CONTAMINATION STATUS AND ECOLOGICAL RISK OF HEAVY METALS IN SURFACE SEDIMENT OF KELANTAN RIVER AND ITS NEARSHORE AREA, MALAYSIA

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FACULTY OF SCIENCE
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**DISSERTATION SUBMITTED IN FULFILMENT
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ABSTRACT

A study on contamination status and ecological risk of heavy metal in surface sediment at selected sites of Kelantan river and its nearshore area was carried out. 10 samples along Kelantan river and 25 samples from nearshore were analyzed using Inductively Coupled Plasma Optical Emission (ICP-OES) to determine heavy metal concentrations. Sediment samples were also analyzed for particle size compositions, total organic matter, and pH content. The average concentrations for As, Cd, Cr, Cu, Mn, Ni, Pb, Zn were 7.38, 1.31, 17.71, 11.40, 507.15, 5.97, 22.61, 32.95 mg/kg for riverine and 14.14, 4.59, 29.79, 14.07, 389.96, 9.65, 62.21, 41.04 mg/kg for nearshore samples, respectively. The potential ecological risk index (PERI) showed station Bekok, Manek Urai, RH under considerable risks followed by station Pasir Mas as moderate risk. The pollution load index (PLI) classified four nearshore sites (KW10, KW17, KW18, KW37) as polluted. The geo-accumulation index (Igeo) categorised moderate contamination for Cd and Pb. The enrichment factor (EF) along river categorised extremely high enrichment for Cd, and significant enrichment for As, Pb and Mn while Pb and As were under very high and significant enrichment at nearshore areas. Pb, Cu, Zn, As, Ni and Cr, showed significant correlations with each other.

Keywords: Heavy metal contamination, ecological risk assessment, surficial sediment

STATUS KONTAMINASI DAN RISIKO EKOLOGI LOGAM BERAT DI SEDIMEN PERMUKAAN SUNGAI KELANTAN DAN KAWASAN PERSISIRAN, MALAYSIA

ABSTRAK

Kajian mengenai status pencemaran dan risiko ekologi logam berat di sedimen permukaan di kawasan terpilih Sungai Kelantan dan kawasan persisiran telah dijalankan. 10 sampel sedimen di sepanjang Sungai Kelantan dan 25 sampel dari persisiran laut dianalisis dengan menggunakan Inductively ICP-OES untuk menentukan kepekatan logam berat. Komposisi saiz zarah, jumlah bahan organik dan kandungan pH juga turut dikaji. Purata kepekatan untuk As, Cd, Cr, Cu, Mn, Ni, Pb, Zn adalah 7.38, 1.31, 17.71, 11.40, 507.15, 5.97, 22.61, 32.95 mg / kg untuk sungai dan 14.14, 4.59, 29.79, 14.07, 389.96 , 9.65, 62.21, 41.04 mg / kg bagi sampel persisiran. Indeks PERI menunjukkan stesen Bekok, Manek Urai, RH di bawah risiko yang agak tinggi diikuti oleh stesen Pasir Mas yang sederhana. Indeks PLI menunjukkan empat stesen berhampiran (KW10, KW17, KW18, KW37) sebagai tercemar. Indeks Igeo menunjukkan pencemaran sederhana untuk Cd dan Pb. Nilai EF di sepanjang sungai dimenunjukkan pengayaan yang sangat tinggi untuk Cd, dan signifikasi untuk As, Pb dan Mn manakala Pb dan As menujukkan pengayaan yang sangat tinggi dan signifikasi bagi kawasan persisiran. Pb, Cu, Zn, As, Ni dan Cr, menunjukkan korelasi yang signifikan antara satu sama lain.

Kata Kunci: Kontaminasi logam berat, analisis risiko ekologi, sedimen permukaan

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LIST OF SYMBOLS AND ABBREVIATIONS

As	:	Arsenic
Cd	:	Cadmium
Cr	:	Chromium
Cu	:	Copper
EF	:	Enrichment factor
HCl	:	Hydrochloric acid
HNO_3	:	Nitric acid
ICP-OES	:	Inductively coupled plasma atomic emission spectroscopy
Igeo	:	Geo-accumulation index
LOI	:	Loss on ignition
Mn	:	Manganese
Ni	:	Nickel
Pb	:	Lead
PCA	:	Principal component analysis
PD	:	Parkinson’s disease
PERI	:	Potential ecological risk index
PLI	:	Pollution load index
PSA	:	Particle size analysis
QA	:	Quality assurance
QC	:	Quality control
TOC	:	Total organic carbon
Zn	:	Zinc

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CHAPTER 1: INTRODUCTION

1.1 Background of the Study

The environment and its resources are imperative for the wellbeing of humans, hence the pollution of coastal waters, estuaries, and rivers caused by anthropogenic sources has eventually become an escalating global concern. The industrial development and growing human population have contributed to deterioration of the environment and alteration of the aquatic material transport pattern. Over the years, anthropogenic activities in the river basin had deteriorated the tropical rainforests. However, due to heavy precipitation sediment flux increases from rivers to sea in tropical area, which results in complicated biogeochemical cycling processes in tropical estuaries and adjacent shelf areas (Syvitski et al., 2005; Tiquio et al., 2017).

