

# Assessment of the characteristic of nutrients, total metals, and fecal coliform in Sibulaut River, Sarawak, Malaysia

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**Abstract** The concentrations of nutrients (nitrogen and phosphorus), total metals, and fecal coliform (FC) coupling with chlorophyll-*a* (chl-*a*), 5-day biochemical oxygen demand (BOD<sub>5</sub>) and other general environmental parameters were evaluated at the sub-surface and near-bottom water columns of 13 stations in the Sibulaut River during low and high slack waters. The results indicated that inorganic nitrogen (mainly nitrate) was the primary form of nitrogen whereas organic phosphorus was the major form of phosphorus. The abundance of total heavy metals in Sibulaut River and its tributaries was in the order of Pb < Cu < Zn < Cd. Fecal coliform concentration was relatively low along Sibulaut River. The shrimp farm effluents contributed a substantial amount of chl-*a*, BOD<sub>5</sub>, nutrients, and FC to the receiving creek except for total metals. Nevertheless, the influence was merely noticeable in the intake creek and amended rapidly along Selang Sibulaut River and brought minimal effects on the Sibulaut River. Besides, the domestic sewage effluents from villages nearby also contributed a substantial amount of pollutants.

**Keywords** Waterquality·Eutrophication·Heavy metals·  
Fecalcoliform·SibulautRiver

## Introduction

Deterioration in water quality is one of the greatest concerns associated with population growth and accompanying land-use changes (Ribeiro and Kjerfve 2002; Davis and Koop 2006; Ham et al. 2009; Whittall et al. 2010; Hadibarata et al. 2012). Eutrophication, metals contamination, and bacterial pollution are among the major problems and stresses of the marine environment and have been receiving global attention and interest (Malik and Ahmad 2002; Parnell 2003; Jonas and Millward 2010; Reopanichkul et al. 2010; Shin et al. 2012).

Eutrophication can be defined as the process of increasing nutrients of an ecosystem that causes changes to its nutritional status (Nixon 1995). Most frequently, this has usually been characterized by increased supplies of nutrients in particular nitrogen and phosphorus to that system, although the supply of excessive decomposable organic carbon leads to eutrophication as well (Young et al. 1999; Pinckney et al. 2001; Howarth and Marino 2006). The discharge of excessive nutrients from urban and industrial wastewaters, or rural and agricultural runoff contributes to the enrichment of inorganic and organic material in marine waters. Eutrophication can lead to the detrimental changes of the structure and function of both living organisms and non-living components in an ecosystem (Paez-Osuna et al. 1998; Bricker et al. 1999; Smith et al. 1999; Meyer-Reil and Koster 2000; Parnell 2003; Del-Pilar-Ruso et al. 2009; Reopanichkul et al. 2010). Often, the “bloom” or greatly increase of aquatic vegetation or phytoplankton and algal in a water body is the first

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response to the increased levels of nutrients or the eutrophication (Pan and Subba Rao 1997; Smith et al. 1999). Hence, chl-*a* concentration which provides a measure of phytoplankton biomass often used as an indicator of eutrophication and studied along with nutrients (Ho et al. 2008; Xu et al. 2008; Madhu et al. 2010; Lefebvre et al. 2011).

Besides nutrients, excessive amounts of heavy metals have been introduced to estuarine and coastal environments as a result of intense industrial development and anthropogenic activities (Fianko et al. 2007; Nduka and Orisakwe 2009; Sundaray et al. 2012; Zhang et al. 2012; Lacerda et al. 2013). Heavy metals are deemed to be dangerous pollutants and have raised environmental concern due to their toxicity, persistence and non-degradability, bioaccumulation in organisms, and ultimately their effect on human health (Burgos and Rainbow 2001; Kress et al. 2004; Otero et al. 2005; Kurun et al. 2007; Du Laing et al. 2008). Special attentions have been paid to Cd, Cu, Pb, Zn and many other heavy metals.

The discharge of domestic and industrial wastewaters with the presence of pathogenic microorganisms in the adjacent water frequently threatened the microbiological quality of the water (Aslan-Yilmaz et al. 2004; Nduka and Orisakwe 2009; Dorsey et al. 2012). The microbiological risks depend on the bacteria concentration in water and on the water usage such as drinking, recreational activities, irrigation, and shellfish harvesting purposes. The microbiological quality of water is classically monitored by searching for and quantifying bacterial indicators of fecal contamination. Total coliform, fecal coliform, fecal streptococci and enterococci are among the most frequently used microbial indicators of fecal pollution (Malik and Ahmad 2002; Cabral and Marques 2006; Djuikom et al. 2006; Kovačić et al. 2011). Although not usually harmful themselves, their presence may indicate the possibility of pathogen presence (Bonadonna et al. 2002; Dorsey et al. 2012).

Sibu Laut River is surrounded by various villages, shrimp farms and tourism's activities make it vulnerable to human activities and susceptible to water quality degradation. Furthermore, Sibu Laut River, which forms the western boundary of the Kuching Wetlands National Park, a wetland classified as a sensitive area also renders it of high research value. What happened to Sibu Laut River and its tributaries now? Is Sibu Laut River suffering from eutrophication, metals contamination or bacterial pollution? The level and the variability in the water quality over neap–spring tidal cycles in this river are yet to be explored in detail. Hence, physicochemical and major water quality data were collected and were employed to determine the water quality characteristics and current health status of the Sibu Laut River and its tributaries. The data presented here provide a snapshot of hydrodynamic conditions and water

quality of the Sibu Laut River and its tributaries experienced during high and low tides.

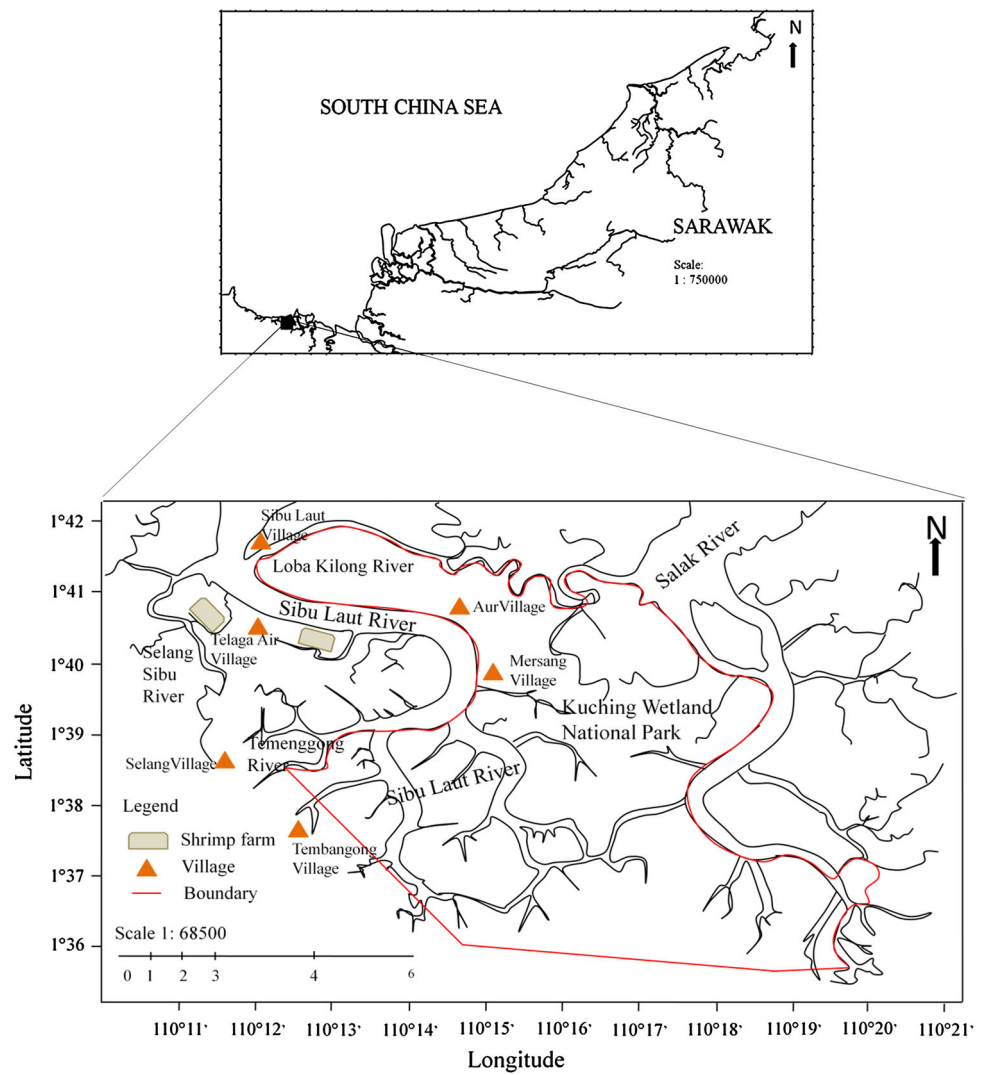
## Materials and methods

### Study area and sampling regime

The Sibu Laut River is in the northwest region of Kuching, Sarawak, Malaysia (Fig. 1). The area experiences a humid tropical climate with an annual rainfall of 3,600–4,000 mm. The region is not directly exposed to the northeast monsoon. Rainfall is the lowest during June and July, and reaches a peak in December and January. Temperature ranges from 19 to 36 °C receives only 5 h of sunshine per day on average (Malaysian Meteorological Department 2012). This river is one of the boundaries of the Kuching Wetlands National Park at which the international importance of the wetland has been recognized when Malaysia designated the park as the RAMSAR site in November 2005 (Ramsar Site No., 1568), which is also the first Ramsar site in Sarawak and the fifth in Malaysia (Wetlands International 2013). The river drains the mountainous region at the upper reaches of the wetland and the mangrove swamp within the delta on the estuary with tidal ranged from 0.0 to 4.7 m. Selang Sibu River and Loba Kilong River are the left and right tributary at the downstream of the Sibu Laut River.

