Relationships between water quality and dissolved metal concentrations in a tropical river under the impacts of land use, incorporating multiple linear regression (MLR)

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Abstract. The water quality and dissolved metal concentrations of the Baleh River were measured at 22 stations over four samplings, in March, June, September and December 2017. It is a tropical river under the influence of numerous land use activities including logging, constructions, plantations and settlements. The selected *in-situ* measured water quality parameters were temperature (Temp), pH, dissolved oxygen (DO), electrical conductivity (EC) and turbidity. The dissolved metals analyzed were aluminum (Al), iron (Fe), manganese (Mn), copper (Cu), zinc (Zn) and mercury (Hg). Multiple linear regression (MLR) was used to model the relationships between water quality and dissolved Al, Fe and Mn. Prominent Fe and Mn were detected with increasing turbidity, implying accelerated surface runoff. Elevated Cu was registered during dry season and a negative correlation was established with Zn. The loadings of Cu and Zn were potentially associated with the brake wear and tire wear, respectively. Temporal variability was identified with consistent correlations established between water quality and metal concentrations. The quadratic model is best fitted with the lowest root mean squares error (RMSE). It was found that the relationships between water quality and dissolved Al and Fe were not well represented with this dataset, because the elements were not detected in most of the samplings, except in September. The considerable zero-clustered data may bias the true correlation. It was found that the pH had a significant effect on the dissolution of Al, Fe and Mn, where Mn^{2+} was ascertained to dissolve in a greater range of pH than Fe^{2+}.
Key Words: inter-metallic relationship, logging, regression model, surface runoff.

Introduction. Tropical rivers in this part of Borneo (Sarawak) form a combined network of 5000 km length. The rivers represent means of transportation for accessing interior areas of Sarawak, supporting the livelihood of locals. The river catchment has been subjected to physical disturbances by continuous land use development. Massive peatlands were cleared for oil palm plantations and riverbank erosion is constantly reported because of degraded vegetation and fluctuant water levels (Hooijer et al 2015; Yuhora et al 2009). The condition of surface runoff is further aggravated by dredging activities and heavy shipping traffic. Staub et al (2000) observes a total sediment input of 24 million MT per year from the drainage basin into the delta of Rajang River.

The Baleh River is a tributary of the Rajang River, with most of its part covered by dense primary forests, the vicinity being sparsely populated. O'Hanlon ventured this part of Borneo in 1984 and he compared his journey with travelling backwards in evolutionary time (Sugnet 1991). In recent years, the vicinity of Baleh has experienced escalated growth and development. Roads, bridges and dam constructions are taking place, along with numerous settlements, plantations and logging activities (RECODA 2017; Sapis et al 2017). Ling et al (2016) revealed that the value of total suspended solids in the river was 5-6 times higher at stations nearby logging. This observation corroborates the elevated iron (Fe), aluminum (Al) and manganese (Mn) concentration values reported by Chai et al (2018) and Sim et al (2016). In light of rapid development, this spurs the need to continuously assess and monitor the environmental conditions of the Baleh River.

It is imperative to understand the metal concentrations in the Baleh River. The development activities could potentially alter the geochemistry cycling of metals, affecting aquatic organisms. High Al, Fe and Mn in river water can be harmful to aquatic organisms. Aluminum deposition in the gills can lead to alteration of the osmoregulation processes (Galar-Martinez et al 2010), can induce oxidative stress (Garcia-Medina et al 2010) and damage mitochondrion (Bondy & Cambell 2000). The accumulation of Al was also reported in freshwater snails at near neutral pH (Elangovan et al 1997). Iron can be deposited on the gill surface when soluble Fe^{2+} is oxidized to insoluble Fe^{3+}. This can cause epithelial damage and the disruption of respiration (Teien et al 2008). The iron bacteria present in water can further colonize the gill surface, oxidizing both Fe^{2+} and Mn^{2+}, aggravating the gill damage (Slaninova et al 2014). The metal speciation is primarily governed by the water chemistry. Hence, this paper reports the relationships between water qualities and dissolved metal concentrations in the Baleh River incorporating multiple linear regression (MLR).

