles, roots etc. were removed, and after further drying, ground to a fine powder and passed through a 2 mm sieve. The three samples from a single site were homogenized to obtain a single composite sample representing the specific soil type of the particular site. The soil samples were stored in desiccators at room temperature till laboratory analysis was completed.



1ml of 1N dichromate is equivalent to 3 mg of carbon = 0.003 g of org. C

% of org. C in the soil (uncorrected) =  $0.5 \times (B-S) \times N$  of  $\text{K}_2\text{Cr}_2\text{O}_7 \times 0.003 \times (100/W)$

% of org. C in the soil (corrected) = % of org. C in the soil (uncorrected)  $\times 1.3$

where B = Vol. of 0.5 N ferrous ammonium sulphate solution used for blank titration, S = Vol. of 0.5 N ferrous ammonium sulphate solution used for sample titration, W = Wt. of the soil. 1.3 is a correction factor

Cu, Fe, Mn, Ni and Zn in the soil samples were determined in a soil extract obtained by digesting 1 g of soil sample in a 1:2:4 mixture of hydrochloric, nitric and sulphuric acids, keeping overnight and making the volume to 100 ml and then, analysing in an Atomic Absorption Spectrophotometer (PerkinElmer AAnalyst 200) with air-acetylene flame. The determined concentrations are converted to mg kg<sup>-1</sup> by dividing the product of AAS reading in mg L<sup>-1</sup> and volume of extract with oven dry weight of soil

### STATISTICAL ANALYSES

Descriptive statistical parameters including mean, maximum, minimum, median, standard deviation, coefficients of variation (CV), skewness and kurtosis and K-sp (Kolmogorov-Smirnov) test for normality are obtained using IBM SPSS 20.0. Pearson correlation coefficients were calculated to determine the relationship between soil pH, organic matter and soil heavy metal contents. Principal Component Analysis (PCA) was carried out using XLSTAT 2014. Principal component analysis (PCA) is the most common multivariate statistical method used in environmental studies and is employed to extract a small number of latent factors for analyzing relationships among the observed variables [16].

### Soil analysis

The soil samples were subjected to physico-chemical analysis by standard methods. Soil pH was measured in 1:5 soil-water suspensions with a digital pH meter (Elico 101E) respectively. The SOC was determined by using Walkey-Black method [14]. In this method, 1 g of soil was oxidised with  $\text{K}_2\text{Cr}_2\text{O}_7$  (SD Fine Chemicals) and concentrated  $\text{H}_2\text{SO}_4$  (Fisher scientific), the mixture was diluted with 200 ml of distilled water. To this, 10 ml of 85 %  $\text{H}_3\text{PO}_4$  (RANKEM 88%) and 1 ml of diphenylamine indicator (CDH 98%) were added and the solution was titrated with 0.5 N ferrous ammonium sulphate (Fisher Scientific 98.5%) aqueous solution. The SOC was calculated from the following relation [15].

### Pollution index

The pollution Index (PI) is obtained from  $\text{PI} = C_i / C_{\text{ref}}$  (where  $C_i$  is the mean concentration obtained by averaging values measured and  $C_{\text{ref}}$  is the reference value for the studied parameter) [17]. Based on PI values, the samples were classified into three pollution intensity levels:  $\text{CF} < 1$  (low contamination),  $1 < \text{CF} < 3$  (moderate contamination),  $3 < \text{CF} < 6$  (considerable contamination) and  $\text{CF} > 6$  (very high contamination) [18].