Sediment, as a carrier of heavy metals and pollution indicator of the aquatic environment, plays an important role in the assessment of metal contamination in natural waters. Heavy metal pollution that is largely caused by rapid industrialisation, urbanisation and most anthropogenic actions eventually builds up in soil and sediment, known as sinks for heavy metal accumulation (Zhu et al., 2012). However, physicochemical processes and change in environment conditions, such as acidification, could transfer metal forms from solid to liquid phase to be directly released into the freshwater and, eventually, marine environments (Moore et al., 2009). Metal-polluted environments cause heavy metal bioaccumulation in lower trophic levels of the food chain, such as plankton and zoobenthos, then biomagnifies towards higher trophic levels of the food chain, endangering public health, wild and agricultural flora as well as fauna (Petkovšek et al., 2012; Hossen et al., 2015). Furthermore, overexposure to heavy metals can cause birth defects, cancer, skin lesions, organ damage, mental and physical retardation (Singh & Cameotra, 2004; Poon et al., 2016). In the current era of rapid urbanization and industrial development, environmental pollution has been a major concern increasingly receiving public awareness concerning the restoration or remediation of metal-contaminated environments.

The Kelantan River is situated in the state of Kelantan with a population of more than 1.8 million in July 2018 according to the Department of Statistics Malaysia (2018). The earliest report on the environmental quality of the Kelantan River was by Ahmad in 2009 disclosed good physicochemical conditions of the river in the year 1993. In the year 2009, the Department of Irrigation and Drainage Malaysia (2009) reported fair conditions of the Kelantan River and its tributaries, the Galas River and Lebir River, in terms of turbidity, total suspended solids and total dissolved solids. Tan & Rohasliney (2013) reported levels of total suspended solids, turbidity and nitrate that exceeded the Malaysian Interim Water Quality Standards (INWQS) in the year 2010. Fish sampled from the Kelantan River in the year 2011 had elevated concentrations of Cd, Ni and Pb but did not exceed the quality standards (Rohasliney et al., 2014). More recently, Wang et al. (2017) reported that the Kelantan River experienced moderate pollution of Pb and Cr metals in the yeas apparent that a very limited number of studies on the Kelantan River was available from 1993 until present, especially concerning heavy metal distribution in the surface sediment of the Kelantan River. Moreover, local agriculture, fishery, tourism, logging, and manufacturing industry depend on the Kelantan River and its proximal water bodies for livelihood and population sustenance (Ibbitt et al., 2002). The growing economic importance and deteriorating environmental conditions of the river entail the need for recent in-depth assessment on the pollution status of the Kelantan River. This study reports the physicochemical influence of anthropogenic activities on the Kelantan River and its coastal area, aid decision making of public and stakeholders for remediation effort and is important for sustaining local ecology and fishery production. The objectives of this study were to analyse the heavy metal distribution and ecological risk in the Kelantan River and its nearshore area.

1.2 Problem Statement

Environmental issues such as marine pollution have been escalating in recent times. Nonetheless, the ecological assessment of heavy metals in the surficial sediment of the fresh and saline waters of Kelantan, Malaysia is scarce. Accordingly, this is a significant concern since the urbanisation and development in the densely populated areas of Kelantan is increasing, especially the capital city, Kota Bharu, that is located along the Kelantan River and its estuary. The elevated input of domestic and industrial effluent could pollute the surrounding aquatic environment and eventually pose risks to the public health. The accretion of metal pollutants in sediments can present severe environmental complications to the adjacent regions, which can be fatal and problematic to the surrounding living organisms (Zeitoun et al., 2014). Heavy metal input may be contributed by anthropogenic, urban, logging, agricultural, fishing, and daily activities, such as bathing and washing, as well as the number of populations residing nearby the river. As reported by Saraee et al. (2011), high arsenic and lead concentrations that were recorded in the studied sediment of Kelantan estuaries originate from motorized discharges and agricultural industries. However, it is worth noting that the heavy metal concentrations recorded in reports may comprise of the innate background levels of the system. Also, environment contamination could affect local agriculture, fishery, tourism, logging, and manufacturing industries that depend on the Kelantan River and its proximal water bodies for livelihood and population sustenance. Specifically, the main economic activity in the State of Kelantan is agriculture by raising paddy rice, oil palm, rubber, and tobacco. Additionally, fishing and livestock farming are also essential livelihood for the population along Kelantan River (Ibbitt et al., 2002). Other industries, such as tourism, logging, and chemical production industries, are also dependent on these water resources, whereby at the same time they accumulatively pollute the river water and sediment. In 2013, Tan & Rohasliney reported that the total suspended solids (TSS), turbidity and nitrate in Kelantan River exceeded the Malaysian Interim Water Quality Standards (INWQS). Besides that, the sediment that was contaminated not only harm the aquatic ecology, but also the terrestrial habitats when contaminated sediment is dredged to be used on land for various purposes, such as construction and landscaping (Tavakoly Sany et al., 2013). With a significant dependency on proximal water bodies for population sustenance, the Kelantan River is under risk of pollution, which may indirectly affect the region's economic status.

1.3 Significance of the Study

This study significantly enlightened the academia and scientific community on the heavy metal levels and ecological risk assessment of their adjacent environment. The findings of this study could promote public awareness to minimize harmful anthropogenic impact to the environment. Furthermore, the findings of heavy metal contamination levels could encourage remediation efforts for the public health, economy, tourism, and ecology of the sixth largest state of Malaysia, Kelantan, and its water bodies. Determination of heavy metal concentration in sediments is capable in deducing the potential impacts associated with the metals concerned (Burton et al., 1994). Data comprising of heavy metal concentration in sediment and particle size analysis of sediment helps in predicting the bioavailability of metals that has impacts on fishes since finer particle size with more surface area tend to adsorb relatively more metals, which is relatively more harmful to the aquatic organisms and consumed by population along the river (Horowitz et al., 1987; Hossen et al., 2015). Studies on heavy metal concentrations, sediment particle sizes are vital for the disclosure of in-depth knowledge associated with the concerned metals (Burton et al., 1994).