Material and Method

Sampling. Water samples were collected from 22 stations along the Baleh River, as indicated in Figure 1, over four samplings in March 2017 (Trip 1), June 2017 (Trip 2), September 2017 (Trip 3) and December 2017 (Trip 4). Along the river, there are oil palm plantations, settlements, logging with constructions activities for dam access roads. The GPS coordinates and descriptions of the sampling stations are presented in Table 1. 250 mL of water sample was collected from a depth of 20-40 cm, in a polyethylene bottle, filtered through 0.45 μm membrane filter *in-situ* and acidified with 2 mL of concentrated HNO_3 in triplicates, according to the APHA Method 3120 (American Public Health Association 1998). The selected *in-situ* water quality parameters, namely temperature, pH, specific conductivity (EC), dissolved oxygen (DO) and turbidity (Turb) were measured using a YSI 6920 V2-2 Multiparameter Sonde. The samples were then stored in a cooler box at 4°C and transported to the laboratory.
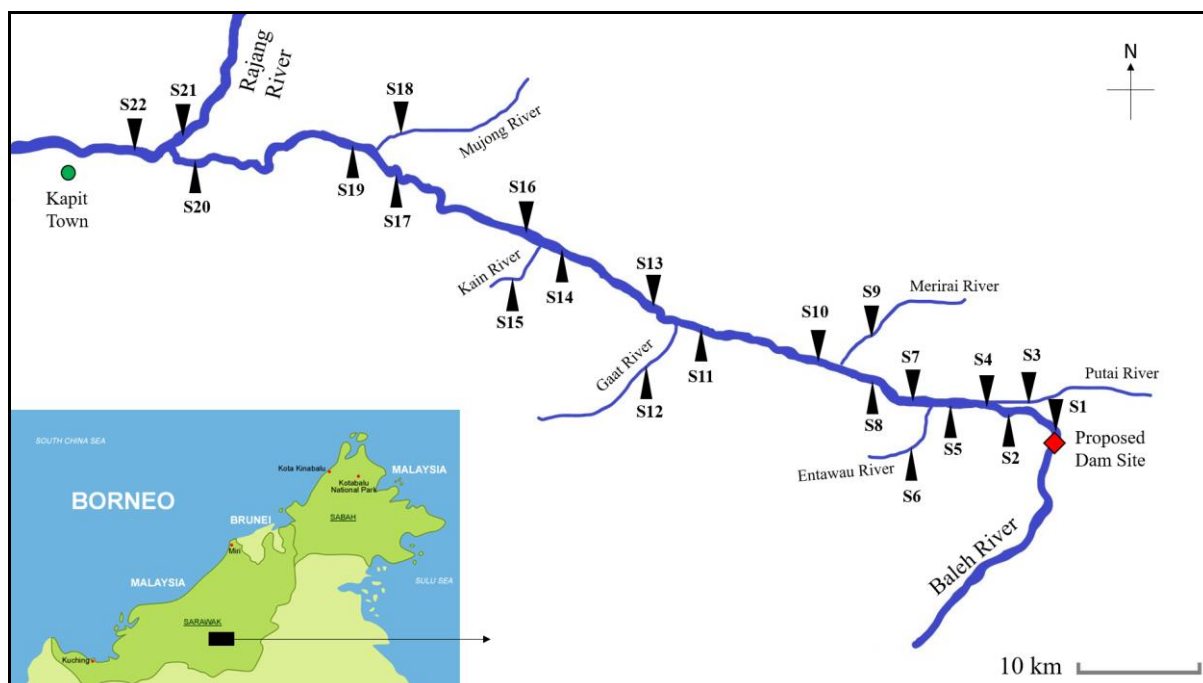

Figure 1. The sampling stations at the Baleh River.