## RESULTS AND DISCUSSION

### Soil pH and organic matter content

The descriptive statistics of pH and organic matter content of 25 agricultural soil samples are shown in Table 2. The mean values of SOC (%) for the six seasons are in the order of W1 (1.24) > S1 > S3 > W3 > W2 > S2 (0.95). The soil pH was from 5.26 (S2) to 4.95 (S1), i.e. the soil was acidic in nature. CVs of SOC and pH vary in the range of 0.32- 0.42 and 0.07- 0.45. The statistical distribution of the SOC contents in the study area are negatively skewed except S3 (with skewness values S3 = 0.97), similarly S3 has the positive kurtosis and other five seasons showing negative values. Positive skewness values observed for pH content (except S2) while S1 and W2 seasons give the positive kurtosis values and the others give negative values. The Kolmogorov-Smirnov (K-Sp) test for conformance to a normal distribution of pH and SOC (%) for all six seasons are normally distributed (K-Sp normality test at a significance level of higher than 0.05). The season to season standard deviation values are small for both pH (0.36 to 0.45) and SOC (%) (0.32 to 0.45).

**Table-2: Descriptive statistics of Soil organic carbon (percentage) and pH of the tilled soil (n=25) in different seasons. Note: S1, S2, S3, summer season; W1, W2, W3, winter season; n: no of samples; Min: minimum; Max: maximum; Skew: skewness, Kurt: kurtosis SD: standard deviation; CV: coefficient of variation; K-Sp: significance level of Kolmogorov-Smirnov test for normality.**

	S1	W1	S2	W2	S3	W3
SOC (%)						
Mean	1.07	1.24	0.95	1.04	1.14	1.15
Max	1.99	1.98	1.48	1.65	2.50	2.10
Min	0.24	0.33	0.29	0.24	0.48	0.24
Median	1.22	1.25	0.98	1.09	1.21	1.29
Skew	-0.02	-0.34	-0.44	-0.37	0.97	-0.17
Kurto	-0.58	-0.58	-0.72	-0.08	2.49	-0.37
SD	0.45	0.44	0.32	0.33	0.44	0.44
CV	0.42	0.35	0.34	0.32	0.39	0.38
K-sp	0.13	0.19	0.20	0.20	0.20	0.20
pH (1:5 suspension)						
Mean	5.08	4.95	5.26	5.04	5.09	5.25
Max	5.88	5.92	5.86	5.92	5.78	6.01
Min	4.66	4.23	4.48	4.02	4.25	4.58
Median	4.98	4.92	5.23	5.00	5.08	5.25
Skew	0.99	0.53	-0.21	0.08	0.01	0.15
Kurto	0.06	-0.16	-0.68	0.03	-0.70	-0.34
SD	0.37	0.44	0.36	0.45	0.43	0.36
CV	0.07	0.09	0.07	0.09	0.08	0.07
K-sp	0.18	.200	.200	.200	.200	.200

#### Descriptive statistics of Cu, Fe, Mn, Ni, Zn, concentrations in agricultural soil

The concentrations of Cu, Fe, Mn, Ni, Zn in agricultural soil for the six seasons are summarized in Table 3. For Cu contents of the soil samples, the mean values of the six seasons vary as S1 (55.44 mg/kg) > W3 > W1 > S3 > S2 > W2 (48.09 mg/kg). The standard deviation is from 12.72 (S2) to 36.08 (S1) and the CVs of Cu vary in the range of 0.25 – 0.43. The values are positively skewed (except for S2 and S3) with negative kurtosis values.

The mean values of Fe for the six seasons are in the order of W1 (3708.16 mg/kg) > W2 > S3 > S2 > W3 > S1 (3621.04 mg/kg). The standard deviation was from 136.74 to 358.54 i.e. wide range of Fe content was observed for different seasons in the soil. CVs of Fe vary in the range of 0.04-0.10. The statistical distribution of Fe concentration is positively skewed except for S1 and W1. The kurtosis values are positive for S1, W1, and W3 and negative for S2, W2, S3. The Kolmogorov–Smirnov (K-Sp) test for conformance to a normal distribution is observed only for seasons S1 (K-Sp= 0.09) and S2 (K-Sp= 0.11).