Studying the psychochemical characteristics of local sediments reveals useful information associated with heavy metal contamination at area of interest. The evaluation of the relationship between the psychochemical sediment characteristics and concentration of heavy metals in sediment is capable of indicating the types of heavy metals with the highest content and that are most easily adsorbed into sediment, thus indicating which heavy metals should be selectively monitored closely to mitigate its release into the river. Non-anthropogenic factors that tend to influence metal concentration in sediments are pH, organic matter content, and texture of sediment. Typically, sediment with pH level above six contained relatively more metals compared to sediment with lower pH. Furthermore, fine-grained particles that harbor more surface area, such as clay, contained more metals than coarse-grained particles, such as sand (Horowitz et al., 1987). Moreover, sediment with high organic matter content tends to harbor relatively higher concentration of heavy metals compared to sediment lacking in organic matter. Combined study of physicochemical sediment characteristics and heavy metal concentration is crucial in understanding their relationship and how it can be utilized to prevent or mitigate metal pollution.

1.4 Objectives of the Study

The objectives of this study are:

1. To characterise the physicochemical characteristics of riverbed and nearshore surface sediments.
2. To determine the concentration level of heavy metals (As, Cd, Cr, Cu, Mn, Ni, Pb, and Zn), contamination status and Potential Ecological Risk (PERI) level in riverbed and nearshore surface sediments.
3. To evaluate the relationship between the physicochemical characteristics and concentration of heavy metals in surface sediments at the study area.

CHAPTER 2: LITERATURE REVIEW

2.1 Environmental Pollution

Based on the Federal Environmental Protection Agency (FEPA) Act of 1990, Section 38, ‘environment includes water, air, land and all plants and human beings and/or animals living there in and the interrelationships which exist among these or any of them.’ The chief global environmental concerns include water pollution, fossil fuel depletion, climate change, and population growth exceeding Earth’s holding capacity (Avlonas, 2013; Mziray & Kimirei, 2016). The categories of environmental pollution are divided into water pollution, air pollution, land pollution, thermal pollution, radiation pollution, and noise pollution. Water pollution refers to the input of detrimental chemical, physical, or biological material to fresh or marine water bodies via suspended or dissolved emission. Examples of emissions are heavy metals, pesticides, and bio-accumulative nondegradable chemical compounds (Allen, 2017).

Environmental pollutants can substantially modify an environment’s properties, or circuitously influence food web equilibrium. The magnitude of pollution has extended public awareness concerning the necessity for efficient environmental planning and anti-pollution procedures. There is pressing demand for individuals, government agencies, and non-governmental organizations (NGO) to acquire more sustainable and green approaches (UNEP, 2012). The escalation of the human populace and the industrialization have amplified the supply and demand of goods and services, as well as the amount of waste and by-product, which in turn raise the requirement for resource management and energy alteration thus aggravating environmental pollution. Main fisheries have been dwindling by around 70% or are at their biological threshold. Forest area has been diminished by around 50% globally; 50% of the wetlands and more than 90% of the grassland have been altered. Around 40% of humankind is facing water scarcity (Avlonas, 2013; El-Moselhy et al., 2014; Sinha & Latrubesse, 2020). The abrupt deterioration in natural resources can instigate key national issues in providing long-term stability.

2.2 Sources of Heavy Metals in Sediment

Metals can be categorised into two groups, autochthonous or allochthonous metals, based on their source. Autochthonous metals occur naturally in the minerals of soils and rocks in trace quantities. Common autochthonous metals in the mineral grains of granites include aluminium in feldspars, and iron and Mn in various oxide and silicate minerals. Trace mineralization comprising other metals can transpire within granite and can contain small vein fillings of sphalerite (copper sulfide) and galena (lead sulfide). The weathering of geologic materials presents autochthonous metals to the river, and then downstream to the ocean as part of the sediment load (Rudnick & Gao 2003; Martin et al., 2015; Wu et al., 2019).

Allochthonous metals stem from external sources and watershed through anthropogenic and natural processes in dissolved and particulate forms (Martin et al., 2015). Volcanic, road, building, coal burning, and trash incineration sources produces Fe, Mn, Cu, Zn, Sr, Sn, Pb, Hg, Cd, As, Cr, and Co metals. The prime source of allochthonous metals to a watershed is through abrasion of man-made metal objects, industrial atmospheric discharges, and fallout (Davis et al., 2001; Grigoratos et al., 2014). Atmospheric build-up of natural and anthropogenic aerosol can be considerable sources of metals to a watershed (Stumm & Morgan 1996). The trace metals of coal involve Mn, Hg, Cr, Co, Cd, As, and Pb, whereas trash incineration contains Sn and Pb (Szpunar, 1992; Chillrud et al., 1999). Volcanism is a substantial supply of various heavy metals to the atmosphere, for instance Cd, Cu, Fe, Hg, Mn, Pb, Sr, and Zn (Schuster et al., 2002; Algul et al., 2020). Development, industrial, commercial, and residential sections concentrate, transport, and use a broad extent of allochthonous metals (Taebi & Droste, 2004; Chung et al., 2018). Aerosol metals, namely As, Cd, Co, Pb, Sn, Cu and Zn, have been categorised as road-specific heavy metals from brake linings, tires, oils and fuel, combustion in vehicles, and galvanized metal (Hildemann et al., 1991; Khan et al., 2017). In particular, brake linings and tires have significant amounts of Cu and Zn, respectively (Davis et al. 2010). Smelting, mining, and manufacturing of alloys, such as batteries and plastics, discharge Cd metal (Rashid et al., 2013; Khan et al., 2017). Conventional construction materials, namely painted wood, unpainted wood, metal, concrete, and brick hold substantial Zn, Cd, Cu, and Pb. Additionally, asphalt shingles are a source of Pb, whereas metal roofing is a source of Cd and Zn metals (Van Metre & Mahler 2003; Huang et al., 2020). However, the composition of metals sequestered in sediment is not only influenced by input sources, but also the changes to water bodies.