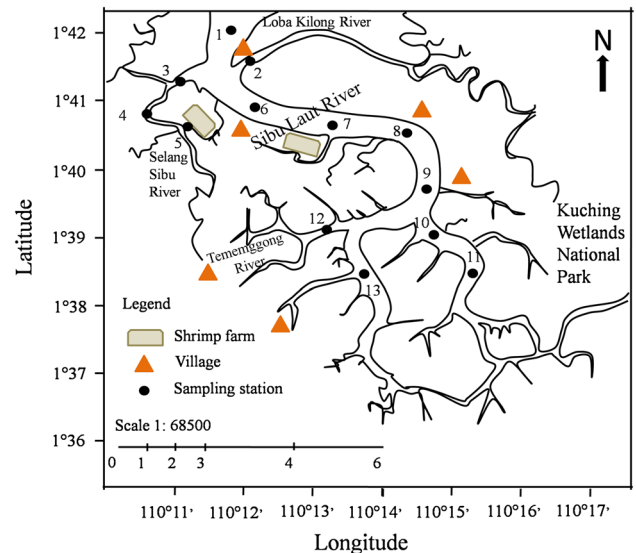
Samplings were divided into two stages. Stage one focused on the eutrophication study while stage two concentrated on heavy metals and bacteriological water quality. Table 1 shows the details of the sampling cruises. Four samplings were conducted to obtain the data of nutrients in 2010 while two samplings were conducted for total heavy metals and bacteriological water quality study in 2011. A total of 13 stations was selected along Sibu Laut River and tributaries (Fig. 2). The total river length studied was approximately 8 km. The locations were chosen mainly due to their proximity to the suspected domestic pollution sources and aquaculture activities; and some relatively unpolluted sites as a control. The distance between stations was ranged from 1 to 2 km. The first station was at the most downstream of the river. Station 2 was in the Loba Kilong River near to the village of Sibu Laut, just before its conjunction with Sibu Laut River. Station 3, 4, and 5 were along the Selang Sibu River. Station 5 was the closest site to the discharge point of a shrimp farm (Sea Horse Marine Life Sdn. Bhd.). These stations are intended to monitor the possible impacts of the discharges of wastewater and the influences to the tributary and main river body. Stations 6, 7, 8, and 9 were along the main river body of Sibu Laut. Station 6 was chosen to observe the possible influences from the surrounding activities of the Telaga Air Town and

**Fig. 1** Study area



**Table 1** Details of the sampling cruises

Sampling Date	Tide	Water column	Parameters	
1st trip	25.2.10	High	Near-bottom	General environmental parameters, nutrients, chl- <i>a</i> , BOD <sub>5</sub>
2nd trip	25.3.10	High	Sub-surface	
3rd trip	29.4.10	Low	Near-bottom	
4th trip	18.5.10	Low	Sub-surface	
5th trip	16.6.11	Low	Sub-surface and Near-bottom	General environmental parameters, total heavy metals, and fecal coliform
6th trip	27.7.11	High	Sub-surface and Near-bottom	



the public jetty. Station 7 was close to another shrimp farm belonging to CCK Pelita Telaga Air Sdn. Bhd. Station 8 and 9 were near to the village of Aur and Mersang, respectively. Station 10 and 11 were at the right upstream

**Fig. 2** Sampling stations along the Sibulaut River and its principal tributaries

while station 13 was at the left upper reach of the Sibulaut River near to Tembangong Village. Station 12 was at the Temenggong River, which is the left upper tributary of the Sibulaut River.

### Sampling techniques

Portable Global Positioning System (GPSMAP® 76CSx, GARMIN) was used to determine the actual coordinates of the sampling stations and to re-confirm the location of the stations during subsequent sampling periods. Hydrological parameters including salinity, pH, temperature, dissolved oxygen (DO), turbidity, and transparency were measured using a refractometer (MR100ATC, Milwaukee), a pH meter with temperature probe (HI 8424, Hanna), a DO meter (HI 9142, Hanna), a turbidity meter (HI 93703, Hanna), and a secchi disc with measuring tape, respectively. Temperature, pH, DO, total dissolved solids (TDS), and conductivity were measured using a multi-parameter water quality meter (PCD650, Eutech) for samplings conducted in stage two samplings in the year 2011.

In stage one sampling for nutrients study, three replicate water samples were taken from the sub-surface and near-bottom water columns using a 2-l Wildco® Van Dorn water sampler. The water sample was taken in the middle of the river with approximately 1 m below the surface water and 1 m above the river bed after confirmed the river width and depth using a rangefinder (Elite 1500, Bushnell) and a depth sounder (PS-7, Hondex). Table 2 summarizes the river width and depth throughout the study period. The concentrations of chlorophyll-*a* (chl-*a*), total suspended solids (TSS), 5-day biochemical oxygen demand (BOD<sub>5</sub>), total nitrogen (TN), ammonium nitrogen (NH<sub>4</sub><sup>+</sup>-N), nitrite

nitrogen (NO<sub>2</sub><sup>-</sup>-N), nitrate nitrogen (NO<sub>3</sub><sup>-</sup>-N), organic nitrogen (Org-N), total phosphorus (TP), inorganic phosphorus (IP), and organic phosphorus (OP) were studied. In the study of total heavy metals and bacteriological water quality, three replicate water samples were also taken from the sub-surface and near-bottom water columns for the determination of TSS, total heavy metals (Zn, Cu, Cd, and Pb) and fecal coliform (FC) bacteria. All sampling bottles were acid-washed, cleaned and dried before use. Sampling bottles for FC bacteria analysis were pre-sterilized with 70 % v/v ethanol before use. Water samples were placed in ice box and transported to the laboratory for immediate analysis (Parsons et al. 1984; APHA 2005).

### Analytical techniques

All analyses were done according to standard procedures for water analysis in triplicate unless otherwise mentioned (Parsons et al. 1984; APHA 2005). Concentrations of chl-*a* were determined from adequate samples filtered through 0.7 µm Whatman GF/F filters and extracted for 24 h using 90 % (v/v) acetone. Five-day biochemical oxygen demand was determined as the difference between initial and 5-day dissolved oxygen content, after incubation of the sample in the dark at 20 °C. Total suspended solids were assayed as the difference between initial and final weight of the 0.7 µm Whatman GF/F filter paper, after filtration of an adequate sample volume and drying at 105 °C.

The filtered water samples were then used to determine nitrogen and phosphorus concentrations. Inorganic phosphorus was determined by the colorimetric ascorbic acid method while TP was determined as IP after potassium persulfate digestion of samples. Organic phosphorus was calculated as the difference between TP and IP. Ammonium nitrogen and NO<sub>2</sub><sup>-</sup>-N were determined by phenate method and colorimetric diazotization method, respectively. Nitrate nitrogen was determined by the ultraviolet spectrophotometric method while TN was determined as NO<sub>3</sub><sup>-</sup>-N after potassium persulfate digestion of the sample. Organic nitrogen was calculated as the difference between TN and inorganic nitrogen (NH<sub>4</sub><sup>+</sup>-N, NO<sub>2</sub><sup>-</sup>-N, and NO<sub>3</sub><sup>-</sup>-N). A five-point calibration curve was constructed for each element. Blank and standard solutions were treated in a similar way as sample.

The unfiltered water samples, acidified to pH < 2 using ultrapure nitric acid (Merck) were used to determine total heavy metals (Zn, Pb, Cu, and Cd) concentrations. Total heavy metals were determined by Flame Atomic Absorption spectrophotometer (iCE 3000 Series AA, Thermo Fisher Scientific) after nitric acid digestion of samples. A three-point calibration curve was constructed for each element. Blank and standard solutions were treated in a similar way as sample. The detection limits for Zn, Pb, Cu,

**Table 2** Summary of the physical characteristic of Sibulaut River pooled from the six samplings in year 2010 and year 2011

Station	Width (m)		Depth (m)	
	Low tide	High tide	Low tide	High tide
1	520.3 ± 67.0	557.3 ± 30.9	17.9 ± 1.5	22.0 ± 2.8
2	176.5 ± 26.3	184.0 ± 30.7	4.9 ± 0.9	7.0 ± 0.6
3	211.3 ± 30.3	241.0 ± 76.5	3.0 ± 1.2	5.3 ± 0.9
4	62.3 ± 9.6	78.9 ± 26.2	8.2 ± 1.7	8.1 ± 1.5
5	13.6 ± 3.6	17.4 ± 0.4	2.0 ± 0.5	3.3 ± 0.5
6	521.8 ± 27.3	517.8 ± 37.9	16.6 ± 0.7	16.7 ± 1.6
7	639.3 ± 20.6	661.6 ± 24.9	13.0 ± 0.6	14.3 ± 0.9
8	527.7 ± 34.1	545.9 ± 18.9	20.8 ± 1.4	23.5 ± 0.8
9	541.1 ± 12.4	549.4 ± 31.1	16.4 ± 1.2	18.3 ± 1.7
10	418.4 ± 5.5	414.1 ± 25.2	12.1 ± 3.4	11.8 ± 3.5
11	308.7 ± 17.8	336.4 ± 55.4	11.8 ± 0.6	11.3 ± 0.9
12	233.5 ± 79.1	240.9 ± 75.8	7.4 ± 1.9	9.0 ± 2.7
13	173.0 ± 113.5	176.8 ± 125.3	8.7 ± 2.6	8.1 ± 0.6

and Cd are 0.0033, 0.013, 0.0045, and 0.0028 mg l<sup>-1</sup>, respectively.

A membrane filtration method was used to enumerate FC bacteria as colony-forming unit per 100 ml of sample, cfu/100 ml. Duplicate water samples were filtered through a 0.45 µm Advantec mixed cellulose ester membrane. Filters were then placed on the m-FC agar (Merck) without rosolic acids. Fecal coliform counts were performed as the number of blue colonies developed on m-FC agar plate after 24 h incubation at 44.5 °C.

#### Statistical analysis

Hierarchical cluster analysis, HCA was used to investigate the grouping of the sampling stations along the Sibulaut River and its tributaries. Ward's method using squared Euclidean distances as a measure of similarity was used. The dendrogram was used to interpret the result of the cluster analysis. Principal component analysis (PCA) was conducted to characterize the loadings of all physicochemical parameters for each of the PCs which were obtained. The PCA was carried out with factors having eigenvectors higher than one (Kaiser criterion). The quality of data for PCA analysis was confirmed with Kaiser–Meyer–Olkin (KMO) measure of sampling adequacy test. The rotation of PCs was made by varimax-normalized algorithm. All the statistical analyses were carried out by the Statistical Software for Social Sciences (SPSS Version 19.0, SPSS Inc. 1995).