Mn mean values of the six seasons are in the order of W1 (304.04 mg/kg) > S1 > S3 > W3 > W2 > S2 (229.68 mg/kg). The standard deviation is from 69.60 to 116.89. CVs of Mn vary in the range of 0.24 –

0.42. The distribution of Mn is positively skewed except for W1 and S3 and the kurtosis values are positive for all seasons. The Kolmogorov–Smirnov (K-Sp) test for conformance to a normal distribution is valid for seasons W1 (K-Sp = 0.2) and W2 (K-Sp = 0.2) and S3 (K-Sp = 0.2) (K-Sp normality test is at a significance level of higher than 0.05).

Ni mean values of the six seasons are from S1 (93.33 mg/kg) > W1 > S3 > W3 > S2 > W2 (65.21 mg/kg). CVs of Ni are in the range of 0.32 – 0.41. The standard deviation of the values are from 22.24 (W3) to 36.08 (S1). The distribution of Ni concentration is positively skewed and the kurtosis values are positive (except W2, W3). The Kolmogorov–Smirnov (K-Sp) test showed normal distribution for seasons W1, S2, S3 and W3 (K-Sp normality test at a significance level of higher than 0.05).

The mean values of Zn for the six seasons are in the order of W1 (38.60 mg/kg) > S3 > S1 > W3 > W2 > S2. The values have standard deviation from 8.62 (S2) to 11.77 (S3). CVs are in the range of 0.25 – 0.35. The statistical distribution of Zn contents is positively skewed and the kurtosis values are also positive (except S1, W1). The Kolmogorov–Smirnov (K-Sp) test showed normal distribution of the values for seasons S1, W1, S2.

**Table-3: Descriptive statistics of Cu, Fe, Mn, Ni, Zn (mg/kg) (n=25) in different seasons**

	S1	W1	S2	W2	S3	W3
<b>Cu (mg/kg)</b>						
Mean	55.44	52.00	50.95	48.09	51.30	53.44
Max	89.25	82.32	78.23	78.25	75.38	89.80
Min	25.44	26.24	26.31	20.13	23.17	25.21
Median	56.54	52.48	52.85	48.14	54.34	53.16
Skew	0.03	0.26	-0.23	0.07	-0.43	0.26
Kurto	-1.69	-0.97	-0.20	-0.37	-0.77	-0.19
SD	23.72	17.21	12.79	14.11	15.10	16.33
CV	0.43	0.33	0.25	0.29	0.29	0.31
K-sp	.047	0.2	0.2	0.2	0.2	0.2
<b>Fe (mg/kg)</b>						
Mean	3621.04	3708.16	3635.88	3702.60	3665.88	3633.72
Max	4235.00	4046.00	3988.00	4093.00	3989.00	3952.00
Min	2832.00	2852.00	3422.00	3521.00	3508.00	3510.00
Median	3715.00	3798.00	3622.00	3625.00	3554.00	3614.00
Skew	-0.85	-1.38	0.65	0.70	0.69	1.13
Kurto	0.22	1.46	-0.67	-0.41	-1.10	0.23
SD	358.54	313.37	170.04	160.00	168.46	136.74
CV	0.10	0.08	0.05	0.04	0.05	0.04
K-sp	0.09	0.02	0.11	0.01	0.00	0.00
<b>Mn (mg/kg)</b>						
Mean	302.20	304.04	229.68	270.68	289.12	279.76
Max	453.00	445.00	425.00	458.00	448.00	468.00
Min	138.00	181.00	153.00	129.00	168.00	132.00
Median	286.00	306.00	187.00	266.00	306.00	256.00
Skew	0.20	-0.04	0.99	0.24	-0.05	0.27
Kurto	-1.15	-1.10	-0.25	-0.90	-0.22	-1.58
SD	92.97	78.57	82.58	93.87	69.60	116.89
CV	0.31	0.26	0.36	0.35	0.24	0.42
K-sp	.126	.200*	.004	.200*	.200*	.019
<b>Ni (mg/kg)</b>						
Mean	93.33	81.56	67.79	65.21	74.05	69.89
Max	193.00	185.20	136.50	120.20	168.00	115.10
Min	28.60	50.30	32.40	28.30	48.10	25.80
Median	76.60	77.90	66.40	56.40	68.30	65.30
Skew	0.79	1.76	0.73	0.83	2.12	0.12
Kurto	0.87	4.36	0.18	-0.11	6.49	-0.26
SD	36.08	30.58	27.40	27.03	25.82	22.24
CV	0.39	0.37	0.4	0.41	0.35	0.32
K-sp	.012	.133	0.2	.004	.105	0.2
<b>Zn (mg/kg)</b>						
Mean	36.08	38.60	30.07	31.51	36.91	34.06
Max	59.60	54.00	55.30	55.40	69.30	55.30
Min	11.20	24.50	17.80	10.50	22.60	12.50
Median	34.90	37.80	28.30	30.50	32.50	31.20
Skew	0.15	0.22	1.75	0.44	1.06	0.47
Kurto	-0.50	-1.39	3.63	0.52	0.78	0.03
SD	11.67	9.60	8.62	11.00	11.77	10.38
CV	0.32	0.25	0.29	0.35	0.32	0.30
K-sp	0.19	0.11	0.06	0.04	0.00	0.07