Table 2.1: Sources of heavy metals (Schuster et al., 2002; Szpunar, 1992; Chillrud et al., 1999; Hildemann et al., 1991; Davis et al., 2001; Algul et al., 2020).

Metal	Volcanic	Road	Building	Coal burning	Trash incineration
As		/		/	
Cd	/	/	/	/	
Co		/		/	
Cr				/	
Cu	/	/	/		
Fe	/				
Hg	/			/	
Mn	/			/	
Pb	/	/	/	/	/
Sn		/			/
Sr	/				
Zn	/	/	/		

Physical and chemical processes influence particle dynamics of metal transmission between the sediment and water column (Turekian, 1977; Olsen et al., 1982). The dispersal and movement of metals between surface water and sediments depends on their interactions with the sediment load, stream morphology, water chemistry, biota, geomorphology, geology, and climate (Luoma & Carter, 1991; Richardson & Kiffney, 2000).

The net effect refers to the metal concentration in the deposited sediment related to the metal concentration in the surface water. Depending on bioturbation and diagenesis, sediment burial conserves the relative conditions at the deposition time (Olsen et al., 1982; Stumm & Morgan, 1996; Thimsen & Keil, 1998). The changes in bioturbation and diagenesis include obstructing water flow through installation of dams, alteration of overland flow through management of storm water, addition of anthropogenic waste to water bodies, and fluctuations of physicochemical characteristics, such as sediment grain size and pH (Thimsen & Keil, 1998; Van Metre & Mahler, 2004; Coles et al., 2010; He et al., 2012). The said changes influence sediment chemistry by modifying the interactions of metals, biota, and interface between the sediment and surface water.

Overall, principal component analysis (PCA) had proven that metals in sediment are chiefly derived from natural sources, as well as surface runoff pollutants of industrial and agricultural origins, such as chemical engineering, electronics, mechanical manufacturing, wastewater, pesticide, and fertiliser (Xia et al., 2011; Li et al., 2017).

2.3 Toxic Effects of Heavy Metals

2.3.1 Arsenic (As)

Arsenic is a carcinogenic composite that naturally occurs in common geologic progressions and anthropogenic activities, for instance the excavating and processing of ores. Arsenate and arsenite are inorganic lethal forms of As. The trivalent form of As is the most noxious as it intermingles with thiol groups of proteins, whereas the pentavalent form of As owns unparalleled oxidative phosphorylation hence is less noxious. However, the mineral form of As is toxic and tends to bioaccumulate (Liao et al., 2004; Alamdar et al., 2017).

The environmental and ecological quality of water bodies can be degraded through weathering of As minerals from sedimentary and meta-sedimentary bed rocks, smelting, the disposal of Asal pesticides, Asal chemicals, and natural mineral deposits (Matschullat, 2000; Smedley & Kinniburgh, 2012). Additionally, an assortment of animal feed, insecticides, and fertilizers discharge As in huge amounts. Furthermore, paints, dyes, soaps, metals, semi-conductors, and medicines also contain As.

Among illnesses that has been connected to prolonged disclosure to low dilution of As are parenchymal cell damage, liver fibrosis, cirrhosis, cancers, hypertension, diabetes mellitus, hyperkeratosis, melanosis and hepatocellular carcinoma (Ahmed et al., 2013; Das et al., 2004; He et al., 2014). Lu et al. (2014) reported that mice subjected to 10 ppm As experienced substantial change in abundance of gut microbiome and its metabolic profile at the abundance and function levels, respectively. Exposure to As at early stage worsens the swelling response to and lowers the clearance of influenza A, which occasioned in lasting and severe alterations in the lung technicalities and air route make-up of mice (Ramsey et al., 2013). Hypertensions, cardiovascular illnesses, and diabetes has been connected to disclosure to As. (Patrick, 2003). Long periods of exposure are linked with irritation of skin and mucous membrane, anaemia, peripheral neuropathy, liver and kidney impairment (Rahman et al,. 2014). Arsenic can be exposed to living organisms through water, food, and air. More than 30 countries worldwide have suffered As toxicity that occurred mostly due to drinking water that has been polluted by As (Chowdhury et al., 2000). It can threaten public health if the level of As in ground water is 10 to 100-fold higher than the accepted value of As in drinking water (10 µg/L) according to World Health Organisation (WHO) (Hoque et al., 2011). Arsenic poisoning can be severe or long-lasting, whereby the latter is also called Arsenicosis. Due to symptom specificity, Arsenicosis diagnosis in man emphasises on skin appearances. Arsenicosis is indicated by specific skin lesions, which are keratosis and pigmentation (Martin & Griswold, 2009). Subjection to lower amount of As can cause lower manufacture of red and white blood cells, nausea, throw up, stinging in legs and hands, irregular heartbeat, and blood vessel impairment. Prolonged exposure leads to the development of diabetes mellitus, heart-related disease and hypertension, peripheral vascular disease, pulmonary disease, neurological disease, and interior tumours (Smith et al., 2000). Fatality is higher with prolonged Arsenicosis as it causes permanent alterations of crucial organs. However, no efficient therapy is available for this sickness regardless of the degree of this possibly deadly noxiousness (Mazumder, 2008).