## Results and discussion

### General environmental variables

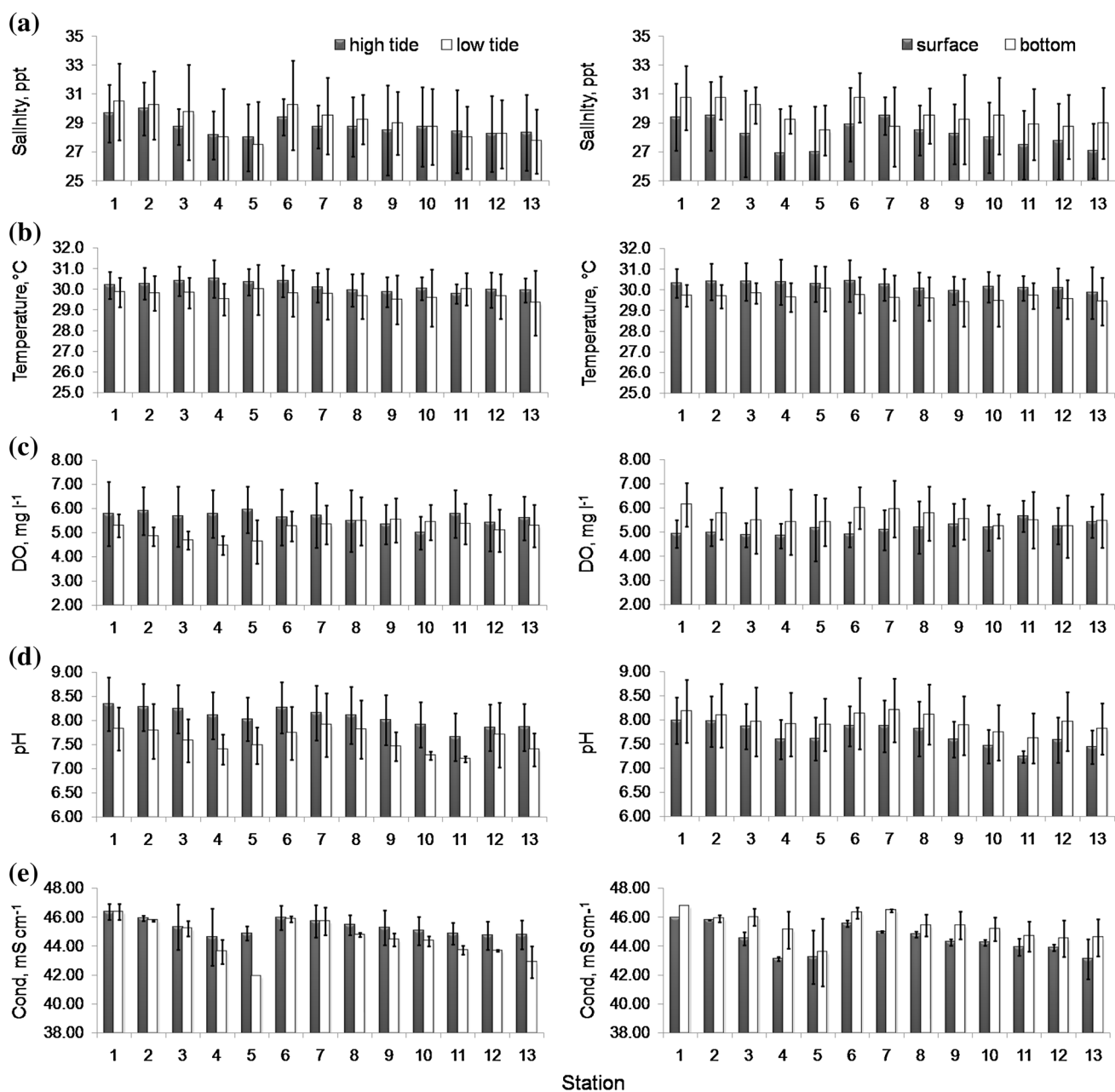
Figure 3 summarizes the general environmental parameters along the Sibulaut River and tributaries. All thirteen stations along Sibulaut River and tributaries were determined to be tidally influenced based on the observed salinity values. Salinity was more than 20 ppt even at the most upstream of the river in the present study. Salinity can reach up to 33 ppt at stations near river mouth. Salinity gradient was evident along the river while near-bottom waters were more saline than the sub-surface waters, resulting in weak horizontal and vertical stratification. The mean upstream to downstream salinity difference measured was around 2–3 ppt while the mean sub-surface to near-bottom salinity difference measured was approximately 2 ppt. Malaysia is a tropic country where the temperature is uniform throughout the year (Malaysian Meteorological Department 2012). The temperature was relatively consistent (≈29.9 °C) along the river throughout the study periods. Generally, near-bottom waters were slightly cooler than the sub-surface waters, resulting in weak vertical

stratification. This is probably attributable to the solar heating of sub-surface waters (Jones et al. 2008). The pH of the Sibulaut River was well within range, which near to 7.8 on average. The pH value was increased towards downstream with the lowest pH value of 7.0 at the most upstream sites and up to pH value of 8.9 at the downstream. Near-bottom waters were generally more alkaline than the sub-surface waters. The pH value was mainly influenced by tides as pH was higher during high tide. It is known that DO concentrations above 5 mg l<sup>-1</sup> are rarely harmful to aquatic life, whereas concentrations below 2 mg l<sup>-1</sup> are almost always detrimental and are considered a hypoxia threshold (Diaz and Rosenberg 1995). In sub-surface water samples collected in the present study, 62 % ( $n = 52$ ) showed DO concentrations more than 5 mg l<sup>-1</sup> while 71 % ( $n = 52$ ) of near-bottom water samples contained more than 5 mg l<sup>-1</sup> of the DO. Dissolved oxygen was generally well aerated along the river. However, lower concentration of DO was observed at station 2 in Loba Kilong River and stations 3–5 along Selang Sibulaut River in low tide condition.

Total suspended solids and turbidity values were steadily increased from the upstream to downstream (Fig. 4). Besides the clear gradient along the river, TDS and turbidity in near-bottom water column were higher than the sub-surface water, most apparent at the downstream and the middle of the river. Total suspended solids and turbidity were also higher during low tide and the variations were larger at downstream and the middle of the main river but smaller at the upstream. Transparency was relatively high in the Sibulaut River with the lowest transparency observed at station 5 (0.56 m) during low tide. The highest averaged transparency was observed at station 7 (>2 m). Transparency was higher during high tide at all stations and the variation was most apparent in the middle of the river and upstream. TDS and conductivity exhibited a similar increment trend towards downstream. The lowest value of TDS and conductivity was observed at station 5 during low tide and at station 4 during high tide. Generally, TDS and conductivity were higher during high tide and in near-bottom water column at all stations. TDS and conductivity concentrations are associated with ocean influence and surface water runoff from the shrimp farm effluent. As salinity increases, TDS and conductivity also increase. Shin et al. (2012) demonstrated similar distribution of TDS but the authors attributed the low TDS concentration at upstream to surface water runoff and eliminated the possibility of ocean influence as their sampling was conducted at low tide.

### Chlorophyll-*a* (chl-*a*)

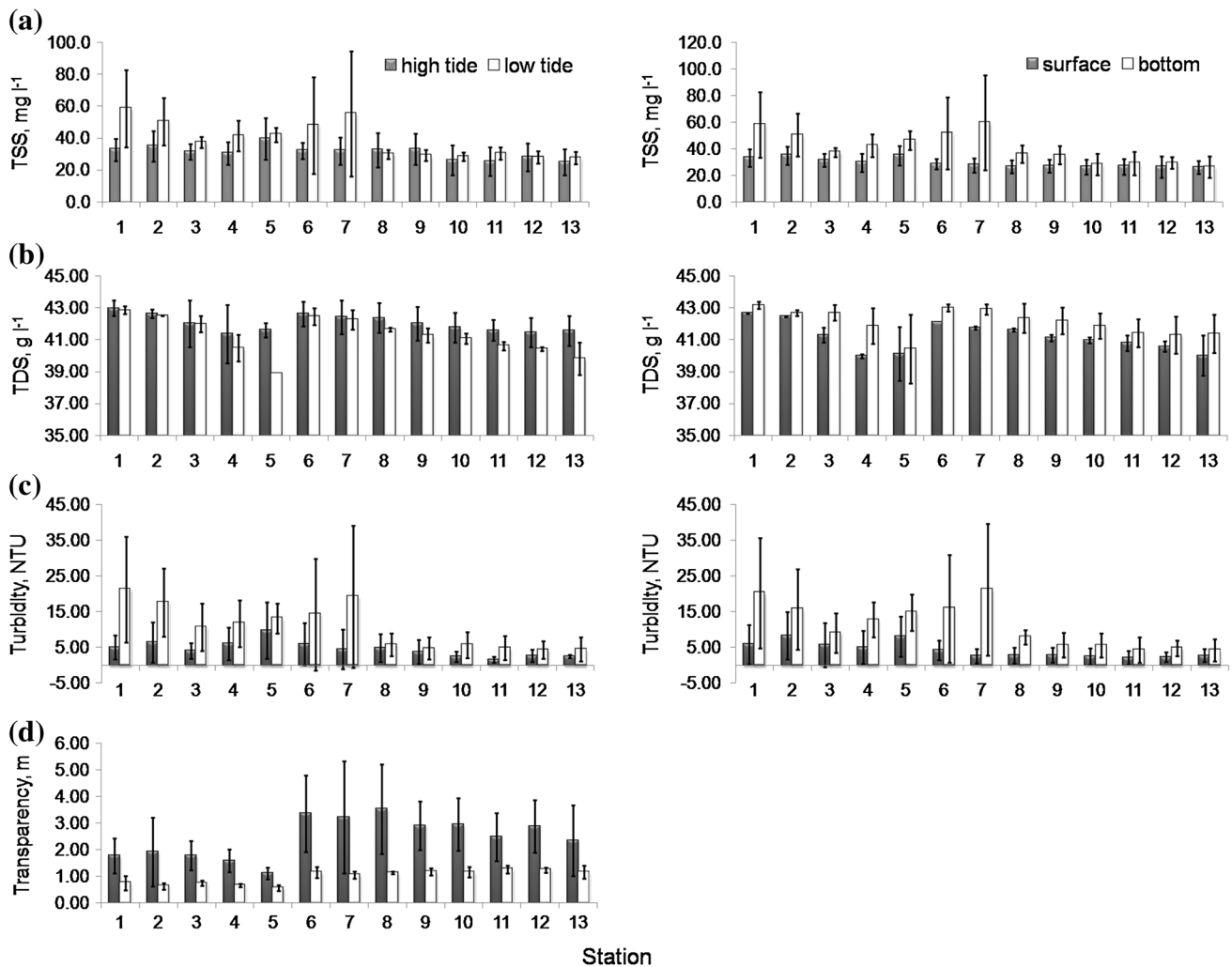
Chlorophyll-*a* content of sub-surface waters samples collected in the present study ranged from 0.207 ± 0.095 to



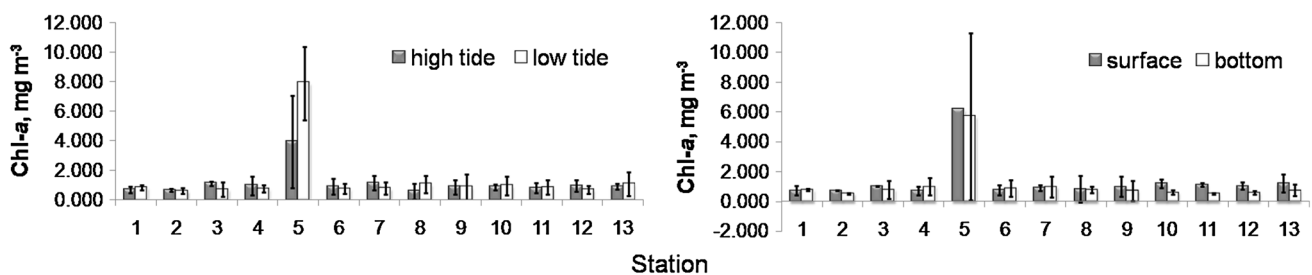
**Fig. 3** Hydrological parameters of **a** salinity (ppt), **b** temperature (°C), **c** DO ( $\text{mg l}^{-1}$ ), **d** pH, and **e** conductivity ( $\text{mS cm}^{-1}$ ) at 13 sampling stations along Sibulaut River and tributaries

