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Anthropogenic trace metals in Setiu Wetland: Spatial and seasonal distribution and implications for environmental health

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ABSTRACT

The growing urban wastewater volume poses a major global environmental challenge, especially in developing nations where inadequate treatment and discharge impact clean water availability. This study focused on Setiu Wetland, aiming to analyze seasonal and spatial variations of trace metals in particulate form from anthropogenic and pathogenic sources. Surface water samples were collected from multiple stations, measuring physical parameters like salinity, temperature, pH, dissolved oxygen (DO), and dissolved organic carbon (DOC). Samples were filtered using ultrafiltration and analyzed with inductively coupled plasma mass spectrometry (ICP-MS). Correlations between trace metals and other parameters were examined to understand their dynamics in the area. The study found that particulate trace metals were influenced by anthropogenic sources, including municipal, industrial, agricultural, and domestic wastewater discharge. The measured concentrations for Cd, Cu, Pb, Fe, and Zn were 10.9 ± 5.1, 31.8 ± 16.8, 72.5 ± 28.5, 20,206 ± 17,929, and 46,742 ± 30,898 μg/g, respectively, indicating their significant presence. To gain deeper insights, a model incorporating principal component analysis (PCA) analyzed the relationships between trace metals, dissolved oxygen levels, and factors like pH, temperature, salinity, and DOC. Regression techniques predicted Cd, Zn, and Fe concentrations based on salinity, with pH serving as an additional predictor for Cu. Moreover, Pb concentration was influenced by temperature, dissolved oxygen, and dissolved organic carbon. These findings emphasize the importance of regular monitoring and assessment of trace metals in protected tropical areas and wetlands, helping mitigate environmental risks associated with wastewater discharge and preserving these ecosystems.

1. Introduction

The increasing global population, intensive agricultural practices, and rapid industrialization have led to a rise in urban wastewater generation, posing a substantial environmental challenge for humanity [1–3]. Despite recent progress in wastewater treatment, improper disposal of wastewater in developing countries can still harm clean water availability [4–9]. This issue requires urgent attention, as it poses a serious threat to the environment and the well-being of people living in these regions.

In the midst of rapid economic progress in Malaysia, disturbing problem has emerged - the deterioration of water quality to a truly alarming degree. Trace metals are common in the environment, originating from both natural and human sources. Natural processes such as atmospheric deposition, erosion, and mineral weathering, along with human activities such as urbanization, industry, and agriculture contribute to their release [10–13]. Anthropogenic sources such as mining, coal combustion, traffic, failure wastewater treatment and the chemical industry also contribute to the presence of trace metals in the environment [14–16]. Trace metals can be pollutants that contaminate water through both soluble and particulate forms. During transportation, these metals are distributed between the aqueous phase and

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suspended particles in the water column. [17,18]. Moreover, trace metals and other pollutants tend to accumulate in sediment and water column [19,20].

Trace metals are defined as metals that exist in minimal quantities at the molecular level within animal and plant cells and tissue [21]. For instance, cadmium (Cd), zinc (Zn), iron (Fe) copper (Cu), and lead (Pb) are important trace metals [22]. They are often referred to as common environmental pollutants, and their presence in water and biota indicates the existence of natural or anthropogenic sources in the area [23,24]. Trace metals play an essential role in biogeochemistry within the forest and aquatic ecosystems, and they can be trapped within the forest floor through adsorption [25]. However, in recent decades, the level of trace metals in the environment has increased significantly due to human activities and inputs [26]. When present at elevated concentrations or availability, certain trace metals can cause harm to humans and the aquatic environment [27]. Trace metals are not biodegradable like organic pollutants, and they can persist in the environment for an extended period. Over time, trace metals have the potential to undergo transformations and change their forms or speciation, which process allows them to enter the biological food chain, which poses a risk of chronic and acute health effects, even at lower concentrations [28].

The increasing body of evidence suggests that trace metals pose a significant concern for human health due to their potential to bioaccumulate through food chains [29]. Exposure to certain trace metals can have various health effects. Cd is primarily toxic to the kidneys and long-term exposure can lead to kidney dysfunction or even kidney failure, as well Cd can accumulate in bones over time, causing bone demineralization, osteoporosis, and increased fracture risk [30]. Pb exposure is particularly harmful to the developing brains of children and can cause cognitive impairment, learning difficulties and behavioural problems [31]. Excessive iron accumulation in the body can result in a condition called hemochromatosis, leading to liver damage, diabetes, heart problems, and joint pain. Zn intake can cause nausea, vomiting, diarrhea, and abdominal cramps [32]. Metal ions such as Cu^{2+} and Cd^{2+} have been found to have detrimental effects on various organs, including the liver, kidneys, skin, and teeth, even at low concentrations (Cu^{2+}, Cd^{2+}, and Ni^{2+}). Moreover, essential metals like Co^{2+} have been observed to negatively impact the growth of animals and plants [33]. It is important to note that the severity of these health effects can vary depending on the level and duration of exposure to these metals [34].

In the presented study Terengganu belonged to Malysia was considered as the research area. In this study, Terengganu located in Malaysia, which experiences three distinct tropical monsoons: the Southwest Monsoon, Northeast Monsoon, and Inter Monsoon seasons [35]. Previous studies in this region, were mainly focused on heavy metals monitoring in fish or other marine organisms. For instance, [36] by application of inductive coupled plasma-mass spectrometer (ICP-MS) revealed that the Fe concentration in fish samples exceeded the established limit of 0.3 mg/kg. Analysis performed by [37] indicated that, while concentrations of heavy metals in the soil and water samples from the same area were below the permissible levels set by the Malaysian Food Regulation (MFR) and USEPA regulations, the concentration of Fe in bivalves was slightly higher than the set value of 5 ppm according to the World Health Organization (WHO) standards.

There is a link between trace metals and dissolved organic carbon (DOC) in their distribution and movement [38]. Studying this connection, allows us to interpret trace metal distribution patterns and assess their ecological and environmental significance within a broader physico-chemical framework. However, there has been no research conducted on the evaluation of trace metals and DOC in the Setiu Wetland, which is an important aspect as it can provide insights into the dissolved trace metal geochemistry during different seasons.

This study aimed to investigate the dispersion and density of trace metals in the Setiu Wetland in Malaysia, while also exploring potential associations between these trace metals and DOC. Furthermore, the study aimed to assess whether there were seasonal variations in the levels of trace metals and to provide recommendations for effective safeguarding and preservation of the aquatic ecosystem based on the findings. A novel compactional model was introduced to analyze the interrelationship between pH, temperature (T), salinity, dissolved organic carbon (DOC), and concentrations of trace metals/dissolved oxygen. The model employed the principal component analysis (PCA) method to uncover the underlying patterns and connections among these variables. Additionally, salinity-based regression models were developed to predict the levels of Cd, Zn, and Fe, while the predictions for Cu incorporated pH as an additional variable.