2.3.2 Cadmium (Cd)

Cadmium is considered as an unnecessary component in our body but can display poisonous propensities even in low dilutions due to its low removal degree (Nawrot et al., 2010; Pretto et al., 2014). It is regarded as a cancer-causing agent that can be released from all tobacco forms, whereby its largest source into the human body is through passive and active smoking (Feldkamp et al., 2014; Mohapatra et al., 2014). Cancers of the kidney, prostate and lungs has been related to exposure towards Cd at work related to battery companies and metal smelting (Nordberg et al., 2005). Cadmium intoxicates the biological system by obstructing sulfhydryl enzyme and interacting with other cells' ligands hence causing the disturbance of oxidative phosphorylation pathways (Jomova & Valko, 2011; Waisberg et al., 2003; Perera et al., 2016). Cadmium impairs and causes breakdown of human bones, nerve tissues, kidneys, liver, and lungs through accumulation in the respective parts (Tsutsumi et al., 2014). Kidney can be harmed by Cd because it is difficult to eliminate from where they were commonly stored. In the liver, albumin operates by carrying Cd to the liver where metallothionein bounds it and the compound produced is discharged back into the circulation. The Cd-metallothionein compound is taken in by the proximal tubular cells of the kidney, which induces oxidative stress and tubular dysfunction (Patrick, 2003; Sabolic et al., 2006). This boosts the formation of kidney stones and influences the respiratory system (Hambach et al., 2013). Through the exposure of human liver towards Cd, it promotes hepatocellular injuries by synthesising metallothionein (Kang et al., 2013). The upregulation of stress gene by Cd damages the actions of many enzymes that defends the liver from oxidative stress. Protein carbonylation, deoxyribonucleic acid (DNA) damage and lipid peroxidation are the damages induced by the impairment of hepatic markers and the enhancement of oxidative stress (Rashid et al., 2013). Yang et al. witnessed that testicular necrosis in rat caused by Cd had augmented degree of glutathione peroxidase and malondialdehyde in them. They discovered that glutathione reacted with the oxygen radicals via the glutathione peroxidase system inside the cell. It is observed that the superoxide dismutase activities have been lowered and the damage of DNA was substantial (Yang et al., 2003).

Cadmium can cause both acute and long-lasting poisoning (Chakraborty et al., 2013). It accumulated in the proximal tubular in high concentration and is very toxic to the kidney. By damaging the bone and disrupting renal function, Cd can lead to bone mineralization. Research on animals and humans has discovered that exposure to Cd can cause severe consequences such as osteoporosis together with hypercalciuria, formation of renal stones and disruptions of calcium metabolism. Inhalation and ingestion of Cd at high amount can seriously harm the lungs and irritate the stomach, which results in vomiting and diarrhoea. Being exposed longer to it at lower concentration leads to lung damage, fragile bones, and kidney disease due to deposition of the Cd metal (Bernard, 2008). Compared with other metals, Cd and its compounds are highly water soluble, and they tend to bioaccumulate because of their high bioavailability. Being exposed to Cd for a long period can alter the morpho-pathological of the kidneys. Smokers are more prone towards Cd intoxication than non-smokers since tobacco plants gather Cd from the soil, just like other plants and act as the main Cd supply to the smokers. Cd exposure to non-smokers happened through food and some other pathways. However, uptake through other pathways is considerably smaller (Mudgal et al., 2010). Interaction between Cd and essential nutrients causes them to become lethal. It is shown in the experimental analysis that animal lungs absorbed 50% of Cd while gastrointestinal tract absorbed lesser. High Cd exposure during human pregnancy rises problems such as lowered weight and premature birth (Flora et al., 2008).

2.3.3 Chromium (Cr)

In various manufacturing activities, Cr is widely used, and this resulted in numerous environmental system contamination (Cohen et al., 1993; Abbas et al., 2016). Cr composites are commercially used in wood conservation, leather tanning, dyes and pigments, industrial welding, and chrome plating. In boilers and cooking systems, it is also used as an anti-corrosive (Norseth, 1981; Wang et al., 2006). Exposure to Cr in a non-job-related occurrence happens through water and food intake while in work-related occurrence happens via inhalation (Langård & Vigander, 1983; Oyetibo et al., 2016). Although inhalation is the main route for human exposure to Cr whereby lung is the prime target, Cr exposure through human skin has also been reported (Costa, 1997; Shelnutt et al., 2007). For instance, dermatitis is a common occurrence among construction employees, and it is credited to their exposure to cement which contains Cr (Shelnutt et al., 2007). It has been identified that exposure to Cr (VI)-comprising complexes in job-related and environmental situations brings multiorgan poisoning, such as respiratory tract cancer in humans, renal injury, allergy, and asthma (Goyer, 2001, WHO/IPCS, 1988). Inhaling Cr (VI) at high quantities can lead to nose ulcers and irritation of nose lining.