$6.144 \pm 1.367 \text{ mg m}^{-3}$ , with 50 % of the samples measured  $<1 \text{ mg m}^{-3}$ , 42.3 % of the samples measured between 1 and  $3 \text{ mg m}^{-3}$ , and 7.7 % of the samples measured more than  $5 \text{ mg m}^{-3}$  (Fig. 5). Near-bottom chl-*a* ranged from  $0.259 \pm 0.097$  to  $9.680 \pm 1.900 \text{ mg m}^{-3}$ , with 69.2 % of the samples measured  $<1 \text{ mg m}^{-3}$ , 26.9 % of the samples measured between 1 and  $3 \text{ mg m}^{-3}$ , and 3.9 % of the samples measured more than  $5 \text{ mg m}^{-3}$ . On average, the distribution of chl-*a* was generally low at all stations ( $0.637 \pm 0.117$  to  $0.992 \pm 0.487 \text{ mg m}^{-3}$ ) except station 5 which exhibited a peak of  $5.921 \pm$

$3.260 \text{ mg m}^{-3}$ . The chl-*a* density at station 5 was about 9.5 times higher than the other stations during low tide, but only around 4.5 times higher than the other stations during high tide. Nonetheless, the high chl-*a* concentration rapidly decreased along the Selang Sibulaut River down to  $0.919 \pm 0.379 \text{ mg m}^{-3}$  at the conjunction with Sibulaut River. The observation was in good agreement with the study of Thomas et al. (2010) and Trott and Alongi (2000). The decrease of the chl-*a* may be due to the dilution or high grazing rate of zooplankton on phytoplankton as suggested by La Rosa et al. (2002). Chlorophyll-*a* data in the present



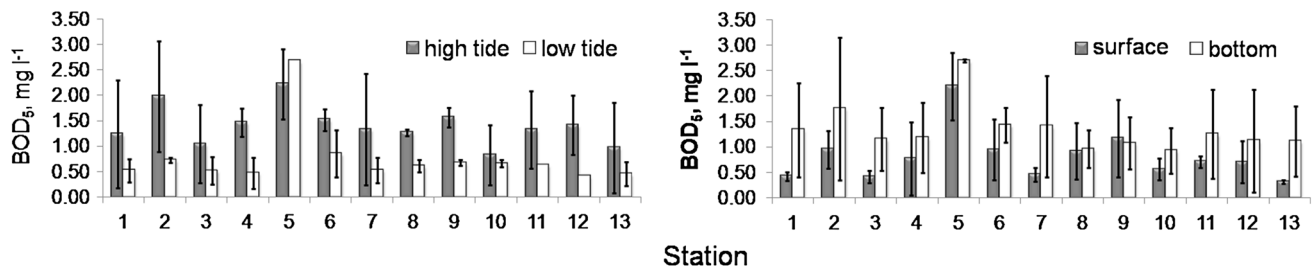
**Fig. 4** Mean concentrations of **a** TSS (mg l<sup>-1</sup>), **b** TDS (g l<sup>-1</sup>), **c** turbidity (NTU), and **d** transparency (m) at 13 sampling stations along Sibulaut River and tributaries



**Fig. 5** Mean concentrations of chl-a (mg m<sup>-3</sup>) at 13 sampling stations along Sibulaut River and tributaries

study were comparable to the study conducted in Pearl River Estuary, South China as chl-a concentration in the estuary was 0.8–5.6 mg m<sup>-3</sup> in ebb tide, and 0.8–7.8 mg m<sup>-3</sup> in flood tide (Huang et al. 2003). The present results are also similar with the surface water chl-a concentration (0.28–8.29 mg m<sup>-3</sup>) observed in the coastal waters of Kalkkham in the study of Satpathy et al. (2010).

Generally, chl-a level less than 3.4 mg m<sup>-3</sup> is indicative of the oligotrophic condition in tidal and fresh waters; ranging from 3.0 to 7.4 mg m<sup>-3</sup> is indicative of mesotrophic condition, and chl-a >6.7 mg m<sup>-3</sup> is indicative of eutrophic condition in the Chesapeake Bay (USEPA 2003). Molvaer et al. (1997) stated that for salinity above 20 ppt, chl-a concentration <2 mg m<sup>-3</sup> is considered as Class I or



**Fig. 6** Mean concentrations of  $BOD_5$  ( $mg\ l^{-1}$ ) at 13 sampling stations along Sibulaut River and tributaries

'very good'. On the other hand, Smith et al. (1999) equated oligotrophic, mesotrophic, eutrophic, and hypertrophic status in marine systems to mean chl-*a* concentrations  $<1\ mg\ m^{-3}$ , ranging from 1 to  $3\ mg\ m^{-3}$ , ranging from 3 to  $5\ mg\ m^{-3}$ , and more than  $5\ mg\ m^{-3}$ , respectively. When compared to the concentrations of chl-*a* in Sibulaut River with the above standard and guideline, the chl-*a* concentration in the Sibulaut River can be defined as 'very good' as the chl-*a* value was lower than  $2\ mg\ m^{-3}$  except station 5 which was characterized as eutrophic or even hypertrophic status. Although chl-*a* concentration at station 5 showed signs of eutrophication, but the problem was localized as chl-*a* concentration was decreased rapidly along Selang Sibulaut River.

#### Five-day biochemical oxygen demand ( $BOD_5$ )

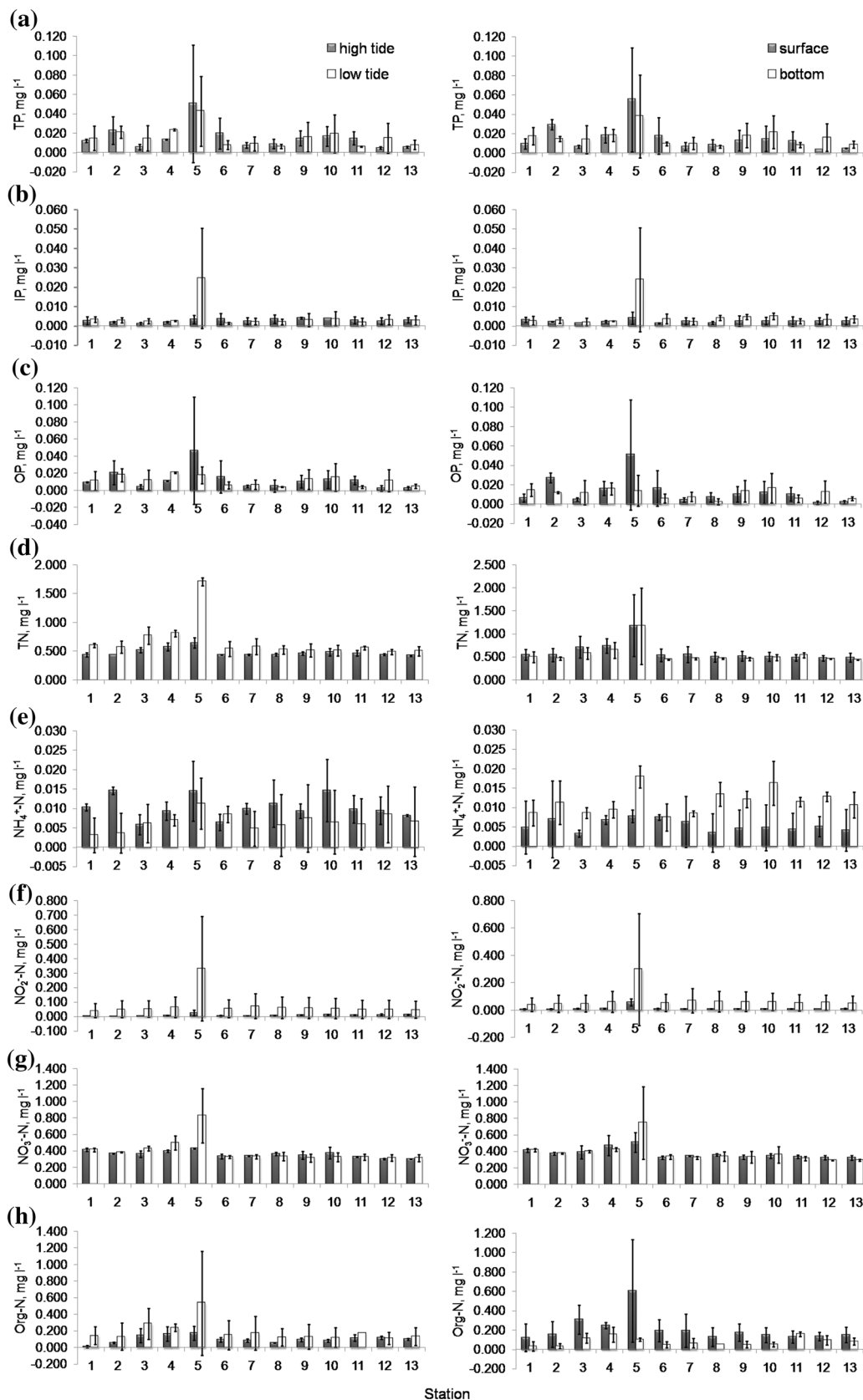
Figure 6 shows that the sub-surface water of the Sibulaut River and its tributaries was generally low in  $BOD_5$ , with 69 % ( $n = 26$ ) of the samples contained  $<1\ mg\ l^{-1}$ , 27 % ( $n = 26$ ) of the samples ranged between 1 and  $2\ mg\ l^{-1}$ , and 4 % ( $n = 26$ ) of the samples had  $BOD_5$  concentration more than  $2\ mg\ l^{-1}$ . In near-bottom water column, 42.3 % ( $n = 26$ ) showed  $BOD_5$  concentration  $<1\ mg\ l^{-1}$ , 42.3 % ( $n = 26$ ) ranged between 1 and  $2\ mg\ l^{-1}$ , and the remaining 15.4 % ( $n = 26$ ) of the samples had more than  $2\ mg\ l^{-1}$ . The highest concentration of  $BOD_5$  was found at station 5 which in the receiving creek of the wastewater discharged from a shrimp farm. Besides, stations 2, 6, and 9 also contained higher  $BOD_5$  values. The high  $BOD_5$  values in those sites indicated that the organic matter being input to the river. Generally,  $BOD_5$  was higher in near-bottom ( $\approx 1.34\ mg\ l^{-1}$ ) water column compared to sub-surface ( $\approx 0.81\ mg\ l^{-1}$ ); and during high tide ( $\approx 1.40\ mg\ l^{-1}$ ) compared to low tide ( $\approx 0.75\ mg\ l^{-1}$ ). The result was in contrast to the study of Yin and Harrison (2007) where the  $BOD_5$  was about  $1\ mg\ l^{-1}$  near the bottom and more than  $1.5\ mg\ l^{-1}$  at the surface water at Victoria Harbour, respectively. Nevertheless, the authors also showed that bottom  $BOD_5$  may be higher as phytoplankton cell sinks to the bottom layer.