**Effects of soil pH and SOC on the metal contents**

The interactions of pH and SOC with Cu, Fe, Mn, Ni, Zn for the different seasons are shown in Fig. 2. Pearson correlation study is used to establish a

relationship among the concentration of heavy metals and the two parameters (Table 4). SOC has good positive correlation with Zn (86%) and Mn (80%) but poor positive correlation with Fe (41%), Cu (27%) and

Ni (32%). Significant positive correlations suggest that the soil organic matter has helped in the accumulation

of Zn and Mn in the soil.

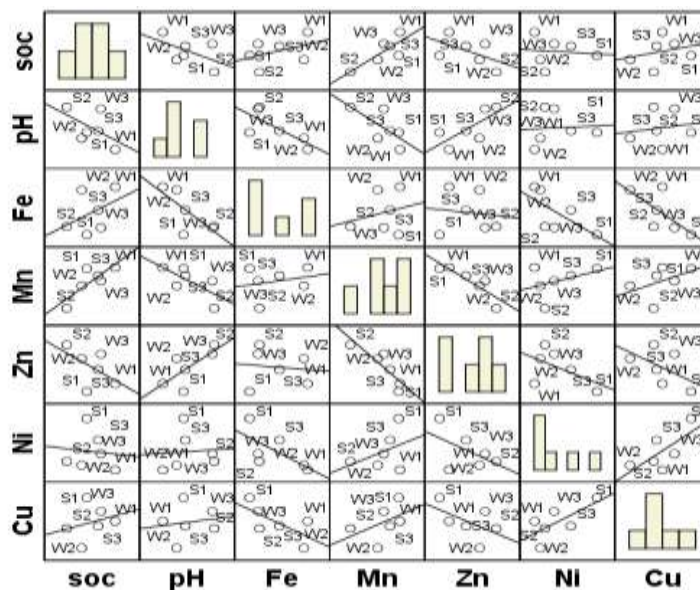


Fig.2. Scatter matrix of mean concentrations of SOC (%), pH, Cu, Fe, Mn, Ni, Zn (mg/kg) for the seasons S1, W1, S2, W2, S3 and W3 (no of sampling points, n=25)

Table-4: Pearson correlation between soil organic carbon SOC, pH, Cu, Fe, Mn, Ni, and Zn

Variables	SOC	pH	Fe	Mn	Zn	Ni	Cu
SOC	1						
pH	-0.5262	1					
Fe	0.4124	-0.7449	1				
Mn	0.8022	-0.6965	0.2296	1			
Zn	0.8677	-0.6293	0.2300	0.9026	1		
Ni	0.3245	-0.4197	-0.2625	0.7012	0.6779	1	
Cu	0.2714	0.1570	-0.6851	0.4655	0.4792	0.7862	1

Among the metals, strong positive correlation observed between Mn and Zn (+0.90). The presence of these metals in the soil is thus interrelated strongly with each other. Comparatively strong correlation is seen between Fe and Cu (-0.69), Mn and Ni (0.70), Zn and Ni (0.68), and Ni and Cu (0.78). Mn oxides in soil have a large capacity for sorption or co-precipitation with heavy metals such as Ni. The correlation among the five trace metals indicates that their enrichment in the soil has originated from a few common sources as observed in other similar work [19].