Concerning the intake of Cr (VI) in animals, the major health issues that had occurred are anaemia, sperm damage, male reproductive system damage, ulcers and irritation in the small intestine and stomach. It has been recorded that the swelling and acute redness of the skin are the allergic responses of people that are very sensitive to Cr (VI) or Cr (III). A rise of stomach cancer was discovered in animals and humans that were exposed to drinking water comprising Cr (VI). Intake of tremendous amount of Cr (VI) compounds by humans unintendedly or purposely leads to critical renal, neurological, hepatic, haematological, gastrointestinal, cardiovascular, and respiratory effects prior death or in surviving patients who received medical treatment (ATSDR, 2008). The cancer-causing mechanism of Cr is not fully comprehended but there are reports of Cr causing cancer in humans and land-dwelling animals (Chen et al., 2009).

Naturally, Cr (VI) composites such as lead chromates, strontium chromate, Zn chromates and calcium chromates are highly lethal and cancer-causing. Conversely, Cr (III) plays a crucial part in glucose metabolism apart from being a vital nutritive supplement for animals and humans. The intake of trivalent Cr composites through the airways and digestive tract is slower compared to hexavalent Cr composites. Work-related sources of Cr involve metal plating, leather tanning, wood preservatives, paper, cement, rubber, paint pigments, metal alloys, protective metal coatings and magnetic tapes (Martin & Griswold, 2009). Schroeder et al. (1970) stated that no substantial statement has been issued on the quantity of Cr inhaled through smoking although cigarettes comprise 390 g/kg of Cr. A deep hole will be formed after wounded skin encounters with any type of Cr composites. Being exposed to Cr can also lead to the ulcer formation that heals slowly and persists for months. Among chromate labourers, ulcers on the nasal septum are very normal. Erythrocyte glutathione reductase will be inhibited if humans are exposed to high quantity of Cr composites, which diminishes the ability of methaemoglobin reduction into haemoglobin (Koutras et al., 1965; Schlatter & Kissling, 1973). Through various *in vivo* and *in vitro* experiments, the outcomes showed that chromate composites can harm DNA in diverse approaches and cause the construction of DNA adducts, modifications in transcription of DNA, alterations in replication, sister chromatid exchange, and chromosomal aberrations (O'Brien et al., 2001; Matsumoto et al., 2006).

2.3.4 Copper (Cu)

Cu is bioaccumulated through water and food, whereby its concentration is high in the liver but low in the blood, hence urinary Cu levels are used as a biomarker for Cu poisoning (Brewer 2003a, b; Wu et al. 2016). Cu poisoning has been reported to cause various illnesses, such as diarrhoea, attention deficit disorder, hair loss, breathing difficulty, severe headache, convulsion, circulatory collapse, cold perspiration, insomnia, chronic infections, cramps, premenstrual syndrome, migraine headaches, stomach ache, vomiting, nausea, fibromyalgia, prostatitis, male infertility, dysmenorrhea, depression, autism, asthma, arthritis, anxiety, anorexia, anaemia, allergies, jaundice, inky urine, salivation, metallic taste in mouth, blue vomiting matter, colicky abdominal pain, fluid brown and bluish stool, candida overgrowth, coma precede death, Indian childhood cirrhosis, idiopathic Cu toxicosis, endemic Tyrolean infantile cirrhosis, Menkes disease, and Wilson's disease (Hordyjewska et al. 2014).

Infants nursed with milk kept in Cu or brass containers developed Indian childhood cirrhosis due to high liver Cu. For Wilson's disease, clinical symptoms include liver disease and neurological damage during juvenile and adult stages of the disease, respectively. Optical cornea is also affected with brown discoloration, a specific symptom of Wilson's disease (Tapiero et al., 2003; Lorincz, 2010). Wilson's disease caused about 140 infant deaths in west Austria, Germany, and Italy during 1900–1980 (Baker et al., 1995). Alzheimer's disease is an age-related disorder with variations in cellular processes.

It is distinguished by the manifestation of neurofibrillary tangles and amyloid plaques, mostly consisting of hyperphosphorylated tau and amyloid-b protein. Alzheimer’s disease entails irritation in the neuron, oxidative stress, low metabolism. Alteration in homeostasis of metallic ions, such as Cu, leads to the pathogenesis of Alzheimer’s disease. Cu checks the excessive activity of N-methyl-d-aspartate receptors, the excitatory neurotransmitter receptors (Eskici & Axelsen, 2012; Noda et al., 2013). With age, Cu increases in blood plasma, serum, and brains suffering from Alzheimer’s disease, hence increased Cu negatively correlates with cognition loss (Eskici & Axelsen, 2012; Arnal et al., 2013; Noda et al., 2013). High Cu level has been reported to cause homodimerisation of the amyloid precursor protein (APP), leading to hyperphosphorylation of tau protein and aggregation of amyloid b (Atwood et al., 2000; Arnal et al., 2013; Gonzalez-Dominguez et al., 2014). Modifications in the intensity of trace element produces an adverse effect on cellular metabolism and therefore, can induce carcinogenesis. Cu stimulates the manufacture of reactive oxygen species, whereby disproportion between reactive oxygen species and antioxidant can lead to direct DNA damage, mutation, and development of cancer (Khoshdel et al., 2016).

2.3.5 Manganese (Mn)

Mn poisoning due to work-related settings, such as unintentional consumption and prolonged contact, is more typically observed than environmental Mn poisoning (Santamaria, 2008). The first report of Mn causing neurological consequence was in 1837 by John Couper, who depicted bent posture, salivation, whispering speech, limb tremor, and muscle weakness as symptoms of Mn poisoning, which has now been known as manganism (Couper, 1837; Khan et al., 2017).