Elevated concentrations of  $BOD_5$  were found near to shrimp farm, villages and jetty in the present study, yet, the  $BOD_5$  concentration was comparatively lower than other studies with sewage and anthropogenic influences. Shin et al. (2012) demonstrated that  $BOD_5$  concentration was higher at upriver near combined sewer overflow and sewage treatment plant effluents in the Hackensack River Estuary with an average of  $BOD_5$  fluctuated between 3.5 and  $12.8\ mg\ l^{-1}$ . Organic pollution is even more severe in Lower Sebou polluted by Gharb plain wastes with the means  $BOD_5$  vary from 19 to  $76\ mg\ l^{-1}$  (Bennasser et al. 1997). Extremely high  $BOD_5$  concentration was also found in Chetumal Bay which registered a mean  $BOD_5$  of  $32.26\ mg\ l^{-1}$  (Ortiz-Hernandez and Saenz-Morales 1999). Similar results with the present study was found in the study of Song et al. (2011) who reported that the  $BOD_5$  concentration ranged from 0.807 to  $7.557\ mg\ l^{-1}$  near the industrial and urban area in a tributary of the Pearl Estuary—Beijiang River. In Malaysia, the mean values of  $BOD_5$  were in the range of 0.26 to  $2.95\ mg\ l^{-1}$  in the Chini Lake (Hamzah and Hattasrul 2008) and from 2.36 to  $15.37\ mg\ l^{-1}$  in Langat River from 1995 to 2002 (Juahir et al. 2011).

#### Dissolved phosphorus

Figure 7 shows the distribution of phosphorus along Sibulaut River and tributaries. The highest TP concentration was observed at station 5 in the Selang Sibulaut River, which steadily reduced along the river from  $0.047 \pm 0.041$  to  $0.010 \pm 0.009\ mg\ l^{-1}$  at station 3. Station 2 which was located in Loba Kilong River also showed elevated phosphorus concentration with an average of  $0.022 \pm 0.009\ mg\ l^{-1}$ . Total phosphorus was lower in the middle and upstream sites, ranging from  $0.007 \pm 0.003$  to  $0.011 \pm 0.006\ mg\ l^{-1}$  except stations 6, 9, and 10 ( $0.014 \pm 0.012$  to  $0.018 \pm 0.013\ mg\ l^{-1}$ ). Organic phosphorus constituted the major proportion of the composition of phosphorus on the river basin, contributing 57.5–88.4 % of the TP. The distribution of OP followed the trend of TP where higher concentrations of OP were found at stations 2, 4, and 5. Inorganic phosphorus was generally low at all





**Fig. 7** Mean concentrations ( $\text{mg l}^{-1}$ ) of **a** TP, **b** IP, **c** OP, **d** TN, **e**  $\text{NH}_4^+$ -N, **f**  $\text{NO}_2^-$ -N, **g**  $\text{NO}_3^-$ -N, and **h** Org-N at 13 sampling stations along Sibulaut River and tributaries

stations, contributing only 11.6–42.5 % of the TP. Nevertheless, station 5 which was near to the discharge point of the shrimp farm exhibited a peak of  $0.014 \pm 0.019 \text{ mg IP l}^{-1}$ .

The Sibulaut River contained extremely low concentrations of IP with an average of  $0.003 \text{ mg l}^{-1}$ . The result was comparable to the study of Troussellier et al. (2004) who reported the orthophosphate concentration was  $0.03 \mu\text{mol l}^{-1}$  ( $\approx 0.001 \text{ mg l}^{-1}$ ) in the Senegal River Estuary. On the other hand, the concentration of IP in the present study was considerably lower than a Pearl River estuarine system of Hong Kong (Huang et al. 2003) and Kalpakkam Estuary in India (Satpathy et al. 2010) with an IP range of  $0.007\text{--}0.017 \text{ mg l}^{-1}$  and  $0.05\text{--}1.96 \mu\text{mol l}^{-1}$  ( $\approx 0.002\text{--}0.061 \text{ mg l}^{-1}$ ), respectively.

Averages of phosphorus concentration exhibited horizontal gradients, which were most pronounced near shrimp farm discharge point on Selang Sibulaut River. The phosphorus concentrations decreased with distance from the shrimp farm discharge point from  $0.047 \pm 0.041$  to  $0.010 \pm 0.009 \text{ mg l}^{-1}$ ,  $0.014 \pm 0.019$  to  $0.002 \pm 0.001 \text{ mg l}^{-1}$ , and  $0.033 \pm 0.040$  to  $0.009 \pm 0.008 \text{ mg l}^{-1}$  for TP, IP, and OP, respectively. This was in good agreement with results from Wu et al. (1994) where the impacts of marine fish farming activities on water quality were localized and did not appear to extend beyond a distance of 1–1.5 km from the fish rafts. Trott and Alongi (2000) suggested that the shrimp farm effluent was dissipated by tides and assimilated by the estuarine food web. Tidal mangrove estuaries have some capacity to process the intermittent inputs of pond-derived nutrients at least over short spatial and temporal scales. Thomas et al. (2010) and Ribeiro and Kjerfve (2002) also reported similar water quality improvement with distance. The dilution factor along Selang Sibulaut River was varied among phosphorus species. Inorganic phosphorus was dissipated faster than TP and OP. The dilution factor for IP ( $\approx 7$ ) was relatively higher than TP ( $\approx 2.5$ ) and OP ( $\approx 2.1$ ) from station 5 to station 4. Inorganic phosphorus was rapidly assimilated within the 1 km distance from station 5 to station 4; but no further decrement was observed from station 4 to station 3. Total phosphorus and OP were continued to decrease from station 4 to station 3 with a lower rate approximately 1.8–1.9 times. It is hypothesized that though phytoplankton is able to assimilate various types of phosphorus, but is preferred IP rather than OP (Cotner and Wetzel 1992), hence IP was decreased faster than OP.

In addition to phosphorus enrichment associated with shrimp farm effluents, Sibulaut River is also susceptible to the domestic sewage effluent. This can be seen from the results that measurements of phosphorus revealed considerable enrichment at station 2 besides of station 5. In the present study, sub-surface TP and OP at station 2 were 2.9

times and 3.4 times higher than the other stations along Sibulaut River; whereas IP concentration was similar to the other stations. The elevated phosphorus concentrations were confined to sub-surface water as near-bottom phosphorus at station 2 was similar or even lower than other stations. Since station 2 is near to the Sibulaut Village, the high mean phosphorus concentration indicated that wastewater discharged from the village was likely to contribute to the high phosphorus observed as phosphorus is an important component of domestic detergents. Ling et al. (2010a) reported high loading of reactive phosphorus into rivers in Kuching, Sarawak, from household wastewater, especially low income residential area which are attributable to more usage of phosphorus detergent. Besides, as station 2 was in the Loba Kilong River near the downstream, the intrusion of the seawater also helps to dilute the phosphorus concentration in the near-bottom water column.

Higher concentrations of phosphorus in the sub-surface water of station 2 and station 5 revealed that the sub-surface phosphorus runoff from shrimp farm and domestic sewage effluent is entering the area. Nevertheless, higher near-bottom IP concentration was observed at station 5. Sedimentation and re-suspension may play a major role in the distribution of phosphorus in the near-bottom water column (Pomeroy et al. 1965). The high load of phosphorus from a shrimp farm effluent may sorb to particulate matter and settle on the sediment in the intake creek of shrimp farm effluents (Sondergaard et al. 2001). There are no noticeable trends of the vertical profiles of phosphorus at other stations.

A mean TP concentration  $<0.010 \text{ mg l}^{-1}$  is characterized as oligotrophic status, ranging from  $0.010$  to  $0.030 \text{ mg l}^{-1}$  is mesotrophic status, ranging from  $0.030$  to  $0.040 \text{ mg l}^{-1}$  is eutrophic status, and more than  $0.040 \text{ mg l}^{-1}$  is hypertrophic status (Smith et al. 1999). Total phosphorus concentration is low in the Sibulaut River as 48 % of the samples ( $n = 52$ ) contained  $<0.010 \text{ mg l}^{-1}$  and 42 % of the samples ( $n = 52$ ) ranged from  $0.010$  to  $0.030 \text{ mg l}^{-1}$ . There are only 6 and 4 % of the water samples characterized as eutrophic and hypertrophic status. On the other hand, IP in all the samples collected in this study complied with the limit of  $75 \mu\text{g PO}_4 \text{ l}^{-1}$  ( $\approx 0.024 \text{ mg l}^{-1}$ ) according to the Malaysia Marine Water Quality Criteria and Standard except a water sample collected from station 5 during low tide ( $0.043 \pm 0.001 \text{ mg l}^{-1}$ ).

#### Dissolved nitrogen

The distribution of nitrogen was relatively consistent along Sibulaut River and tributaries, ranging from  $0.466 \pm 0.063$  to  $0.697 \pm 0.144 \text{ mg l}^{-1}$  except station 5 which exhibited a peak of  $1.177 \pm 0.618 \text{ mg l}^{-1}$ . In addition, concentrations of  $\text{NH}_4^+\text{-N}$ ,  $\text{NO}_2^-\text{-N}$ ,  $\text{NO}_3^-\text{-N}$ ,

and Org-N were also the highest at station 5 and steadily reduced along Selang Sibul River. Inorganic nitrogen (InOrg-N) constituted the major proportion of the composition of nitrogen in the river basin, contributing 75.6 % of TN. Nitrate nitrogen was the major composition among the InOrg-N, contributing about 67.0 %, followed by  $\text{NO}_2^-$ -N (7.1 %), and  $\text{NH}_4^+$ -N (1.5 %). Ammonium nitrogen was low in the study area, ranging from  $0.006 \pm 0.003$  to  $0.013 \pm 0.006 \text{ mg l}^{-1}$ . Nitrite nitrogen was relatively consistent along the river, ranging from  $0.024 \pm 0.035$  to  $0.040 \pm 0.062 \text{ mg l}^{-1}$  when excluding station 5 ( $0.179 \pm 0.273 \text{ mg l}^{-1}$ ). Concentration of  $\text{NO}_3^-$ -N was found higher at the river mouth ( $0.415 \pm 0.018 \text{ mg l}^{-1}$ ) while Org-N exhibited a different trend as concentrations were higher at the upper reaches of the river and lower at downstream sites.

The present study showed that the influence of the shrimp farm was reduced along Selang Sibul Laut as indicated by the decreasing nitrogen concentrations as similar to phosphorus distribution described previously. The nitrogen concentrations decreased with distance from the shrimp farm discharge point from  $1.177 \pm 0.618$  to  $0.647 \pm 0.174 \text{ mg l}^{-1}$  of TN,  $0.013 \pm 0.006$  to  $0.006 \pm 0.003 \text{ mg l}^{-1}$  of  $\text{NH}_4^+$ -N,  $0.179 \pm 0.273$  to  $0.030 \pm 0.043 \text{ mg l}^{-1}$  of  $\text{NO}_2^-$ -N,  $0.630 \pm 0.298$  to  $0.396 \pm 0.047 \text{ mg l}^{-1}$  of  $\text{NO}_3^-$ -N, and  $0.355 \pm 0.421$  to  $0.216 \pm 0.143 \text{ mg l}^{-1}$  of Org-N, respectively. The dilution factor from station 5 to station 4 was relatively similar among TN,  $\text{NH}_4^+$ -N,  $\text{NO}_3^-$ -N, and Org-N, ranging from 1.40 to 1.76 times. Nitrite nitrogen was the fastest dissipated nitrogen species with a dilution factor of 4.84 times. A dilution factor of about 40 times for nitrogen components was reported from the head of channels to the Santos Bay (Braga et al. 2000). The authors attributed the dilution to assimilation, physical mixture and other removal processes that present in the system. Nitrogen was rapidly dissipated within the 1 km distance from station 5 to station 4; but no further apparent decrement was observed from station 4 to station 3 as the nitrogen was decreased at an extremely slow rate. It is hypothesized that  $\text{NO}_2^-$ -N as an unstable intermediate form of  $\text{NH}_4^+$ -N and  $\text{NO}_3^-$ -N will tend to reduce to  $\text{NH}_4^+$ -N or oxidize to  $\text{NO}_3^-$ -N forms, hence the dilution factor of  $\text{NO}_2^-$ -N was the highest. Organic nitrogen was the second fastest nitrogen species that dissipated other than  $\text{NO}_2^-$ -N. Seitzinger and Sanders (1997) suggested that in estuaries with residence times on the order of weeks to months, such as Delaware Bay, river inputs of the biologically available portion of dissolved organic nitrogen (DON) are first utilized within the estuary. In contrast, in estuaries with residence times of less than a week, such as New York Bay, a portion of the biologically available DON may be utilized first within the estuary, with the remainder exported and utilized in continental shelf waters.