2. Methodology

2.1. Study area

The study primarily focused on the Setiu Wetland located in Terengganu, with ten sampling stations (S1-S10) presented in Fig. S1 in the supplementary information (SI). The area was selected due to its exposure to significant water runoff from nearby regions with pathogenic and anthropogenic sources and encompassed a range of ecosystems including an estuary, mangrove, wetland, and lagoon. The sampling took place during both the wet season (October–March) and the dry season (April–September). Table S1 in SI provides descriptions of each sampling station, and water samples were collected on a monthly basis from the surface water at approximately 0.5 m depth during high tide conditions. In-situ measurements of physical parameters such as pH and DO were conducted using a pH meter and YSI Multiparameter model #85.

2.2. Sampling

Once a month, using a Van Dorn sampler, two sets of surface water samples were collected. To minimize biological activity during transport to the laboratory, the bottle sample was rinsed with the water sample and stored in an ice chest at +4 °C. In this study, the measurements of trace metal concentrations were conducted in triplicate.

Upon arrival at the laboratory, the water sample was immediately filtered using a Whatman 0.7 μm pore size glass fiber filter (GF/F) in a Class-100 laminar flow hood on the same day as the sampling to differentiate between particulate and dissolved fractions. The filtrate was then filtered again through a 0.45 μm cellulose acetate membrane filter to obtain another fraction. The particulate form from both filtrations (> 0.7 μm and 0.45–0.7 μm) was used to determine particulate trace metals.

2.3. Chemical analyses

2.3.1. Chelating ion exchange method

To separate trace metals in the water samples, a reconcentration step was necessary due to their low concentration. This was achieved using the chelating ion exchange method with a Chelex-100 resin column [39]. The metals were trapped in the column and subsequently eluted using nitric acid. To determine the optimal elution conditions, a series of flow rate determinations were performed.

To prevent contamination from airborne particulate metals, all Chelex-100 columns were packed in a Class-100 laminar flow hood. 0.50 g of dry Chelex-100 resin, with a mesh size of 100–200 and in sodium form (Bio-Rad Laboratories), was soaked in 2.5 M nitric acid (HNO_3) for five days. Subsequently, the resin was poured meticulously into an empty reservoir, which had a block with a small amount of glass wool at the bottom end to support the resin. Using a peristaltic pump, 50 mL of 2.5 M HNO_3 was flowed through the column at a rate of 2 mL/min to elute any trace metal contamination from the resin. Next, the resin was rinsed with 50 mL of deionized (DI) water to remove any excess nitric acid. Further, to convert the resin to the ammonium form, 50 mL of 2 M ammonia solution (NH_3) was poured through the column. Next, the column was washed with 50 mL of deionized water to eliminate any remaining ammonia. Finally, the column was buffered to pH 6.0 with 1 M ammonium acetate (NH_4OAc) [40].

To determine the optimal flow rate for eluting trace metals from a Chelex-100 column, two types of standards were utilized: a mixture of trace metals standards and certified reference material (CRM). The process involved preparing a solution containing a mixture of trace metals (Cd, Cu, Pb, Zn, and Fe) with a concentration of 50 μg/g, spiking it into 200 mL of selected samples, and adjusting the pH to 6.0 using ammonia solution before passing it through the preconditioned Chelex-100 column. The column was then subjected to both standards (mix trace metals standards and CRM) within a range of 1.0 to 5.0 mL/min and eluted with 2.5 M HNO_3. Finally, the concentrations were determined using ICP-OES.

A 200 mL sample was first adjusted to a pH of 6.0 by adding ammonia solutions. The sample was then passed through a Chelex-100 column with a flow rate by 2 mL/min utilizing a peristaltic pump. Subsequently, the column was subjected to a wash with 25 mL of NH_4OAc to eliminate the saline matrix and then rinsed with 10 mL of deionized water. The 2 M HNO_3 was used to elute the trace metals from the column at a flow rate of 2 mL/min and collected in a 15 mL centrifuge tube. To purify the resin in the column, it was washed with 50 mL of 2.5 M HNO_3 and 50 mL of deionized water, in sequence. Finally, the column was regenerated with a 2 M ammonia solution.

2.3.2. *Size fractionation measurements*

Fig. S2 in SI displays the schematic diagram of the size fractionation analysis. Initially, the water sample was pre-filtered and then passed through a 0.7 μm pore size membrane. Subsequently, it underwent a further filtration step using a 0.45 μm pore size membrane made of cellulose acetate and immediately acidified with ultrapure HNO_3 to preserve its composition. Trace metals of column were then eluted using a flow rate of 2 mL/min and 10 mL of 2 M HNO_3, followed by storage in a 15 mL centrifuge tube before subjecting to analysis using ICP-MS.

2.4. *Particulate trace metals determination*

It used filter papers with pore sizes of 0.7 μm and 0.45 μm to analyze trace metals in particulates. Prior to filtering, we recorded the sample volume passing through the filter paper and the initial weight of the paper. The particulate matter collected on the filter papers was then dried in an oven and weighed multiple times until a constant weight was achieved. The particulate matter's weight was calculated by subtracting the weight of the dried filter from the combined weight of the original filter and the particulate matter. Afterward, we used a Teflon bomb in an oven at 100 °C for 7 h to digest the particulate metals with a mixture of HNO_3 (65 %), HCl (37 %), and HF (40 %) in a ratio of 3:3:1 (v:v:v). After cooling, the digested samples were brought up to a volume of 10 mL in 15 mL centrifuge tubes, and their concentration was measured using ICP-OES, as shown in Fig. S3 in SI.

The seasonal variation of trace metal concentrations at Setiu Wetland was investigated through seven monthly sampling over one year, resulting in a total of 84 data (1 samples × 7 points × 12 months). Sampling was conducted monthly during high tide conditions, ensuring that each sampling station was accessible by boat. The sampling bottles utilized in this study were composed of high-density polyethylene (HDPE). To ensure the removal of any trace metals present on the apparatus' surfaces, all equipment, including pipettes, beakers, and sampling bottles, underwent pre-cleaning by soaking in 10 % HNO_3 for a minimum of three days, followed by rinsing with deionized water. In addition, the glass wool employed to support the Chelex-100 resin in the columns was subjected to a two-hour immersion in concentrated HNO_3, after which it was rinsed with deionized water. This step aimed to eliminate any contaminants within the glass wool.

2.5. *Instrumentation*

Inductively coupled plasma-mass spectrometry (ICP-MS) ICP-MS was utilized for the purpose of quantifying the levels of trace metals (Cd, Cu, Pb, Fe, and Zn) present in a dissolved state. For optimal accuracy, the detection has been limited for the trace metals to be in the range of 0.1 to 100 μg/L. ICP-MS is capable of detecting trace metals even at very low concentrations, ranging from a few parts per trillion (ppt) or higher [41]. Table S2 in SI, displays the operating conditions of the instrument. The ELAN 9000 ICP-MS from Perkin Elmer, USA was used in this study and was calibrated according to the manufacturer's instructions.