Manganism is a neurological disorder that bear a resemblance to Parkinson’s disease (PD), but manganism and PD target different areas of the brain than those affected by PD

(Calne et al., 1994; Olanow, 2004). PD mainly involves dopaminergic neuron loss within the substantia nigra, which is a brain region that is spared in manganism (Guyton & Hall, 1996; (Davutluglu et al., 2011)). The clinical symptoms of manganism and PD are rigidity, resting tremor, dystonia, bradykinesia, and propensity to fall backward (Calne et al., 1994). The resemblances can be elucidated by the accretion of additional Mn in the basal ganglia in manganism with basal ganglia dysfunction in PD (Dobson et al., 2004).

Additionally, Mn poisoning through chronic inhalation, battery production, steel manufacture, mining, and smelting can cause neuropsychiatric disruptions, central nervous system irregularities, neurodegenerative disorder, and adverse male reproductive, endocrine, or haematological effects (ASTDR, 2000; Huang et al., 2001; Bowler et al., 2007; Kim et al., 2007; Santamaria et al., 2007). However, Greiffenstein & Lees Haley (2007) concluded that the neurobehavioural influences reported to be related with Mn-exposure were likely due to covariate effects.

A crucial first stage in comprehending the function that Mn works in neurotoxicity is to ascertain exposure circumstances that cause heightened Mn levels in the central nervous system (Dorman et al., 2002). This comprehension is markedly essential for Mn because its toxicity mechanism is not thoroughly studied hence poorly grasped. Research had focused on the fundamental issues of Mn toxicity, including Mn absorption, bioavailability, ingestion or inhalation exposure, toxicity mechanism, neurological effects, dose-response relationship, tissue distribution, and homeostatic regulation of various Mn forms. Nevertheless, most animal models are insufficient for accurately assessing the neurological effects in humans, hence challenging to study the mechanisms involved in neurotoxicity. Most animal model studies assessed the neurochemical effects of Mn with non-human primates (McMillan, 1999; Newland, 1999). Recent research employed lower Mn dose for more chronic exposure, whereas initial research employed higher Mn dose to mimic human exposure. In general, the Mn neuropathological, neurochemical, and behavioural findings in primates were in agreement with those in humans (Aschner, 2007).

2.3.6 Nickel (Ni)

The path of Ni exposure chiefly influences the intensity of Ni impact on development, reproduction, neurology, immunology, system biology, and carcinogenicities by means of chronic or acute exposure. One of the most common routes to Ni toxicity is an allergic skin reaction. Ni generates multiple reactions in the human immune system in a diverse fashion (Hostynek, 2006). Ni is a potential carcinogen, allergen, immunotoxic and immunomodulatory agent that causes headache, nausea, abdominal cramps, diarrhea, vomiting, dermatitis, immunologic urticarial irritation, vertigo, weakness, visual disturbances, sweating, palpitations, tachycardia, cyanosis, dyspnea, constant cough, chest stiffness, breathlessness, giddiness, respiratory disorders, asthma, and bronchitis, whereby the indications can persist for several hours to days (Das & Buchner, 2007; Das et al., 2008; Das et al., 2010). Fatal cardiac arrest of a 2-year-old girl was reported for accidental consumption of nickel sulfate, whereas fatal acute respiratory distress syndrome (ARDS) of spray-painting employees was reported (Rendall, 1994).

Work-related exposure to Ni from welding is also among the most common chronic exposure routes in humans. Ni refinery workers were reported to experience pulmonary and nasal cancer (Enterline & Marsh, 1982). However, female Ni refinery workers in another study did not reveal any growth or reproductive hazards (Warner, 1979). In terms of work-related incidental exposure, Ni was hazardous to growth, digestion, and reproductive health (Chashschin, 1994). Chronic contact of Ni compounds to rodents caused lung tumour, adenoma, adenocarcinoma, squamous cell carcinoma, fibrosarcoma cancer, bone marrow micronuclei formation, decreased offspring survival, weight loss, albuminuria, emphysema, inflammatory and proliferative alterations (Dhir et al., 1991; Diwan et al., 1992; Vyskocil et al., 1994).

2.3.7 Lead (Pb)

Pb poisoning can occur through piping, (Brochin et al., 2008). Most of the Pb is deposited in skeletal bones (Papanikolaou, 2005). Pb is considered a carcinogen, according to the Environmental Protection Agency (EPA). Pb poisoning has been associated with neurological disorder, hypertension, cognitive impairment, and disruption of enzyme activation, trace mineral absorption, structural protein synthesis, or sulfhydryl antioxidant production (Ercal et al., 2001; Patrick, 2003). Additionally, Pb poisoning has also been observed to affect the gastrointestinal tract and central nervous system in adults and children (Markowitz, 2000).

Acute exposure, such as through occupation, can cause loss of appetite, headache, hypertension, abdominal pain, renal dysfunction, fatigue, sleeplessness, arthritis, hallucination, and vertigo. Chronic exposure of Pb can result in oedema, mental retardation, birth defect, psychosis, autism, allergy, dyslexia, weight loss, hyperactivity, paralysis, muscular weakness, brain damage, kidney damage and may even cause death (Teo et al., 1997; Martin & Griswold, 2009; Taylor et al., 2012).