There was noticeable variation in the nitrogen concentration explained by differences in tidal stage. In general, the concentrations of TN,  $\text{NO}_2^-$ -N, and Org-N were found higher during low tide in most observations. Higher  $\text{NO}_3^-$ -N concentration was also observed at station 3 to station 5 on Selang Sibul River during low tide. A similar observation was reported by Ribeiro and Kjerfve (2002) where the Bay water quality was best at high tide at the time of maximum tidal dilution. On the other hand,  $\text{NH}_4^+$ -N exhibited different trends where higher concentration was observed during high tide at most of the stations. Marginally high sub-surface TN and Org-N concentrations were noticed at all stations except station 11. The largest variety of sub-surface and near-bottom Org-N was observed at station 5 indicated that the sub-surface water in the intake creek is influenced by the shrimp farm effluents rich in Org-N. Contrary to TN and Org-N concentrations, near-bottom  $\text{NH}_4^+$ -N and  $\text{NO}_2^-$ -N concentrations were higher than sub-surface water. Sub-surface and near-bottom concentrations of  $\text{NO}_3^-$ -N were relatively similar, except station 5 where near-bottom  $\text{NO}_3^-$ -N was higher than the sub-surface. Accumulation of high amounts of nutrients in sediments was evident from the high concentration of the overlying water discharged from a shrimp farm (Shahidul et al. 2004). The presence of higher nitrogen in near-bottom water samples could be due to either re-suspension of fine sediments by currents or to the disturbance of the bottom during sampling (Balls et al. 1994).

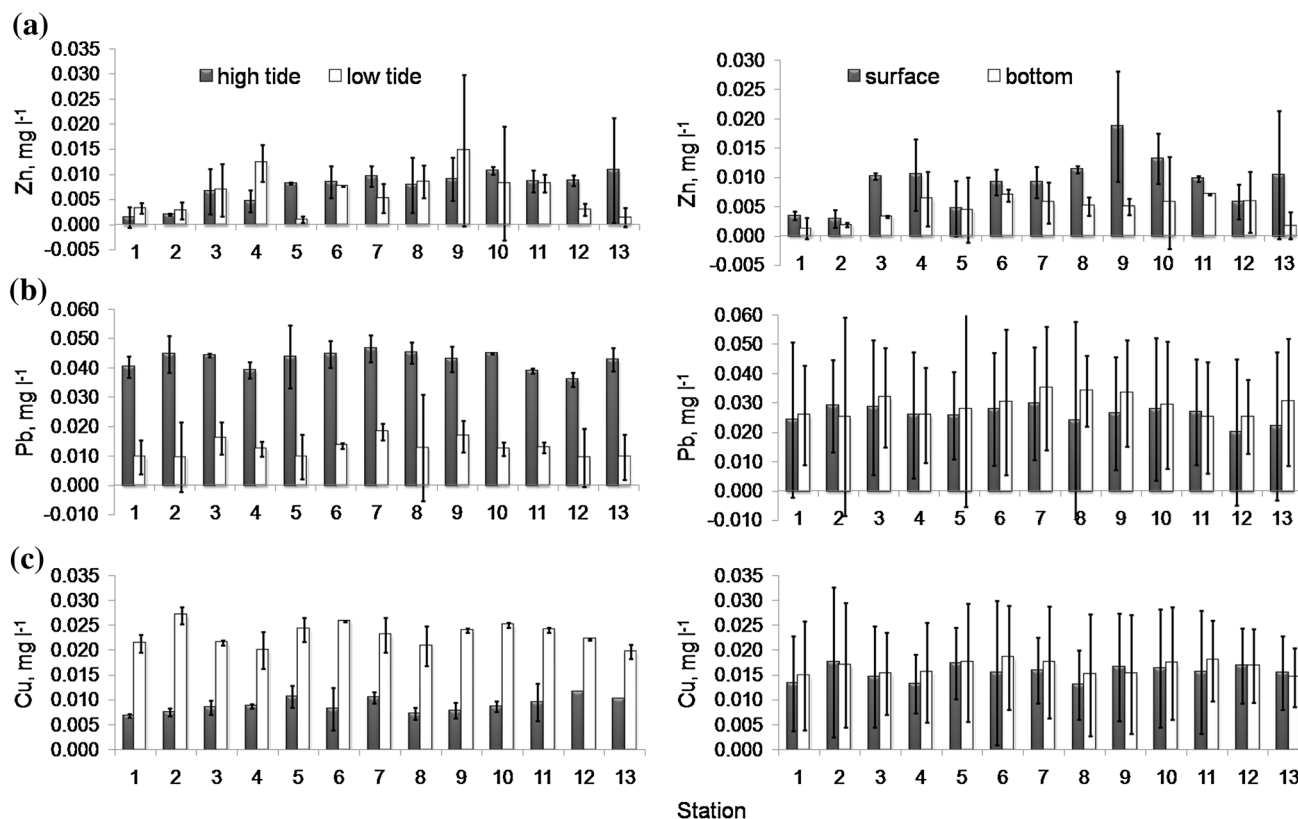
Smith et al. (1999) equated oligotrophic, mesotrophic, eutrophic, and hypertrophic status in marine systems to mean TN concentrations  $<0.26 \text{ mg l}^{-1}$ , ranging from 0.260 to  $0.350 \text{ mg l}^{-1}$ , ranging from 0.350 to  $0.400 \text{ mg l}^{-1}$ , and more than  $0.400 \text{ mg l}^{-1}$ , respectively. Sibul Laut River and its tributaries are characterized as hypertrophic status as the whole system contained more than  $0.400 \text{ mg TN l}^{-1}$ . Comparisons of the results of the present study with the Malaysia Marine Water Quality Criteria and Standard (Department of Environment 2010) indicated that  $\text{NH}_4^+$ -N in Sibul Laut River complied with the guideline value set by the Department of Environment, Malaysia. All the  $\text{NH}_4^+$ -N concentration observed in the present study was lower than  $70 \mu\text{g l}^{-1}$  of un-ionized ammonia ( $\approx 0.054 \text{ mg l}^{-1}$  of un-ionized ammonia nitrogen). Nitrite nitrogen complied with the guideline value of  $55 \mu\text{g NO}_2 \text{ l}^{-1}$  ( $\approx 0.017 \text{ mg l}^{-1}$ ) at most of the stations. However, near-bottom  $\text{NO}_2^-$ -N concentration in the Sibul Laut River exceeded the guideline value during low tide with  $\text{NO}_2^-$ -N concentration ranged from 0.077 to  $0.587 \text{ mg l}^{-1}$ . On the other hand,  $\text{NO}_2^-$ -N concentrations on three out of four water samples at station 5 were violated the guideline value. Nitrate nitrogen violated the guideline value of  $60 \mu\text{g NO}_3 \text{ l}^{-1}$  ( $\approx 0.014 \text{ mg l}^{-1}$ ) at all stations throughout the study periods. Nitrate is

thermodynamically the most stable form of InOrg-N in well-oxygenated waters. The reference level of  $0.014 \text{ mg l}^{-1}$  for nitrate may be too low because nitrate is relatively non-toxic to aquatic organisms (Wickins 1976; Rubin and Elmaraghy 1977). No formal water quality criteria and standard exist for nitrate in estuarine waters. The Canadian Council of Ministers of the Environment (2003) has recommended nitrate concentration ranging  $2.9\text{--}3.6 \text{ mg l}^{-1}$  to protect freshwater and marine life; whereas Camargo et al. (2005) proposed a maximum level of  $2 \text{ mg l}^{-1}$  for the protection of sensitive aquatic animals. What is more, the nitrate concentration for USA federal maximum level for drinking water is  $10 \text{ mg l}^{-1}$  (USEPA 2009). Accordingly, nitrate concentrations in the Sibulaut River are still under the recommended value as nitrate concentration was below  $2 \text{ mg l}^{-1}$  even at station 5.

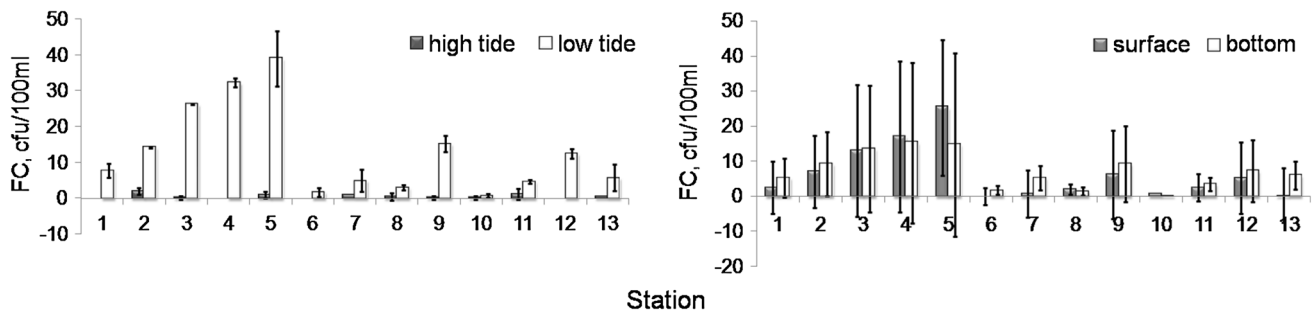
Total heavy metals (Zn, Pb, Cu, and Cd)