Table 1

GPS coordinates of the sampling stations and their adjacent land use activity

Stations	GPS coordinates	Land use activity
S1	N 1°48'5.7" E 113°46'20.9"	Logging camp and longhouses
S2	N 1°48'47.7" E 113°45'11.5"	Logging camp
S3	N 1°48'51.0" E 113°45'19.6"	Land clearing for Baleh Hydroelectric Dam and road construction
S4	N 1°48'52.9" E 113°44'13.3"	Logging camp and road construction
S5	N 1°49'16.3" E 113°40'57.4"	Longhouses and road construction
S6	N 1°49'15.9" E 113°40'24.7"	Logging
S7	N 1°49'35.9" E 113°39'48.7"	Longhouses and school
S8	N 1°51'05.3" E 113°34'40.9"	Longhouses
S9	N 1°51'36.6" E 113°34'39.8"	Longhouses, logging camp, land clearing for road construction
S10	N 1°51'24.2" E 113°33'22.1"	Longhouses and logging
S11	N 1°53'7.5" E 113°27'24.8"	Logging camp, longhouses
S12	N 1°53'11.2" E 113°26'47.4"	Logging camp, longhouses
S13	N 1°53'32.4" E 113°26'28.9"	Logging camp
S14	N 1°56'52.1" E 113°19'55.6"	-
S15	N 1°56'57.7" E 113°19'20.6"	Longhouses and school
S16	N 1°57'13.7" E 113°18'53.9"	Longhouses and road construction
S17	N 2°1'13.7" E 113°11'2.5"	Longhouses
S18	N 2°1'45.7" E 113°10'46.6"	Logging, longhouses and construction camp
S19	N 2°1'24.6" E 113°9'57"	Logging camp and longhouses
S20	N 2°0'47.1" E 113°1'51"	Logging camp and longhouses
S21	N 2°1'30.5" E 113°2'0"	-
S22	N 2°0'57.1" E 113°1'3.5"	Longhouses, logging camp and land clearing for road construction

Dissolved metals analysis. The filtered samples were subjected to standard analysis protocols with Microwave Plasma – Atomic Emission Spectrometer (Agilent MP-AES 4200) for Cu, Zn, Al, Mn and Fe. Mercury (Hg) was analyzed using the flow injection system (Perkin Elmer, FIMS 400). The limit of detection (LOD) was evaluated based on 21 acid blanks, expressed as LOD=S_{bl}/m, where S_{bl} is the standard deviation of the response and m is the slope of the calibration curve. The LOD for the aforementioned elements are: Al (0.03 mgL^{-1}); Fe (0.003 mgL^{-1}); Mn (0.001 mgL^{-1}); Cu (0.005 mgL^{-1}); Zn (0.04 mgL^{-1}).

Statistical analysis. The water quality data was organized into a matrix of (264 × 5), X. The respective metal concentrations were denoted as *y*. Principal component analysis (PCA) was used to explore the underlying pattern of the metal distributions in water according to sampling trips. Prior to PCA, the data was square rooted and standardized to ensure all variables are approximated to normal distribution and comparable. Pearson's correlation analysis was performed to compute the inter- and intra- relationships of water quality and metal concentrations. The output also serves to suggest the water quality variables significant to the distribution of metals in the Baleh River. Analysis of Variance (ANOVA) was used to determine whether there are any significant differences between the means of independent groups at 95% confidence level, with Tukey's test applied for multiple comparisons. MLR was employed to evaluate the relationships between the independent variables of water quality, X and the dependent variable of metal concentration, *y* (specifically Al, Fe and Mn), according to the following equations:

$$y_{pred} = X \times b$$

$$b = (X' \times X)^{-1} X' \times y$$

Where: b - coefficients; X - matrix of water quality; y - observed metal concentration; y_{pred} - predicted/expected metal concentration.

The regression was fitted according to linear, interaction and quadratic models as follows. The model adequacy was described based on the root mean squares error (RMSE). A smaller RMSE value implies closer difference between the predicted and observed concentration where M is the number of samples.

Linear model:

$$y_{pred} = b_0 + b_1 \times Temp + b_2 \times pH + b_3 \times EC + b_4 \times DO + b_5 \times Turb$$

Interaction model:

$$y_{pred} = b_0 + b_1 \times Temp + b_2 \times pH + b_3 \times EC + b_4 \times DO + b_5 \times Turb + b_6 \times Temp \times pH + b_7 \times Temp \times EC + b_8 \times Temp \times DO + b_9 \times Temp \times Turb + b_{10} \times pH \times EC + b_{11} \times pH \times DO + b_{12} \times pH \times Turb + b_{13} \times EC \times DO + b_{14} \times EC \times Turb + b_{15} \times DO \times Turb$$

Quadratic model:

$$y_{pred} = b_0 + b_1 \times Temp + b_2 \times pH + b_3 \times EC + b_4 \times DO + b_5 \times Turb + b_6 \times Temp \times pH + b_7 \times Temp \times EC + b_8 \times Temp \times DO + b_9 \times Temp \times Turb + b_{10} \times pH \times EC + b_{11} \times pH \times DO + b_{12} \times pH \times Turb + b_{13} \times EC \times DO + b_{14} \times EC \times Turb + b_{15} \times DO \times Turb + b_{16} \times Temp^2 + b_{17} \times pH^2 + b_{18} \times EC^2 + b_{19} \times DO^2 + b_{20} \times Turb^2$$

$$RMSE = \sqrt{[(\Sigma(y - y_{pred})^2)/M]}$$

All the statistical analyses were performed using Matlab R2013a.