**Principal component analysis**

Table 5 shows relationships among the metals, SOC and pH. There are positive and negative associations between soil parameters. Fig. 2 shows the trends of Cu, Fe, Mn, Ni, Zn, Ni, Cu and pH, SOC. The calculated factor loadings, together with cumulative percentage and variability percentages, explained by each factor, are listed in Table 5. The values show that

the cumulative percentage is highest for PC3, followed by that of PC2 and then PC1 while the variability percentage is in the opposite order. The parameters SOC, pH, Cu, Fe, Mn, Ni, Zn can be identified with the PC1.

The first three component factors are able to account for 97.74% of the variance of all variables. PCA1 shows high positive loadings with SOC (+0.84), Mn (+0.97), Zn (+0.96) and Ni (+0.75) and moderately positive loadings with Cu (+0.48) and weak positive loading with pH (-0.73) and it explains 56.61% variance. PCA2 shows high positive loadings with Fe (+0.95) and SOC (+0.20). High negative loading is shown with Cu (-0.87) and moderately negative loading with pH (-0.56) and Ni (-0.51), and weak loading with Zn (-0.02), Mn (-0.01) with 32.54% variance. PCA3 shows moderate and weak positive loadings with SOC (+0.50), pH (+0.39), Mn (+0.10), Zn (+0.15) and weak negative loading with Ni (-0.41), Fe (-0.033).

**Table-5: Results of principal component analysis (PCA)**

Element	PC1	PC2	PC3
Variability (%)	56.61	32.54	8.58
Cumulative (%)	56.61	89.15	97.74
SOC	0.84	0.20	0.50
pH	-0.73	-0.56	0.39
Cu	0.48	-0.87	0.06
Fe	0.27	0.95	-0.03
Mn	0.97	-0.01	0.01
Ni	0.75	-0.51	-0.41
Zn	0.96	-0.02	0.15

**Pollution index**

The pollution indices, PI for each metal at different seasons are presented in Table 6. The reference values taken for Fe, Mn, Zn, Ni, and Cu were 38, 000, 488, 70, 29 and 38.9 mg/kg, respectively [5, 20, 21]. The PI values of Fe, Mn, Zn are indicative of uncontaminated state ( $PI < 1$ ). PI values are different for each season but the differences are not so distinct.

However, the soil is moderately polluted with respect to Ni and Cu ( $1 \leq PI < 3$ ) for all seasons but concentration of Ni lies considerable pollution level ( $3 \leq PI < 6$ ) in season S1. The higher PI observed in the rainy season can be traced to rainfall discharge and dispersion of metals under tropical conditions where soils are scarcely vegetated and the subsequent severe erosions due to runoff and landscape topography [22].

**Table-6: Pollution indices (PI) for Cu, Fe, Mn, Ni, Zn at different seasons**

Metal	S1	W1	S2	W2	S3	W3
Cu	1.425	1.336	1.309	1.236	1.318	1.373
Fe	0.095	0.098	0.096	0.097	0.096	0.096
Mn	0.619	0.623	0.471	0.555	0.592	0.573
Ni	3.220	2.890	2.340	2.250	2.350	2.410
Zn	0.515	0.551	0.429	0.450	0.527	0.486

**CONCLUSION**

This work has been able to show the pattern of behaviour of soil organic carbon in agricultural field vis-a-vis pH and five important metals. The results are interpreted on the basis of detailed basic statistics and are supported with the calculation of correlation indices, principal component analysis and the pollution indices.

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