2.6. *Quality assurance and control*

The precision of the pre-concentration technique was evaluated in this study, utilizing CASS 5 from the National Research Council, Canada. Samples consisting of artificial seawater spiked with CASS 5 were processed concurrently in the same batch. The concentrations of all samples and certified reference materials (CRMs) were adjusted for any blank readings. Additionally, to account for any possible contamination of trace metals from the surrounding environment, a blank consisting of 200 mL of deionized (DI) water was introduced through the column. The procedures were performed within a laminar flow hood of Class-100. To evaluate the accuracy of the digestion technique, the standard reference material (SRM) 1466a was utilized, which was obtained from the National Institute of Standards and Technology (NIST). To establish the blank for particulate metals, clean filter paper underwent the same digestion procedure as the samples. The concentrations of all samples and CRMs were adjusted for any blank readings.

2.7. *Statistical analysis data*

The statistical analysis of trace metals (Zn, Cu, Pb, Fe, Cd) and DOC in relation to surface water parameters (pH, T, DO) was performed using PCA in R-cran software (Fig. S4) in SI, as reported by [42]. Tukey test was used to prove that changes in heavy metals are seasonal over the annual cycle [43]. A statistical significance level of 0.05 was used for the calculations, when the determined *p*-values with the Tukey test were below, it was assumed that there was no basis for assuming that the mean concentration values in the following months differed. A database of heavy metal measurements was used for the analyses, where seven measurements were taken in each month, resulting in 60 data over the annual cycle.

Cluster analysis (k-means) was employed to identify distinct clusters and determine their variable ranges. To determine the number of clusters, the hierarchical cluster analysis (HCA) method was applied, as described by [44]. HCA separated similar objects from others and gradually combined them into groups until a single cluster was obtained, utilizing linkage rules to determine the distance between the new clusters created. The Euclidean distance function was used, which has been established in previous studies, as confirmed by [45]. The distance between clusters was estimated using Ward's method, which utilizes the analysis of variance approach.

An attempt was made to determine empirical models for calculating trace metals on the basis of Sal, DO, T, pH of general form:

$$y = \alpha_0 + \sum_{i=1}^{j} \alpha_i \cdot x_i + \sum_{i=1}^{n} \alpha_{ii} \cdot x_i^2 + \sum_{i<j}^{n} \sum_{j=2}^{n} \alpha_{ij} \cdot x_i \cdot x_j \tag{1}$$

where: α_0, α_i, α_{ii}, α_{ij} – coefficients estimated by the method of least squares; $x_{i,j}$ – independent variables.

For non-linear models, sensitivity coefficients were calculated by [46,47], using the local sensitivity analysis method:

$$S_{xi} = \frac{\partial y}{\partial x_i} \cdot \frac{x_i}{y(x_i)} = \frac{y(x_{i,0} + \Delta x_i) - y(x_{i,0})}{(x_{i,0} + \Delta x_i) - x_{i,0}} \cdot \frac{x_{i,0}}{y(x_{i,0})} \tag{2}$$

where: Δx_i - the difference in the values of i - these independent variables, the paper assumes:

$$\Delta = \frac{(\boldsymbol{x_{i,0}} + \boldsymbol{\Delta x_i}) - \boldsymbol{x_{i,0}}}{\boldsymbol{x_{i,0}}} = 0.05 \tag{3}$$

where: $x_{i,0}$ - the value of i - this independent variable at point x_0; $y(x_{i,0})$ - the values of the dependent variable at point $x_{i,0}$.

3. Result and discussion

3.1. Physical parameters of surface water distribution

The physical parameters will be discussed in the following order: salinity, temperature, pH, and DO. Table 1 presents the distribution of surface water salinity, which ranged from 6.63 to 26.77 ppt in this study. The minimum and maximum values of salinity were recorded at stations S1 and S3, respectively.

The ANOVA test with two factors showed temporal significant differences ($p < 0.05$) among sampling stations, as shown in Table S3 in SI file. The station with the lowest mean value of salinity was S1 (6.6 ± 5.9 ppt), which was the freshwater end-member station, while the station with the highest mean value of salinity was S3 (26.8 ± 7.9 ppt), which was the seawater end-member station. In general, significantly higher salinity values were observed for most of the stations within the wetland area because the sampling periods were performed during high-water conditions. However, relatively low mean salinity values were recorded for stations S2 and S9, which might be due to the mixing process between the freshwater and seawater end-members. These two stations, S2 and S9, received freshwater input from Setiu and Ular Rivers, respectively, as shown in Fig. 1a.

The mean temperature values for each station in Fig. 1b ranged from 28.4 to 29.8 °C, with no significant variation observed between stations. This trend was also observed in a previous study in the same area by [48]. During the dry season, surface water temperatures were warmer due to increased sunlight, while in the wet season, trace metals rainfall caused a drop in water temperature [49]. [50] also reported a similar observation of increased temperatures after wet seasons in the in coastal areas. Mean pH values for each station ranged from 6.56 to 7.57, with the lowest values recorded at freshwater stations S1 and S2. Four stations, namely S5, S8, S9, and S10, which are situated near significant agricultural and aquaculture activities in the wetland, recorded lower mean pH values, as shown in Fig. 1c. These lower values were attributed to anthropogenic activities, which were considered contributing factors in reducing the pH value. According to [51], the pH of coastal seawater within the wetland area was found to decrease due to freshwater input. Fish contributes to the reduction of pH levels as they produce CO_2 through their gills as a by-product of metabolism. This CO_2 then reacts with water to form H_2CO_3, HCO_3^-, and CO_3^{2-} [51]. The range of DO during the study was 3.06 to 6.65 mg/L, with the minimum and maximum values recorded at stations S10 and S9, respectively. The mean DO value ranged from 4.47 to 5.42 mg/L, with the highest mean DO value recorded at station S3 and the lowest at station S10. Stations S8 and S10, which are located in aquaculture areas, recorded relatively low mean DO values, likely due to waste from fish farming with higher organic matter content [15]. Other stations that recorded low mean DO values were S1, S2, and S9. Stations S1 and S2 were located downstream of Setiu River and received input of domestic waste from Penarik town. While the lower mean DO value in station S9 was probably due to runoff from a palm oil plantation upstream of Ular River (Fig. 1d).

3.2. Distribution of dissolved organic carbon

In Fig. 2, the mean DOC concentration for each station is displayed, ranging from 2185 to 4914 μg/L. The stations with the lowest mean DOC concentration were S3, S4, S5, S6, S7, S8, and S10, ranging from 2185 to 3838 μg/L. In contrast, the highest mean DOC concentration was found downstream of Setiu River (S1, S2) and River Ular (S9), ranging from 4047 to 4914 μg/L. This suggests that the river received a significant amount of organic compounds from tributaries, leading to an increase in the DOC concentration in the water column through the decomposition of these materials [52]. Station S9 is mainly used for palm oil plantation upstream of Ular River, supporting statement that rivers flowing across agricultural areas have much higher DOC content [52]. Additionally, stations S1, S2, and S9 recorded a low mean pH value compared to other stations, ranging from 6.56 to 6.94, indicating slightly acidic water. [53] suggested that the presence of organic compounds such as humic and fulvic acids can be indicated by slightly acidic waters. The presence of organic compounds leads to microbial activity, resulting in oxygen consumption and the release of carbon dioxide. Furthermore, the application of fertilizer at station S9 may have contributed to lowering the pH value [54].