Results and Discussion. The concentration range of Al (nd – 0.091 mgL^{-1}), Fe (nd – 0.450 mgL^{-1}), Mn (nd – 0.193 mgL^{-1}), Zn (nd – 0.140 mgL^{-1}), Cu (nd – 0.028 mgL^{-1}) and Hg (nd – 0.399 µgL^{-1}) detected in the Baleh River over four samplings were within the guidelines of the National Standards of Drinking Water Quality (NSDWQ) [Al: 0.2 mgL^{-1}; Fe: 1.0 mgL^{-1}; Mn: 0.2 mgL^{-1}; Cu: 2 mgL^{-1}; Zn: 3 mgL^{-1} and Hg 0.001 mgL^{-1}] (MOH 2000). The metal concentrations were subjected to PCA to examine the underlying clustering pattern. Figure 2 illustrates the scores plot of metal concentrations according to sampling trips. The scores plot indicates temporal variations as samples of Trip 2 and 3 are clearly distinguishable with the former demonstrating positive scores of Principal Component 1 (PC1) whilst the latter contrarily showing negative scores.
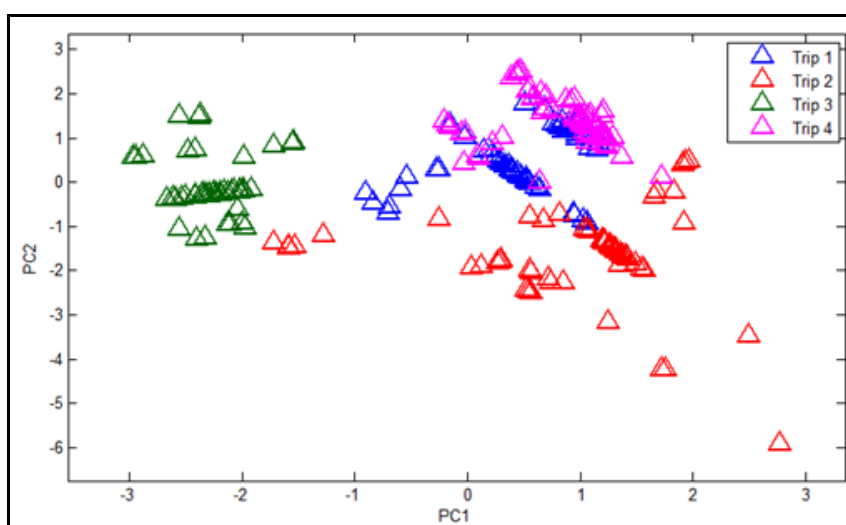

Figure 2. Scores plot of metal concentrations according to four sampling trips.