Figure 8 shows the distributions of total Zn, Pb, and Cu along Sibulaut River and tributaries. The distribution of total Cd was not shown as the concentration of total Cd was below the detection limit at all stations throughout the study period. Lead appears to be the most abundant metal

measured in the present study. The distribution of total Pb was relatively consistent along the river and tributaries when the data was pooled from the 13 stations during high and low tides, with an average of  $0.0277 \text{ mg l}^{-1}$ . Total Pb in this study was lower than the values quoted by Shin et al. (2012) in the study of total metal accumulation in the Hackensack River, Northern New Jersey ( $0.0349\text{--}0.2139 \text{ mg Pb l}^{-1}$ ) but was higher than the reported value for Potiguar Basin ( $\leq 0.002 \text{ mg l}^{-1}$ ) and Thames Estuary ( $\leq 0.008 \text{ mg l}^{-1}$ ) by Lacerda et al. (2013) and Pope and Langston (2011), respectively. Copper is the second most abundant metal in the river and is distributed geochemically in a similar manner to total Pb, with an average of  $0.0160 \text{ mg l}^{-1}$ . Total Cu concentration was within the range of Thames Estuaries ( $0.0097\text{--}0.2346 \text{ mg l}^{-1}$ ) and Hackensack River ( $0.0102\text{--}0.0363 \text{ mg l}^{-1}$ ) and higher than the reported values ( $\approx 0.002 \text{ mg l}^{-1}$ ) for Potiguar Basin. The concentration of total Zn was comparatively more fluctuated along Sibulaut River and tributaries. The lowest concentration of total Zn was found in downstream ( $0.0024 \text{ mg l}^{-1}$ ) while the highest concentration of total Zn was observed at station 9 ( $0.0119 \pm 0.0097 \text{ mg l}^{-1}$ ). Other than that, total Zn were relatively consistent on the main river with an average of  $0.0079 \text{ mg l}^{-1}$ . Total Zn was lower than the highest values



**Fig. 8** Mean concentrations ( $\text{mg l}^{-1}$ ) of **a** Zn, **b** Pb, and **c** Cu at 13 sampling stations along Sibulaut River and tributaries



**Fig. 9** Mean concentrations of FC (cfu/100 ml) at 13 sampling stations along Sibulaut River and tributaries

reported in the Hackensack River (0.0312–0.0849 mg l<sup>-1</sup>) and Thames Estuary (0.0330–0.0773 mg l<sup>-1</sup>). Cadmium was the least metal measured in the present study at which it is below the detection limit at all stations throughout the study periods. Total Cd concentration is similar with Potiguar Basin, which is also below detection level and is lower than values reported in the Hackensack River (0.0042–0.0328 mg l<sup>-1</sup>). Nduka and Orisakwe (2009) reported high level of Cd on the surface water of Aja-Etan and Ijala ( $\approx 1.33$  mg l<sup>-1</sup>) while high Pb was found on the surface water of Ifie-kporo and Ekpan ( $\approx 0.95$  mg l<sup>-1</sup>).

Total heavy metals in the intake creek of shrimp farm effluent (station 5) were within ambient level with other stations in the Sibulaut River. The results showed that the shrimp farm effluent had no major impact on heavy metals pollution in the receiving creek. Heavy metals are not major pollutants in shrimp farm effluents. In many cases, the study of heavy metal related to shrimp farm is more concerned on the accumulation of heavy metals in shrimps as it may adversely affect the health of the final consumption user (Amaraneni 2006; Kurun et al. 2007).

The main natural sources of metals into the aquatic system are the weathering of soils and rocks and precipitation; or from anthropogenic activities such as industrial, domestic, and urban sewage runoffs (Rippey 1982; Caccia and Millero 2003; Du Laing et al. 2008; Nduka and Orisakwe 2009; Sundaray et al. 2012). Elevated concentrations of total Zn were observed at station 9. These concentration peaks may represent events of re-suspension or the presence of Zn-enriched particles as suggested by Lacerda et al. (2013). In contrast, the total metal concentrations of Cu and Pb in the river showed no particular trend and this indicated that the source might be diffused. The source of total Pb on Sibulaut River could be due to the anthropogenic activities from adjacent villages as domestic sewage discharge and seepage from waste disposal sites could contribute greatly to the Pb concentration (Simeonov et al. 2003; Fianko et al. 2007). Soil leaching is the main factor that contributes to the Cu concentration as suggested by Simeonov et al. (2003). Besides, Cu released from the

antifouling paints used on boat hulls could also contribute to the Cu concentration (Caccia and Millero 2003; Whitall et al. 2010).

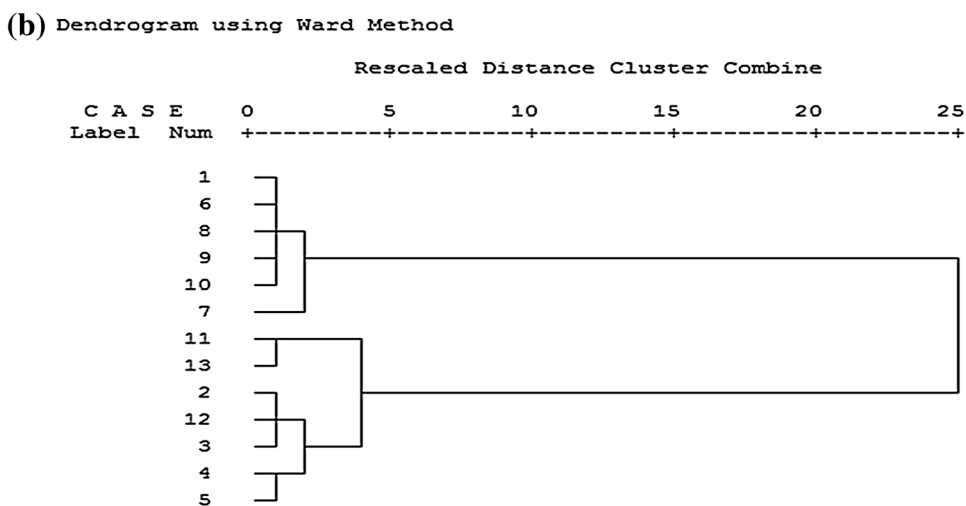
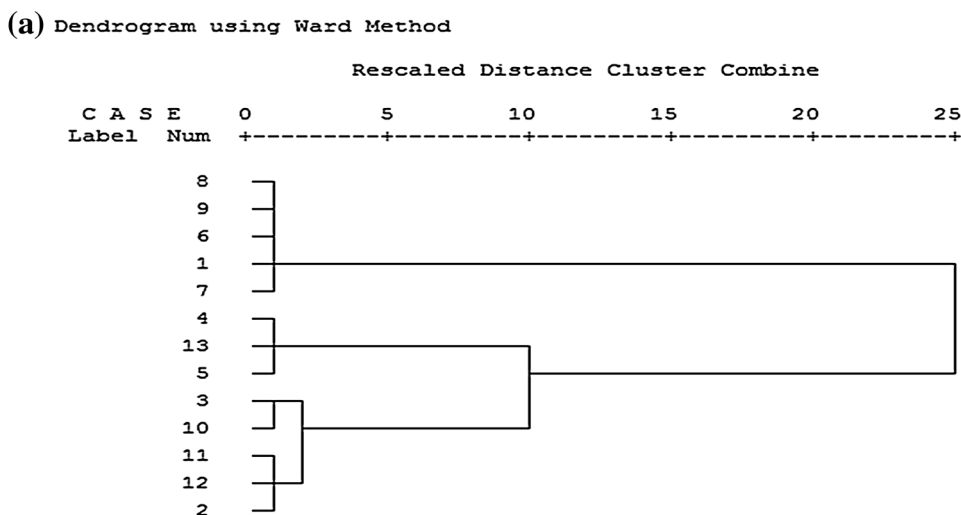
Sub-surface Zn were generally higher than near-bottom water, whereas Pb and Cu were not notably different between sub-surface and near-bottom water column. Lourino-Cabana et al. (2011) attributed the highly variable of Zn concentration in the Deûle River to re-suspension of polluted sediment and biological activities. In contrary, metal concentrations in surface and bottom waters in Potiguar Basin, Northeastern Brazil under the influence of the Guamare outfalls showed no difference, suggesting completing mixing of the water column in this relatively (<20 m) shallow waters (Lacerda et al. 2013). On the other hand, the present study showed that Zn and Pb were higher during high tide while Cu was considerably higher during low tide. Hatje (2003) demonstrated that concentrations of particulate trace metals, in general, tended to be higher at flood than ebb tide in Port Jackson Estuary, Australia. The higher concentration of Cu during low tide in the present study might indicate the contribution of Cu from the upstream sites.

No formal water quality criteria exist for total heavy metals in brackish waters. Department of Environment in Malaysia has constructed a system for the criteria and standard for metals in marine waters but was limited to dissolved metals. Nevertheless, the dissolved Zn, Pb, Cu, and Cd complied with the guideline value set by the Department of Environment, Malaysia as all the dissolved metals measured in the present study were below detection limits (data not shown).

#### Bacteriological water quality (fecal coliform bacteria)

Figure 9 shows that, on average, FC was lower in the main river body ranging from  $1 \pm 1$  cfu/100 ml to  $8 \pm 9$  cfu/100 ml. The highest value of FC was observed at station 9, followed by station 12. Station 2 at Loba Kilong River also contained relatively higher FC with a mean of  $8 \pm 7$  cfu/100 ml. Fecal coliform was found high, but steadily decreased along Selang Sibulaut River from  $20 \pm 24$  cfu/

**Fig. 10** Cluster of sampling stations along the Sibulaut River and its tributaries based on the water quality data collected in **a** 2010 and **b** 2011, respectively



100 ml at station 5 to  $13 \pm 15$  cfu/100 ml at station 3. Changes in concentrations in the downstream direction reflect the attenuation of FC through sedimentation, dilution, and die-off. The results were consistent with Ortiz-Hernandez and Saenz-Morales (1999) who demonstrated that the densities of FC were the highest in discharge samples and decreased with increasing distance from the discharge.

At low tide condition, FC was found high in Selang Sibulaut River especially at station 5 which is near to the discharge point of shrimp farm as illustrated in Fig. 8. Fecal coliform was steadily decreased from  $39 \pm 8$  cfu/100 ml at station 5 to  $26 \pm 0$  cfu/100 ml at station 3 in the sub-surface water column. Besides, FC was also found high at stations 2, 9, and 12 in both sub-surface and near-bottom water columns with an average of 14 cfu/100 ml. In addition, FC was found higher in the near-bottom water column at stations 1, 10, and 13 with an average of 10 cfu/100 ml. Nonetheless, FC was low at the rest of the stations during low tide,

ranging from BDL to  $6 \pm 4$  cfu/100 ml. Contrary to low tide condition, FC was generally low during high tide as most of the stations contained  $<2$  cfu/100 ml on average. The highest value of FC during high tide was found in near-bottom water column at station 2. Fecal coliform was not notably different between sub-surface and near-bottom water columns. Nevertheless, FC was drastically higher during low tide at most of the stations. A similar observation was described by Mallin et al. (1999) who noted an increase in the abundance of FC at or near low tide in tidal creeks that received polluted water from feeder creeks in North Carolina. Bordalo (2003) attributed the dramatic improvement in microbiological water quality to the increase in salinity during high tide in coastal beaches of Portugal.