3.3. Seasonal distribution of total trace metals

The present study found that the mean concentration of Cd ranged from 4 to 20 μg/g, with the lowest and highest concentrations recorded at stations S6 (4 μg/g) and S9 (20 μg/g), respectively (Table 2).

Mean Cd concentrations at the freshwater end-member varied between 16 and 20 μg/g, while mean concentrations at the wetland estuary were generally between 4 and 10 μg/g (Fig. 3a). This suggests that particulate Cd originated from the freshwater region and that applying fertilizer in the S9 oil palm plantation increased the Cd content in the topsoil, which then washed into aquatic environments through precipitation or runoff [53].

Compared to other studies conducted in Turkey by [55], the mean particulate Cd concentration in this study was higher, ranging from 0.20 to 3.20 μg/g. [55] suggested that the metal source was due to anthropogenic activities. Wet seasons typically had higher mean concentrations of particulate Cd due to increased rainfall, which can cause Cd to

Table 1
Displays the mean values of physical parameters across the various sampling stations.

Station no	Salinity		Temperature		pH		DO		DOC	
	Average	Variance	Average	Variance	Average	Variance	Average	Variance	Average	Variance
S1	6.63	34.92	28.84	3.71	6.58	0.71	4.95	0.55	4914.27	2659795
S2	12.79	89.34	28.66	2.14	6.55	0.35	4.86	0.82	4790.94	5179757
S3	26.77	61.95	29.65	1.35	7.57	0.34	5.419	0.67	3195.38	2320380
S4	26.02	55.75	29.53	2.00	7.52	0.41	5.28	0.51	2861.06	2379824
S5	25.99	58.97	29.26	2.64	7.31	0.55	5.38	0.75	2185.19	732850.2
S6	25.87	56.21	29.57	2.19	7.53	0.41	5.05	0.49	3444.72	4538540
S7	25.88	68.48	29.55	1.70	7.55	0.38	5.08	0.59	3482.54	5608288
S8	25.98	54.09	29.76	2.77	7.44	0.34	4.69	0.65	3324.33	3432475
S9	12.67	123.31	29.13	7.45	6.94	0.39	4.82	0.57	4046.88	6968370
S10	25.69	56.80	29.92	2.25	7.29	0.31	4.57	1.36	3837.53	3134579

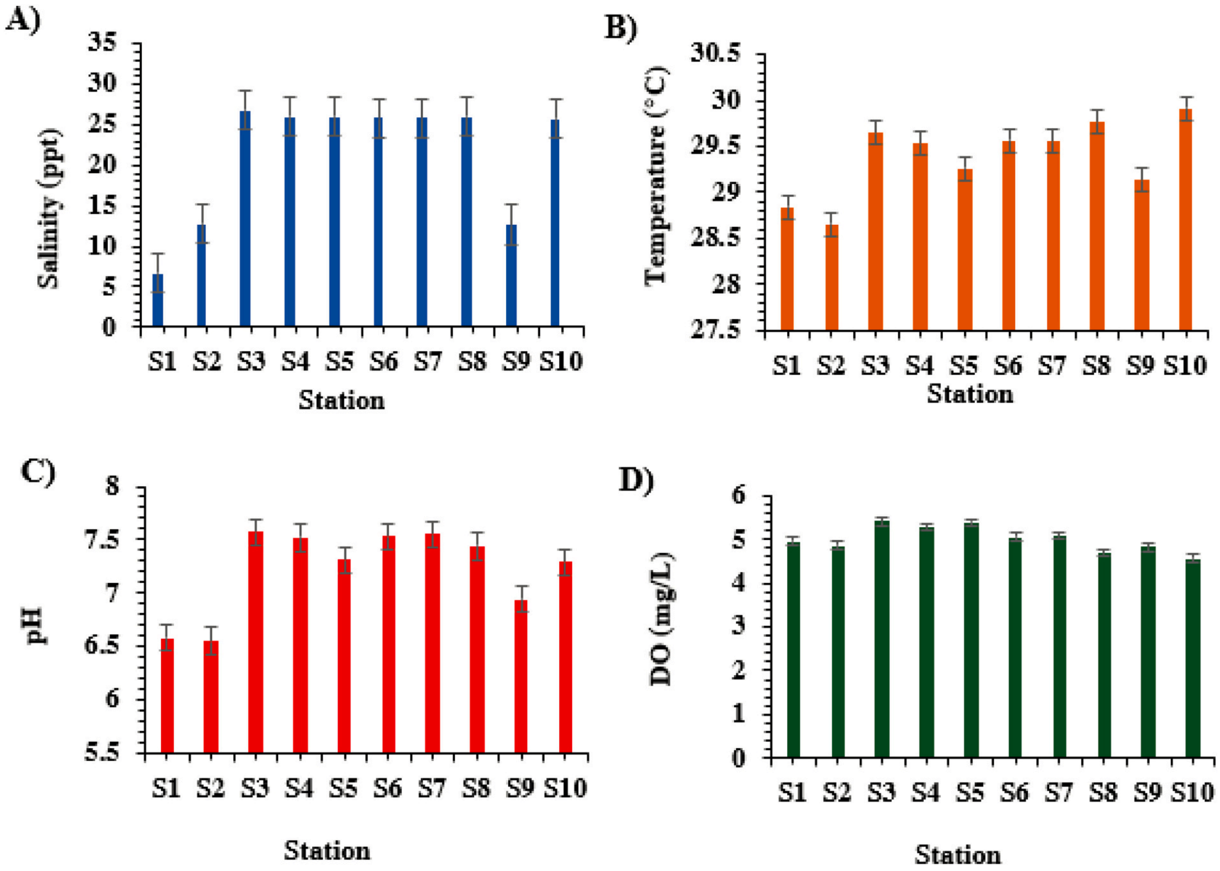

Fig. 1. Displays the average fluctuation of Salinity (a), Temperature (b), pH (c), and DO (d) levels at each Sampling Station.

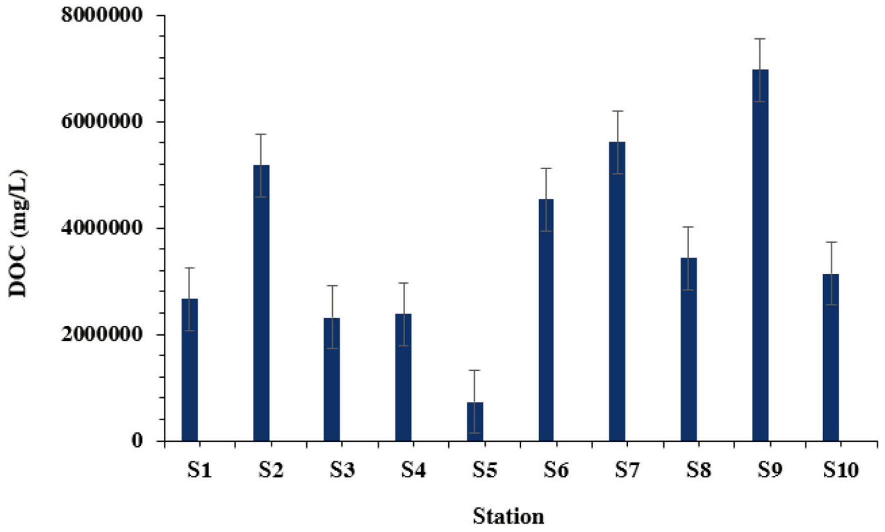

Fig. 2. Shows the average fluctuation of DOC measurements at the Fach Sampling Station.

naturally enter the environment through the erosion of rocks and soils [56].