The metal concentrations are primarily governed by water chemistry. For example, the dissolution of Al and Fe is chemically encouraged under low pH condition. Figure 3 illustrates the metal concentrations across the four samplings and their corresponding water quality (pH, DO and turbidity). Table 2 summarizes the average water quality over the four samplings where temporal variation was highlighted, with statistical differences established.
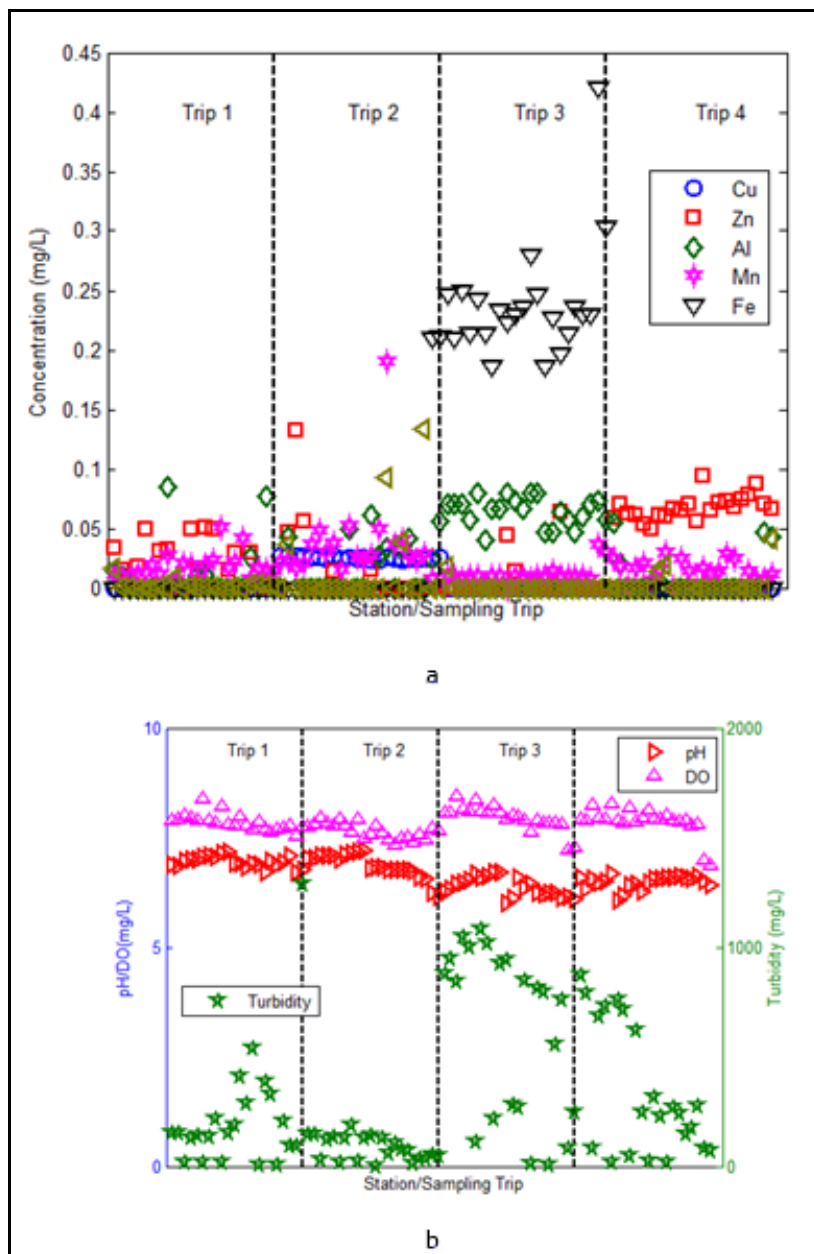

Figure 3. a - the distribution of metals in river over four samplings; b - the corresponding water quality of pH, dissolved oxygen (DO) and turbidity.

Table 2
Average water quality over the four sampling trips

Trip	Temp (°C)	pH	EC (µScm⁻¹)	DO (mgL⁻¹)	Turb (NTU)
1 (Mar)	26.21±0.42[b]	6.95±0.13[d]	38.86±6.45[c]	7.84±0.19[b]	227.38±272.32[b]
2 (June)	27.15±0.48[c]	6.85±0.28[c]	40.47±6.38[c]	7.64±0.18[a]	86.03±53.93[a]
3 (Sep)	25.01±0.73[a]	6.38±0.22[a]	24.65±5.47[a]	7.93±0.29[b]	624.57±379.29[c]
4 (Dec)	25.21±0.61[a]	6.48±0.15[b]	31.22±4.60[b]	7.85±0.33[b]	339.19±292.49[b]

Note: Temp - temperature; EC - electrical conductivity; DO - dissolved oxygen; Turb - turbidity. The same letters in a column indicate no significant difference (P>0.05).

Pearson's analysis reveals significant correlations between water quality and dissolved metal concentrations (P<0.05) (results not shown). This suggests that the water quality parameters, namely Temp, pH, DO, EC and Turb, can be used as independent variables to explain the variability of dissolved metals using MLR. Yao et al (2016) employs linear regression approach to predict five metal concentrations based on turbidity. Table 3 summarizes the R^2 and RMSE based on linear, interaction and quadratic models. As presented, the quadratic model is best fitted with the lowest RMSE and R^2 between 0.60 and 0.83. The lower is the RMSE, the better is the model in describing the relationships between water quality and metal concentrations. The estimated regression coefficients for the quadratic model of are summarized in Table 4 and the response surface plots are illustrated in Figure 4.