Occasionally, slightly elevated concentration of FC ( $>10$  cfu/100 ml) was found at stations 1, 2, 9, and 12 in the present study. Fecal coliform is typically found in fecal material originating from human or other warm-blooded

**Table 3** Loadings of environmental variables on the first six varimax-rotated PCs for water quality data collected from Sibulaut River and its tributaries in 2010

Rotated component matrix <sup>a</sup>						
Parameter	Component					
	1	2	3	4	5	6
IP	0.937					
NO <sub>3</sub> <sup>-</sup> -N	0.878					
NO <sub>2</sub> <sup>-</sup> -N	0.840					
Chl-a	0.818					0.365
TN	0.811		-0.428			
pH		0.900				
DO		0.822				
BOD <sub>5</sub>	0.392	0.767				
Temperature		-0.763	-0.435			
Org-N			-0.819			
NH <sub>4</sub> <sup>+</sup> -N		0.348	0.727			
Salinity			0.637		0.434	
Width				0.907		
Depth				0.905		
Transparency		0.412		0.451	-0.416	
TSS					0.934	
Turbidity					0.835	
OP						0.976
TP	0.402					0.883
Initial eigenvalue	5.814	3.517	2.593	1.782	1.497	1.145
% of variance	30.598	18.513	13.647	9.381	7.878	6.028
Cumulative %	30.598	49.111	62.758	72.139	80.017	86.045

<sup>a</sup> Rotation converged in 7 iterations

animals. One of the most common FC sources is the non-point sources and uncontrolled discharge of sewage from densely populated urban and touristic areas (Aslan-Yilmaz et al. 2004; Ling et al. 2010b). Station 2 and station 12 received untreated waste discharge from Sibulaut and Temenggong Village. On top of that, station 1 at downstream received polluted water from Selang Sibulaut River and Loba Kilong River that contained shrimp farm discharge and domestic waste. It can be concluded that the untreated sewage discharge from the adjacent villages may cause the higher counts of FC at the area. However, those villages are not large enough and generate large enough sewage to contaminate the waters in a noteworthy way when compared to other studies. Mallin et al. (2009) demonstrated that frequent excessive FC concentrations were found particularly at some of the most urbanized sites in Burnt Mill Creek (955 cfu/100 ml) while periodic high counts occurred in some rural (536 cfu/100 ml) and suburban (493 cfu/100 ml) locations. Besides, the geometric mean concentration of FC bacteria was 790 cfu/100 ml in Tama

**Table 4** Loadings of environmental variables on the first three varimax-rotated PCs for water quality data collected from Sibulaut River and its tributaries in 2011

Rotated component matrix <sup>a</sup>			
Parameter	Component		
	1	2	3
Total Cu	-0.941		
Total Pb	0.929		
Temperature	0.923		
Transparency	0.808		0.332
Turbidity	-0.658	0.639	
FC	-0.575		-0.546
Salinity		0.802	
Conductivity	0.365	0.780	0.398
TDS	0.380	0.766	0.403
TSS	-0.326	0.750	
pH	0.565	0.592	
Total Zn		-0.522	0.305
Width			0.931
Depth			0.875
DO		0.396	0.578
Initial eigenvalue	5.654	3.828	1.850
% of variance	37.696	25.518	12.330
Cumulative %	37.696	63.213	75.544

<sup>a</sup> Rotation converged in 5 iterations

River, an urban river flowing through densely populated areas of Tokyo (Ham et al. 2009). Even more, Troussellier et al. (2004) delineated that high TTC abundances (33,000 cfu/100 ml) were recorded in oceanic water near the large village of Guet N'dar where around 15000 fishermen live with their family.

The present mean FC value was comparatively lower when comparing to the FC abundances reported in other riverine, estuarine or coastal areas; the Sibulaut River is far from being among the polluted sites. The mean concentration of FC bacteria in Sibulaut River was complied with water quality standards established for FC (<100 cfu/100 ml) by the World Health Organization (WHO 1998) or the European microbiological standards for bathing waters (Barrell et al. 2000). In fact, despite the influence of the shrimp farm wastewater, the contamination level of the Selang Sibulaut River appeared not so high; with the highest observed value of 50 ± 12 cfu/100 ml in sub-surface water at station 5 during low tide.

#### Hierarchical cluster analysis

Hierarchical cluster analysis was applied to detect similarities between the sampling stations along the Sibulaut

River and its tributaries using the sub-surface and near-bottom water quality data collected during low and high tide in 2010 and 2011, respectively. A total of 19 variables and 14 variables collected in 2010 and 2011 were first subjected to HCA, respectively. The 19 variables include the general environmental parameters, nutrients, chl-*a*, TSS, and BOD<sub>5</sub> collected in 2010 while the 14 variables include the general environmental parameters, total metals, and fecal coliform bacteria collected in 2011. The dendrograms showing the cluster of the sampling stations along the Sibulaut River and its tributaries are presented in Fig. 10. The result showed that water quality was grouped into three and two groups along the Sibulaut River and its tributaries in 2010 and 2011, respectively.

In 2010, cluster 1 of the water quality consisted of stations 8, 9, 6, 1, and 7 which were all located at main river. Station 4 and station 5 which were located near to a shrimp farm at Selang Sibulaut River; and station 13 which was located at the left upper reach of the Sibulaut River near to Tembangong Village were grouped into cluster 2. Lastly, cluster 3 consisted of sampling stations 3, 10, 11, 12, and 2, which are mainly located on the tributaries of the Sibulaut River. On the other hand, water quality collected in 2011 was grouped into two major clusters which were the main stem of the Sibulaut River and the tributaries of the river. The cluster analysis further confirmed that the effluents from the shrimp farm near Selang Sibulaut River consisted relatively higher nutrients compared to total metals and fecal coliform bacteria; hence, the stations near the shrimp farm were dissimilar with the other stations located on the main stem and other tributaries.

### Principal component analysis

Principal component analysis was conducted to characterize the loadings of 19 and 14 variables in the water quality of the Sibulaut River and its tributaries in 2010 and 2011, respectively. The KMO measure of sampling adequacy obtained was 0.445 and 0.760 for water samples in 2010 and 2011, respectively. Six PCs were obtained for the water quality in 2010 (Table 3); and three PCs were obtained for the water quality in 2011 (Table 4), with eigenvalues larger than 1, explaining 86.045 and 75.544 % of the total variance, respectively.

The first PC for water quality in 2010 has high positive loadings for the IP, NO<sub>3</sub><sup>-</sup>-N, NO<sub>2</sub><sup>-</sup>-N, chl-*a*, TN, BOD<sub>5</sub>, and TP, accounted for 30.598 % of total variance. The second PC accounted for 18.513 % of the total variance and positively loaded with pH, DO, BOD<sub>5</sub>, NH<sub>4</sub><sup>+</sup>-N, and transparency; but negatively loaded with temperature. The third PC which contributed 13.647 % of the total variance showed positive loadings on NH<sub>4</sub><sup>+</sup>-N and salinity, and negative loadings on TN, Org-N, and temperature. Positive

loading of width, depth, and transparency was observed in PC 4 which accounted for 9.381 % of total variance. The PC 5 and PC 6 contributed 7.878 and 6.028 % of the total variance, respectively. The PC 5 was found to be positively loaded with salinity, TSS, and turbidity; and negatively loaded with transparency. Positive loading was observed in chl-*a*, TP, and OP in the last PC.

In the present study, PCA showed positive loadings on nutrients, phytoplankton, and organic matter indicated that those enrichments on the Sibulaut River were derived from the same sources which were mainly regulated by the shrimp farm effluent and riverine runoff of domestic sewage. The positive loading of chlorophyll and nutrients indicated that high chl-*a* concentration may be due to the availability of higher concentration of nutrients which favor their growth. The BOD<sub>5</sub> and NH<sub>4</sub><sup>+</sup>-N were positively loaded in PC 2 indicated that they are possibly derived from the same sources particularly the shrimp farm. The oxidation of NH<sub>4</sub><sup>+</sup>-N contributes to high BOD<sub>5</sub>, and the process is strongly regulated by pH, DO, and temperature as BOD<sub>5</sub> and NH<sub>4</sub><sup>+</sup>-N are positively loaded with pH and DO, but negatively loaded with temperature. Positive loading on salinity and negative loadings of TN and Org-N were observed in the present study. In other word, TN and Org-N concentrations decreased as salinity increased which may be the result of mixing those nutrients with high salinity coastal water as depicted by Whitall et al. (2010). On the other hand, the positive loadings on salinity, TSS, and turbidity indicated that suspended solids were high in the downstream due to the movement of water in and out of the estuary by tides, causing fine particles to mix and be stirred up.

In 2011, PC 1 accounted for 37.696 % of the total variance, showing positive loadings on total Pb, temperature, transparency, conductivity, TDS, and pH; but negatively loaded with total Cu, turbidity, FC, and TSS. Positive loadings on turbidity, salinity, conductivity, TDS, TSS, pH, and DO; but negatively loaded with total Zn was observed on PC 2 which contributed 25.518 % of the total variance. The contribution of PC 3 to the total variance was 12.330 % and positively loaded with transparency, conductivity, TDS, total Zn, depth, width and DO; but has a negative loading on FC.

Positive loadings on pH, salinity, conductivity, TDS, and negative loadings on total Cu and total Zn were observed in the PCA. This phenomenon indicated that the most important sources of these metals could be the riverine and estuarine discharge, or the anthropogenic sources in the upstream. The decrease in metals concentration is probably the result of the dilution by seawater (Norton et al. 1984) or the precipitation of metals in the tidal area with higher pH (Grande et al. 1999). Positive loadings on temperature, conductivity, TDS, total Pb, and total Zn; and



negative loading of FC were observed in the PCA. This agrees well with the previous studies that increase in temperature (Chigbu et al. 2004; Noble et al. 2004), salinity (Hamzah et al. 2011; Yayintaş et al. 2007), and heavy metals (Malik and Ahmad 2002; Jana and Bhat-tacharya 1988) were harmful to FC survival in natural environments. Besides, the negative loadings on FC, turbidity and TSS are not surprising. Transport of FC via re-suspension of the sediments through the storm or tidal actions into the overlying water column is an important mechanism in the aquatic environment (Brauwere et al. 2011; Ouattara et al. 2011) as FC has longer survival in association with sediment particles (Ishii et al. 2007; Lee et al. 2006).

## Conclusions

Sibu Laut River is a highly dynamic, tidally dominated and well-oxygenated river with relatively consistent temperature. The pH value of the water is well within range and demonstrates an increasing trend from upstream to downstream. Total suspended solids and turbidity are low in sub-surface waters; show increasing values from upstream to downstream, but may be high at the intersection between freshwater and seawater. TDS and conductivity concentrations show changes with river miles, with higher concentration were found in near-bottom water column and in high tide condition. The spatial distribution pattern of the parameters studied indicates substantial inputs of contaminants possibly from shrimp farms as well as domestic sewage from villages nearby Sibu Laut River. The higher elemental concentrations which included chl-*a*, BOD<sub>5</sub>, nitrogen, phosphorus, and FC were associated with the stations near the intake creek of shrimp farm effluent in Selang Sibu River, and sewage discharge points of the villages of Sibu Laut, Telaga Air, and Temenggong. Nevertheless, the shrimp farm effluent had no major influence on heavy metals pollution. Sibu Laut River was characterized as oligotrophic and/or mesotrophic status according to chl-*a* and TP inputs, but was characterized as hypertrophic status according to the TN input. Nitrate concentrations in the Sibu Laut River violated the national standard, while occasional violation of NO<sub>2</sub><sup>-</sup>-N concentration was observed in near-bottom water column during low tide. Other water quality criteria in the Sibu Laut River are well within range and complied with the international and national standard.

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