The mean Cu concentration in the present study ranged from 4 to 60 μg/g, with the lowest and highest concentrations recorded at stations S6 (4 μg/g) and S1 (60 μg/g), respectively (Fig. 3b). The higher particulate Cu concentration in the freshwater at stations S1 and S9 may be due to riverine inputs containing pathogenic particles [57]. An increase in pH can influence the adsorption of metals onto iron and manganese oxides, which could contribute to this situation [57]. Higher mean concentrations of particulate Cu were recorded during wet seasons, likely due to the runoff of substances containing high copper from land and riverine inputs from the Ular and Setiu Rivers.

On the opposite end of the spectrum, particulate Pb concentrations ranged from 35 to 116 μg/g (Fig. 3c), with higher mean concentrations observed in the surrounding wetland areas. This could be attributed to the various activities conducted in the wetlands, such as agriculture, aquaculture, boating, and riverine input. Suspended materials can scavenge trace metals in water. However, these metals can enter natural

Table 2
Displays the mean concentrations and standard deviations (Std. Dev) of trace metals across the various sampling stations

Station No	Cd			Cu			Pb			Fe			Zn		
	Average	Std. Dev.	Variance	Average	Std. Dev.	Variance	Average	Std. Dev.	Variance	Average	Std. Dev.	Variance	Average	Std. Dev.	Variance
S1	17.9	25.4	645.6	59.8	111.7	12482.8	91.9	145.6	21199.3	64935.6	60249.5	3.63E+09	127142.7	137840.5	1.9E+10
S2	15.7	19.8	391.7	23.4	33.5	1119.8	101.1	217.4	47278.8	38229.2	22956.5	5.27E+08	55304.6	69928.5	4.89E+09
S3	10.1	17.5	306.2	26.2	63.1	3982.2	39.3	56.1	3145.2	13426.9	25179.4	6.34E+08	32899.32	56035.7	3.14E+09
S4	6.8	9.8	95.6	19.6	46.4	2150.7	60.3	102.3	10460.9	9263.4	7627.9	58185938	27974.8	42426.4	1.8E+09
S5	8.3	9.5	90.3	55.3	165.7	27444.0	116.0	174.6	30497.9	15257.5	10630.1	1.13E+08	32192.6	35355.3	1.25E+09
S6	4.2	5.8	33.3	4.1	5.3	27.6	35.1	52.4	2743.9	6708.9	8780.5	77096328	15106	15684.4	2.46E+08
S7	6.9	6.1	39.6	29.5	54.2	2936.9	49.0	70.3	4938.1	12433.7	11269.4	1.27E+08	33006.3	28195.7	7.95E+08
S8	10.4	13.9	195.3	23.3	31.6	997.4	100.5	149.4	22323.1	13362.8	12247.4	1.5E+08	51498.1	70427.3	4.96E+09
S9	19.9	29.6	875.2	41.6	78.9	6239.3	74.6	99.3	9868.4	17099.7	15905.9	2.53E+08	52724.7	62048.4	3.85E+09
S10	9.3	13.8	189.9	34.9	87.4	7640.5	57.7	76.9	5914.5	11344.8	10862.8	1.18E+08	39579.9	52153.6	2.72E+09

water bodies through various natural processes such as weathering, hydrothermal activities, and biosynthesis, as well as human activities [58]. Typically, particulate Pb concentrations were higher during the wet season as the rainfall would wash out substances from the soil in the coastal areas and discharge them into the wetlands. This process is similar to that observed by [59] in Santos Bay, Brazil, where higher rainfall led to increased soil leaching and chemical weathering, resulting in greater rainwater runoff.

Similar to other metals, the average concentration of Fe ranged from 6709 to 64,936 μg/g, with the lowest and highest concentrations recorded at stations S6 (6709 μg/g) and S1 (64,936 μg/g), respectively, (Fig. 3d). During the study, stations S1 and S2, located at the Setiu River and upstream of the Penarik town, showed the highest mean concentration of particulate Fe, indicating riverine input. [60] suggested that high mean particulate Fe levels could result from terrestrial inputs, such as anthropogenic activities, rainfall, or weathering of silicate rocks. Seasonal variation of Fe exhibited a similar trend with Cd, Cu, and Pb, with higher mean particulate Fe concentrations during wet seasons. The highest concentration of particulate Fe during wet seasons in this study suggested terrestrial input and atmospheric deposition, similar to the phenomenon observed in Okinawa Islands, Japan, where the study area was far from industrial activities, and air pollution was mainly caused by vehicle emissions, domestic waste incineration, and long-range transportation [61]. During wet seasons, the low mean particulate Fe concentration indicated dilution of the lithogenous component of suspended particulate matter by biogenic particles, resulting in low concentrations of particulate trace metals.

The mean Zn concentration ranged from 15,106 to 127,143 μg/g, with the minimum and maximum concentrations recorded at stations S6 and S1, respectively, as shown in Fig. 3e. The particulate Zn distribution was comparable to other metals such as Cd, Cu, Pb, and Fe. Station S1 had the highest average value, indicating that the majority of particulate Zn came from the upstream of Setiu River. It can be inferred that all the trace metals measured had a common source. A similar observation was made at Reclaimed Tidal Riparian Wetlands in South Estuary, China, where trace metals contamination was due to anthropogenic sources from river water [62]. The seasonal variation of particulate Zn was similar to Fe and other metals, with the highest concentration of particulate Zn recorded during wet seasons.

3.4. *Sesonality trace metals*

The Tukey test calculations revealed that the average levels of trace metals exhibited a seasonal pattern. For the majority of cases, the calculated *p*-values comparing averaged trace metal concentrations (Zn, Cu, Cd, Fe, Pb) between consecutive months were below the significance threshold of $p = 0.05$, as shown in Fig. 4 and Tables S6-S10 in SI file.

Regarding Zn concentrations, the winter months, specifically November to January, exhibited the highest levels, surpassing >100,000 μg/g. These concentrations differed significantly from those observed in the other months, where Zn content tended to be around 20,000 μg/g. Furthermore, a significant relationship was found between the Zn concentrations during these months.

Similar trends were also observed for Pb and Cu concentrations. From November to January, the averaged concentrations of Pb and Cu were the highest, significantly differing from the concentrations observed throughout the rest of the year. During this period, Pb levels exceeded 180 μg/g, while Cu levels surpassed 45 μg/g. These elevated levels indicate a notable increase in both Pb and Cu content during winter months compared to the remaining months of the year, where averaged concentrations stabilized at around 21 μg/g and 10 μg/g respectively.