Table 3
R^2 and RMSE (root mean squares error) for linear, interaction and quadratic models of water quality and dissolved metal concentrations

Model	R^2/RMSE		
	Al	Mn	Fe
Linear	0.361/0.0245	0.384/0.0177	0.519/0.0762
Interactions	0.575/0.0204	0.639/0.0138	0.809/0.409
Quadratic	0.6/0.02	0.761/0.0113	0.833/0.0463

Table 4
Estimated regression coefficients for quadratic models of Al, Mn and Fe

Term	Estimated regression coefficients		
	Al	Mn	Fe
Intercept	−52.89*	20.19*	−128.12*
Temp	2.44*	− 0.87*	7.53*
pH	− 0.69	−2.30*	−8.06*
EC	−0.09	0.15*	−0.16
DO	6.38*	−0.98	15.17*
Turb	0.001	0.0021*	−0.001
Temp x pH	0.017	0.04*	0.079*
Temp x EC	0.0012	−0.0029*	0.0012
Temp x DO	−0.15*	0.01	−0.39*
Temp x Turb	-3.48×10^{-5}	-4.42×10^{-5}*	-1.54×10^{-5}
pH x EC	0.0051*	−0.0034*	0.032*
pH x DO	−0.0015	0.16*	0.40*
pH x Turb	1.72×10^{-4}*	1.20×10^{-5}	9.07×10^{-4}*
EC x DO	0.0024	−0.0089*	−0.014
EC x Turb	-9.49×10^{-7}	3.44×10^{-6}	-1.56×10^{-5}*
DO x Turb	-1.68×10^{-4}*	-1.27×10^{-4}*	-5.37×10^{-5}*
Temp²	−0.027*	0.012	−0.095*
pH²	0.0023	0.0049	0.10
EC²	7.92×10^{-5}	2.45×10^{-4}*	7.95×10^{-5}
DO²	−0.16*	−0.0078	−0.43*
Turb²	1.69×10^{-8}	-4.21×10^{-8}*	1.33×10^{-7}*