For Fe concentrations, the highest levels (>20,000 μg/g) and the greatest variability were observed between November and March. In contrast, the Fe concentrations for the remaining months, with the exception of August, showed comparable levels, averaging around

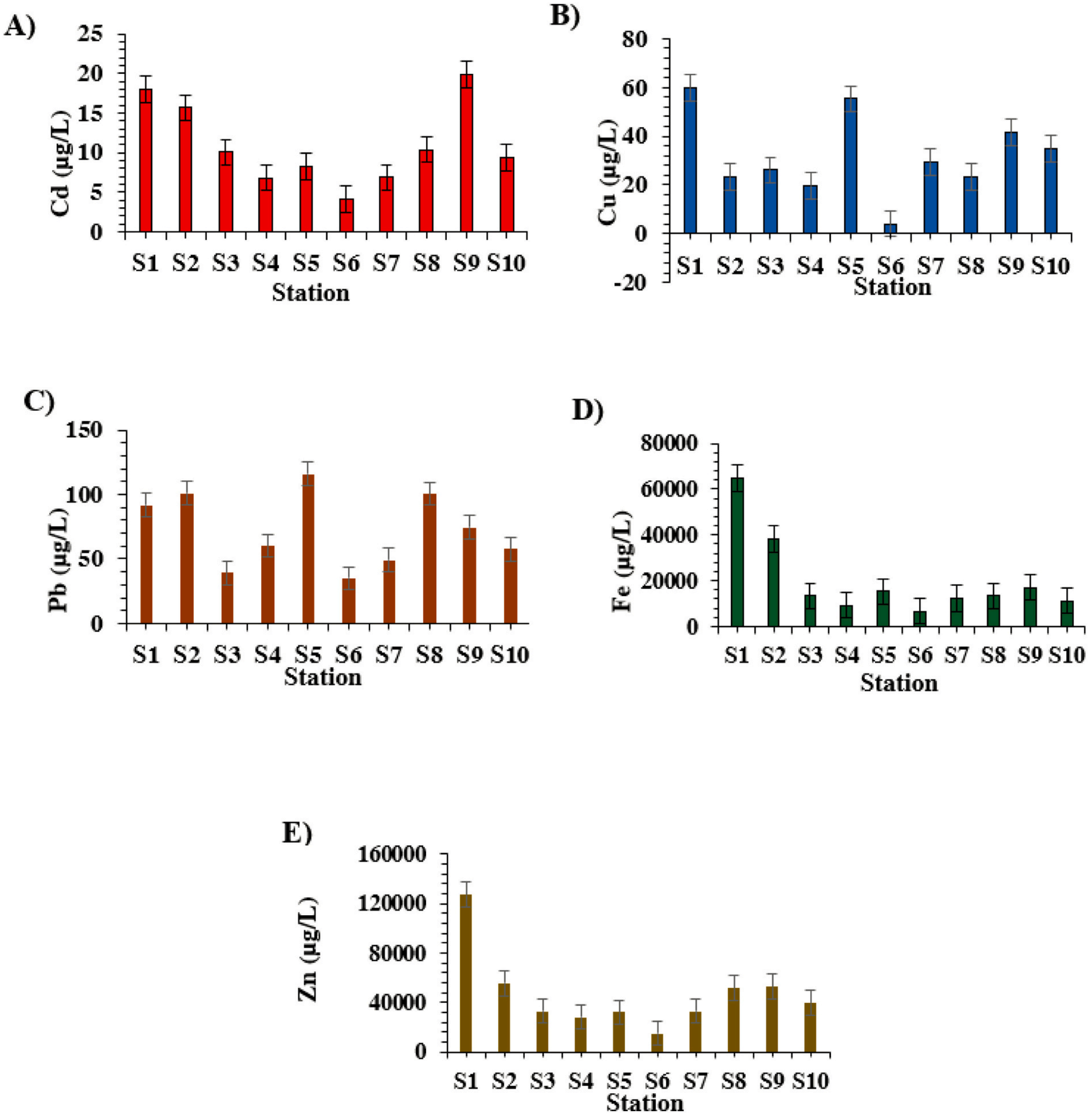

Fig. 3. Displays the fluctuations in the average dissolved concentration of Cd, Cu, Pb, Fe, and Zn across all sampling stations.

10,000 µg/g and displaying a tendency to stabilize.

Cadmium (Cd) exhibited the highest variability in terms of averaged concentrations compared to the other analyzed metals. Similarly, to the other metals, elevated concentrations (>15 µg/g) were detected from November to January. However, an additional peak in Cd levels (>20 µg/g) was observed in August and September. For the remaining months, the averaged Cd concentrations were significantly similar, measuring below 3.5 µg/g. This indicates that Cd levels experienced notable fluctuations throughout the year.

3.5. *Interaction parameters of surface water distribution – trace metal*

Through the use of PCA, it was demonstrated that two components (F1, F2) can describe the variability of parameters that characterize water quality and trace metals. The factor loading for each variable can be found in Table S4 in SI. The F1 component accounts for 64.4 % of the variance in the data, while the F2 component accounts for 14.63 %. Upon analyzing the factor loading values, it was observed that the F1 component describes the variation of trace metals metals in relation to Sal, pH, T, and DOC, while the F2 component illustrates DO. F1 is heavily influenced by Sal, Fe, Cd, Zn, and pH (0.89–0.96), whereas F2 is primarily influenced by DO (0.71). The F1 - F2 configuration of points S1 - S10 (Fig. S5 in SI) reveals that the measurement data can be segregated into 3 clusters: CL1 (S1, S2, S9), CL2 (S3, S4, S6, S7, S8, S10), and CL3 (S5). The lowest concentration of DOC (2187) was observed at point S5, while CL1 exhibited a narrow range of variation in DOC (4046–4914).

Furthermore, the outcomes of the k-means clustering analysis computations (refer to Fig. 5) revealed an inverse relationship between trace metals metal concentrations and the levels of Sal, T, and pH value. You can find the mean values of these variables in Table S5 in SI.

Based on the above analysis results, the following relationships were determined:

$\mathrm{Cd} = -0.61 \cdot \mathrm{Sal} + 24.01\ (R^2 = 0.798).$

$\mathrm{Fe} = -2047.3 \cdot \mathrm{Sal} + 64077.88\ (R^2 = 0.72).$

$\mathrm{Zn} = -3376.9 \cdot \mathrm{Sal} + 119106.4\ (R^2 = 0.70).$

$\mathrm{Cu} = 0.43 \cdot \mathrm{Sal}^2 - 14.71 \cdot \mathrm{Sal} + 1937.50 \cdot \mathrm{pH} - 137.90 \cdot \mathrm{pH}^2 - 6214.7$

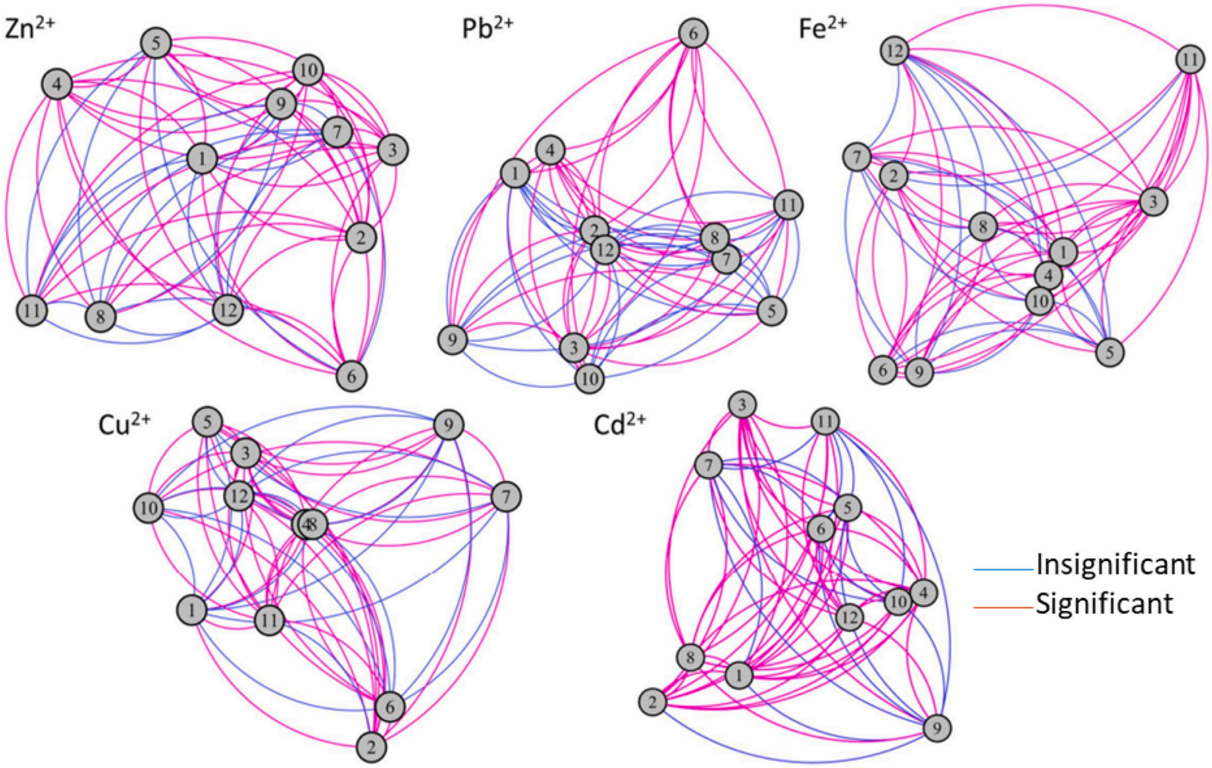

Fig. 4. Networks of relationships between months (indicated by numbers) for which the mean values differ at the assumed confidence level for trace metals (Cu, Cd, Pb, Fe, Cr).

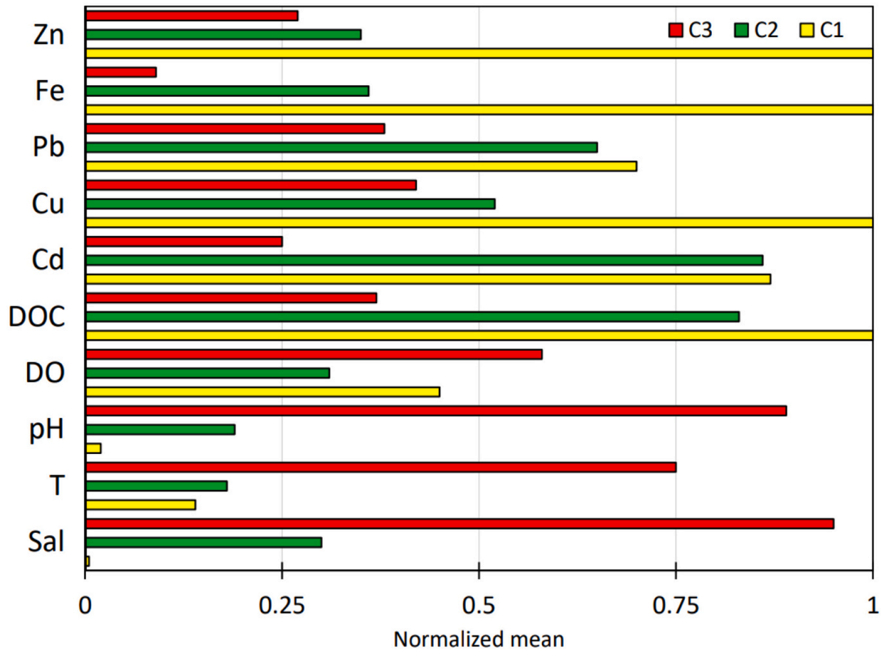

Fig. 5. Displays the projection of the measurement points.

($R^2 = 0.82$).

$Pb = 3248.73 - 87.87 \cdot T - 89.78 \cdot DO - 0.04 \cdot DOC$ ($R^2 = 0.72$).

The calculations performed confirmed the possibility of identifying the concentration of Cd, Fe, Zn from Sal, Cu using Sal and pH, for Pb the dependence on T, DO, DOC was shown. Based on Eq. (2), sensitivity coefficients S_{Sal}, S_{pH} were determined to establish the selected independent variables on Cu concentration (Fig. 6a-d).

The data presented in Fig. 6a indicate that an increase in Sal results in a decrease in DOC, while an increase in temperature T within the range of 28.6–29.0 °C leads to a reduction in DOC (approximately 2 %) to a minimum. However, exceeding a temperature of approximately 29 °C results in an increase in DOC. In addition, the data presented in Fig. 6b demonstrate that an increase in Sal within the range of 6–18 results in a decrease in Cu to a minimum value. However, exceeding a