Note: * - P<0.05. Temp - temperature; EC - electrical conductivity; DO - dissolved oxygen; Turb - turbidity.
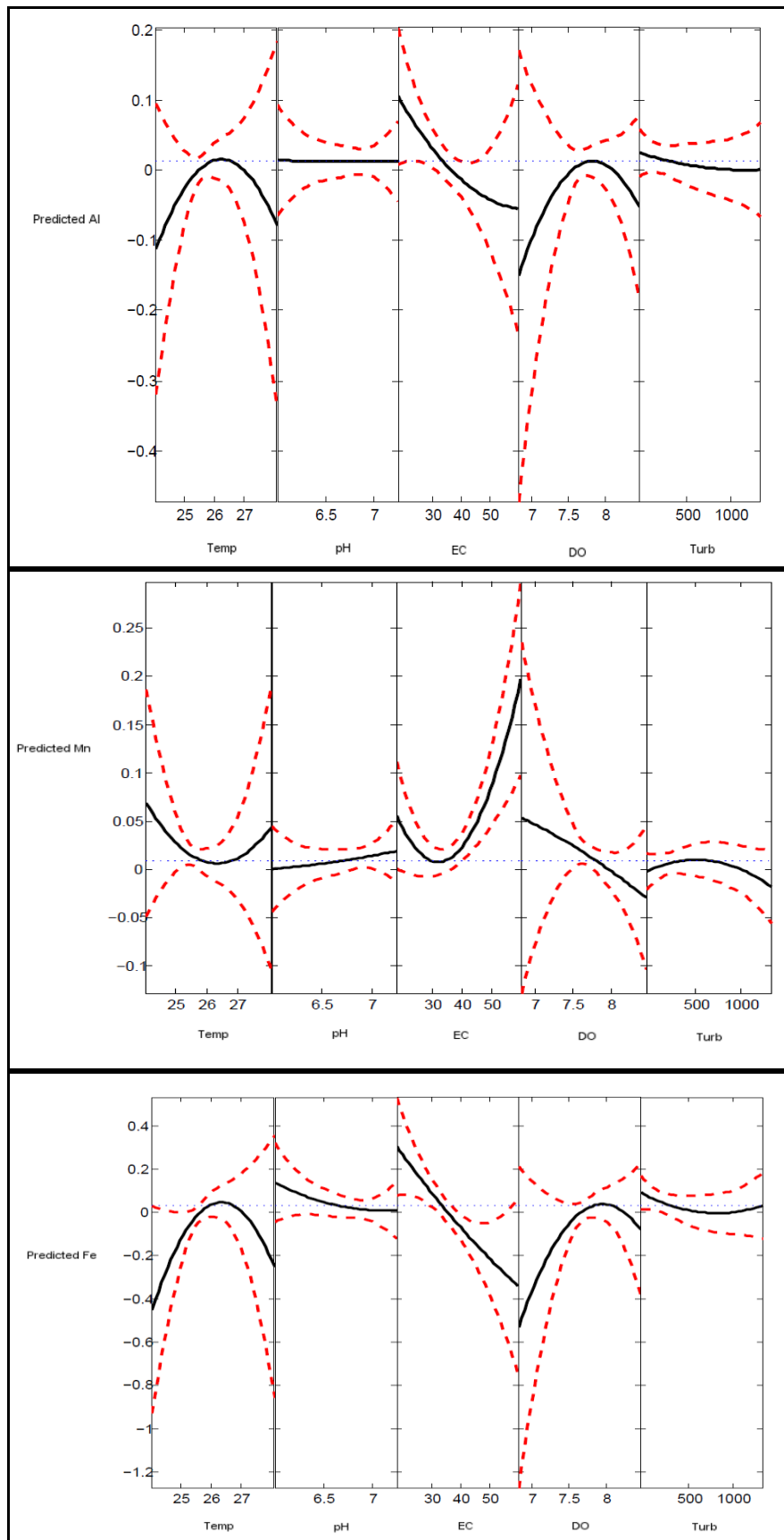
Figure 4. The response surface plots of full quadratic model for Al, Mn and Fe. The dashed lines indicate 95% confidence band for the fitted response.

Mn and Fe were the most abundant elements found in the river water. The predominating presence of Mn and Fe is likely associated with the extensive land clearing activities. According to Bryan et al (2010), approximately 80% of the land area in Sarawak is affected by the logging operations. The suspended solids value in the Baleh River was reported as being 6 to 20 times higher than the normal level (water quality of Class I<25 NTU) (Then 2009). In 2015, water samples were collected from similar stations at S2, S5 and from S13 to S19, and Al, Fe, Mn, Cu and Zn were found below LOD across all stations, except S14 (Chai 2017). In the present study, these elements are consistently detected, suggesting water quality deterioration over time, signifying the impacts of the development activities on the river. The LODs established in both studies (Chai 2017 and current study) are comparable.

Observably, elevated levels of Al and Fe were detected during Trip 3. This is seen to correlate with the increase in turbidities, likely attributed to the increase in stream flow and accelerated surface runoff as a result of heavy rains. According to the daily rainfall data, provided by the Meteorology Department Malaysia, Sarawak, there were several events of heavy precipitation two weeks prior to the sampling. Among the four samplings, Trip 3 was the most rainy month, recording a total monthly rainfall amount of 494.8 mm [Trip 1 (Mar): 324.8 mm; Trip 2 (June): 208.6 mm; Trip 4 (Dec): 425.2 mm].

In June (Trip 2), Cu was seen to increase across all stations whilst Zn predominated in December (Trip 4). Dissolved Cu and Zn were commonly found in highway runoff. They were reported to accumulate and increase in surface runoff after a period of the dry season (Ernst et al 2016). The levels of Cu were associated with wear of bushings and bearings, whilst Zn originates from tire attritions (Amrhein et al 1992). The loadings of Cu and Zn in the Baleh River could have been associated with the on-going road construction activities involving heavy duty vehicles. There is a significant inverse intermetallic relationship between Cu and Zn ($P<0.05$), corroborating the findings of Saleem et al (2014). According to some studies, the dissolution of Cu and Zn is profoundly affected by pH and dissolved organic matter (Malecki et al 2016; Albrecht et al 2011; Mesquita & Carranca 2005). Both soluble Cu^{2+} and Zn^{2+} are the dominant metals at a pH less than 7 and 8.7, respectively. As the pH increases, $Cu(OH)_2$ and $Zn(OH)_2$ will form and precipitate. Seasonal variations of Cu in lake water were evident to follow the alga productivity, where Cu dominates the organic matter complexation over Zn (Xue et al 1995). This competition is believed to have contributed to the dynamics of Cu and Zn in the Baleh River, however, the present available data is insufficient to draw further inference.