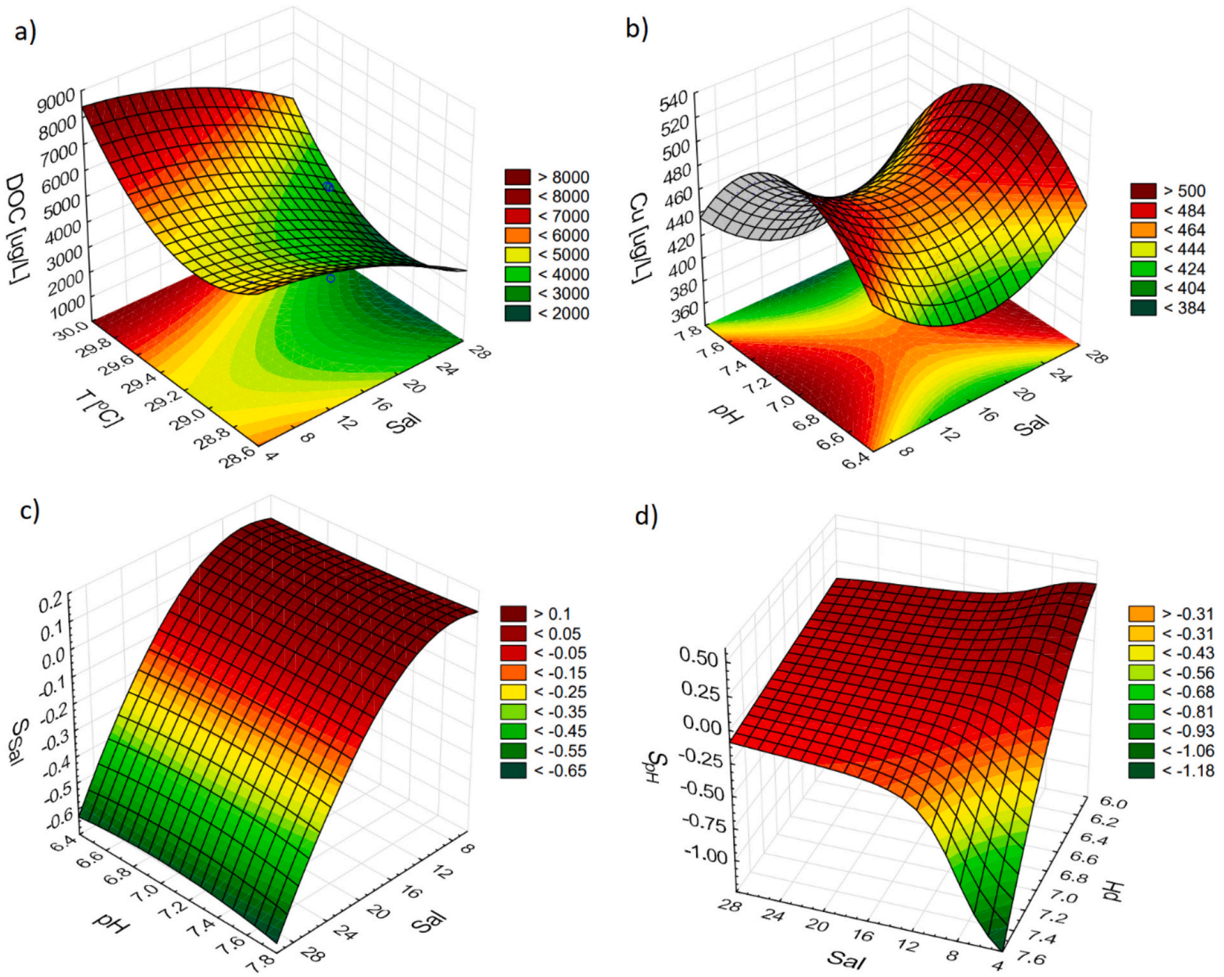

Fig. 6. Depicts the relationships as follows: (a) the dependence of dissolved organic carbon (DOC) on temperature (T) and salinity (Sal), (b) the relationship between copper (Cu) and pH and salinity (Sal), (c) the dependence of dissolved copper (SCu) on salinity (Sal) and pH, and (d) the relationship between pH and salinity (Sal) that gives rise to specific pH values (SpH).

salinity of 18 leads to an increase in Cu, with a similar trend observed for pH value.

To establish the influence of the independent variables (Sal and pH) on Cu concentration, sensitivity coefficients (SSal and SpH) were determined using Eq. (2). The data suggests that there is a correlation between an increase in Sal and an increase in the model's sensitivity to predicting Cu in response to changes in Sal (Fig. 6c). Furthermore, for Sal within the range of 6–14 and pH values >6.8, an increase in the influence of pH on Cu is observed (Fig. 6d).

3.6. *The impact of anthropogenic activities and the significance of the study*

Anthropogenic activities emphasize the importance of sustainable practices, conservation, and adopting cleaner, more efficient technologies to address the challenges of human-caused environmental changes. These activities include domestic sewage discharge, industrial activities related to the palm oil industry, agricultural practices such as the application of fertilizers, and boating activities. Each of these activities can have significant impacts on the surrounding ecosystem and contribute to changes in water quality, habitat degradation, and biodiversity loss [63,64].

The South China Sea stands out as a globally renowned marine ecosystem, characterized by remarkable diversity. It serves as a habitat for an impressive 76 % of the world's coral species and 37 % of reef-fish species [65]. Additionally, this region possesses abundant and valuable natural resources, including fisheries, tourism, as well as substantial reserves of natural gas and oil [66]. These resources hold the potential to generate substantial economic benefits for the neighbouring countries involved. Given the significant rise in fishing and aquaculture activities in the South China Sea and the rapid socioeconomic development of the surrounding regions. It is crucial to acknowledge that such intensive human activities along the Terengganu coast can lead to the discharge of pollutants into the local environment [67]. This poses a serious threat to marine organisms and the delicate balance of the marine ecosystem in the area [68].

This study aimed to investigate trace metal distributions in surface water, spatially and temporally. Water samples were collected at different stations during dry and wet seasons. Physicochemical parameters (e.g., temperature, pH, DO) and DOC were measured to understand water quality comprehensively. Analyzing the distribution of trace metals in particulate forms aimed to enhance our understanding of their behavior in surface water. By studying the dispersion, density, and seasonal fluctuations of trace metals and their relationships with environmental parameters, valuable insights into trace metal dynamics in wetland ecosystems were provided. The findings have implications for future research and river monitoring, aiding in the development of targeted strategies to identify areas or seasons with higher trace metal concentrations. Including physicochemical parameters helps understand factors influencing trace metal distribution and correlations with other water quality indicators. These insights can inform policymakers, healthcare professionals, and researchers in devising effective interventions. The findings of the study can also contribute to global knowledge exchange, fostering collaboration and evidence-based approaches worldwide.

4. Conclusion

The findings of the study conducted on the Setiu Wetland highlight the presence of varying concentrations of trace metals, namely Cd, Cu, Pb, Fe, and Zn. These metals were detected at levels 10.9 ± 5.1; 31.8 ± 16.8, 72.5 ± 28.5, 20,206 ± 17,929 and 46,742 ± 30,898 μg/g, respectively, with their distribution indicating higher concentrations in both the upstream and downstream areas. This suggests a significant contribution from riverine input. The study also revealed a seasonal cycling pattern of particulate trace metals, with higher concentrations observed during wet seasons due to trace metal rainfall. This can bring metals from the land and increase their concentration. Notably, the research identified a positive correlation between DOC and trace metals, with statistical significance ($p < 0.05$). The variability of water quality parameters and trace metals can be effectively captured by using two PCA. The first component of the PCA is heavily influenced by salinity, pH, temperature, dissolved organic carbon, and trace metals, while the second component is primarily influenced by DO. By grouping the measurement data into three clusters, the study observed an inverse relationship between trace metal concentrations and salinity levels, temperature, and pH value. Regression models were developed to forecast the levels of Cd, Zn, and Fe, considering salinity as a parameter. For Cu, pH was included as an additional variable in the models. Furthermore, the models for Pb content were based on the values of temperature, dissolved oxygen, and dissolved organic carbon. These results underscore the importance of continuous monitoring and comprehensive sampling to gain a comprehensive understanding behaviours of trace metals seasonal cycling, and distribution in the Setiu Wetland. Regular sampling is recommended to facilitate proper management and protection of the aquatic environment.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The Metadata used to support the findings of this study have been deposited in the University Malaysia Terengganu repository at http/umt.edu.my/.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jwpe.2023.104172.

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