The response surface plots depicts similar relationships between Fe and Al, with the five variables of water quality. Chemically, Al and Fe, along with Mn, are known to share similar chemistry in soils and other environments (Barron & Torrent 2013). However, this corresponding intermetallic relationship was not evidenced in Mn. Mn was found to correlate negatively with temperature, opposing the relationships observed in Al and Fe. According to Paufler et al (2018), the release of Mn is largely biologically facilitated and a significant increase in concentration can only be observed at 35°C. Besides temperature, the dynamics of Al, Fe and Mn in water are primarily controlled by pH. Under low pH conditions, the dissolution of these elements is favored, forming Al^{3+}, Fe^{2+} and Mn^{2+}. All these three elements are negatively correlated with pH, as expected.

During the trips in Sep and Dec, the turbidity was high and the pH was relatively low at 6.38 and 6.48, respectively. Under this pH, the release of Al, Fe and Mn were encouraged. Table 5 shows the average metal concentrations over the four samplings. Al and Fe demonstrated significant differences between both trips but not Mn. Al and Fe were almost absent in all the trips, except Trip 3, whilst Mn was consistently detected throughout the samplings. Mn^{2+} naturally exhibits greater stability than Fe^{2+}. It can remain soluble even at a pH above 7, with a wide range of Eh values (Tomassini et al 2014). In turbid river water affected by suspended solids, Al, Fe and Mn are anticipated to dominate. A significant positive linear correlation was established between Mn and EC as well as turbidity. However, this was not identified for Al and Fe. The weak correlation between EC and turbidity with Al and Fe is likely due to the presence of considerable zero-clustered data, which is defined as a group of observations with zero values. Al and

Fe were mostly undetected during Trip 1, 2 and 4. This may lead to the underestimation of the true correlation and the bias could increase with increasing proportion of zeros in a dataset, as evidenced by Huson (2007). For this reason, the relationship of Al and Fe with DO may be unrepresentative, given the substantial zero-value measurements. It is worthwhile to demonstrate how a biased dataset could contribute to erroneous statistical interpretations. The concentration of Mn is inversely correlated with DO as expected; nevertheless, no statistical significant is inferred (P>0.05). Salazar et al (2015) assessed the soluble Mn based on a linear model, revealing that pH, redox potential and conductivity are the best explaining variables, but DO is not. This postulation concurs with the findings of the study.

Table 5

Average metal concentrations over the four sampling trips

| Trip | mgL^{-1} (except Hg in μgL^{-1}) | | | | | |
	Cu	Zn	Al	Mn	Fe	Hg
1 (Mar)	0.00±0.00[a]	0.02±0.02[b]	0.01±0.02[ab]	0.02±0.01[a]	0.00±0.00[a]	0.001±0.006[a]
2 (June)	0.03±0.00[b]	0.01±0.03[ab]	0.02±0.02[b]	0.04±0.04[b]	0.02±0.06[b]	0.014±0.062[a]
3 (Sep)	0.00±0.00[a]	0.01±0.02[a]	0.06±0.01[c]	0.01±0.01[a]	0.24±0.05[c]	0.000±0.007[a]
4 (Dec)	0.00±0.00[a]	0.07±0.01[c]	0.01±0.02[a]	0.01±0.01[a]	0.00±0.00[a]	0.004±0.017[a]

Note: same letters in a column indicate no significant difference (P>0.05).

Conclusions. The metal concentrations of the Baleh River are subjected to temporal variations, with elevated Fe and Al recorded during rainy seasons, whilst Cu was more prominent during the dry season. The dissolved Al, Fe and Mn can be best described with the quadratic model of water quality. Nevertheless, the large zero-clustered data of Al and Fe could lead to erroneous interpretations of the relationships between water quality and metal concentrations. The Baleh River is affected by the land use activities associated with land clearing and road constructions.

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