Animal model studies of Pb poisoning revealed symptoms of embryonic toxicity, behavioural alteration, memory deficit, weight loss, reproduction inhibition, altered calcium homeostasis, and kidney failure (Chen et al., 2012; Zhang et al., 2013; Agrawal et al., 2014; Luo et al., 2014). Exposure of HEK293 kidney cells to Pb caused reduced cell viability, distorted cell, cohesion loss, lipid peroxidation, and manufacture of superoxide anions (Gargouri et al., 2013). Environmental and domestic sources of Pb ions are the main cause of Pb poisoning but with proper precautionary measures it is possible to reduce the risk associated with Pb toxicity (Brochin et al., 2008).

2.3.8 Zinc (Zn)

Zn poisoning, through inhalation, smelting, welding, can cause metal fume fever (MFF), cough, chest pain, respiratory effect, fatigue, nausea, muscle soreness, fever, and dyspnoea (Brown, 1988; Jan & Edward, 2016). Immediate symptoms after uptake of toxic amounts of Zn include abdominal pain, nausea, vomiting, lethargy, anaemia, and dizziness (Porea et al., 2000). Zn was also suggested to be involved in prostate cancer at high or prolonged doses, as Zn levels in prostate adenocarcinoma were significantly lower than in the surrounding normal prostate tissues (Leitzmann et al., 2003; Franklin et al., 2005; Jarrard, 2005; Jan & Edward, 2016). This increased risk may not be due to direct carcinogenicity of Zn, because it is known that immunosuppression significantly increases the incidence of cancer, and high doses of Zn can be immunosuppressive.

High Zn ingestion were also reported from food or drink storage in galvanized containers, resulting in abdominal cramps, epigastric pain, vomiting, nausea, and diarrhoea (Brown et al., 1964). Furthermore, a death was reported from oral intake of 28 g zinc sulfate after symptoms of tachycardia, hyperglycaemia, haemorrhagic pancreatitis, vomiting, and renal failure (Fox, 1989). However, no dermal irritation was found when 25% Zn oxide patch was tested on human skin for 48 hours (Lansdown, 1991).

2.4 Bioavailability of Heavy Metals

Bioavailability involves (1) a physiologically triggered effect or accumulation in the organism, known as toxicological bioavailability, (2) a physiologically regulated uptake, known as environmental bioavailability, and (3) a physicochemically controlled desorption, known as environmental availability (Peijnenburg et al., 1997; Lanno et al., 2004; Harmsen, 2007; ISO 17402 2008). The principle of dynamic processes was adopted in the International Standards Organisation (ISO 17402 2008) and US National Research Council (NRC) report (NRC 2003). Bioavailability is a complicated dynamic progression intensely influenced by exposure, metal speciation, and organism (Hund-Rinke & Koerdel 2003).

Additionally, bioavailability had been defined as an amount of heavy metal (mol kg-1) in the soil prevailing in types and quantities that plant roots can absorb (Warrington and Skogley, 1997). Bioavailability had also been alternatively defined as transport rate or flux (mol m-2 s-1) where metals are transmitted to organisms through soil (Taghon et al., 1999; Shor & Kosson, 2000). According to Harmsen (2017), the former content-based bioavailability was a more feasible approach than the latter flux-based bioavailability due to sounder analytical practicability.

The NRC Committee (2003) first familiarised bioavailability as "the individual physical, chemical, and biological processes that determine the exposure of organisms to chemicals present in soils and sediments", which included five developments: (1) incorporation into a living system through metabolic processes, (2) passageway through a physiological membrane, (3) carriage of attached contaminants on organism membrane, (4) transportation of unconfined contaminants, and (5) pollutant release from solid stage. Biological, physical, and chemical methods, such as spectroscopy, extraction, toxicity, were assessed to conceive a standard gauge of bioavailability derived from a few criteria to acquire a mechanistic insight of bioavailability, and metal associations and forms. According to the NRC, however, no singular method can be universally employed to gauge bioavailability, and that comprehending site-specific circumstances is vital to opt for a suitable tool.

Moreover, internal bioavailability is the capacity of metals to be absorbed and exert subsequent toxicological effects in target tissues, whereby external bioavailability is the capacity of metals to be dissolved and released from environmental matrix or media, such as soil or sediment (Caussy et al., 2003). According to Kramer & Ryan (2000) as well as

Semple et al. (2004), bioaccessibility is used synonymously with external bioavailability. However, Reichenberg & Mayer (2006) reported that bioaccessibility and chemical activity should be distinguished to measure bioavailability. Bioaccessibility functionally refers to metal amount that can be mobilised for uptake at a timescale under certain environmental conditions.

2.5 Effect of Physicochemical Characteristics on Heavy Metals

The grain size distribution (PSA), total organic matter content (LOI), and pH are some of the physicochemical traits that are commonly correlated with concentrations of heavy metals. pH plays a role in affecting solute concentration and absorption in soil, while soil texture influences the mobility of soil metals. The coarse nature of the sediments had lower organic matter content. Locations with abundant clay had greater organic matter and carbon, whereas locations with abundant soil organic carbon had greater concentration of heavy metals (Ayolagha & Onwugbuta, 2001; Oyedele et al., 2008; Quenea et al., 2009). The deterioration in soil organic carbon in the control sites could be an outcome of leaching due to relatively high sand content.

Impacts of physico-chemical feature of sediments, such as organic matter and particle size, are essential on the dispersal of heavy metals (Zhu et al., 2016). Particles can be carried into estuaries through longshore currents, whereby coarse sediments are sedimented rapidly while fine grains are more easily suspended in the water column (Zhang & Gao, 2015), and progressively transmitted farther from the coast.

Organic matter comprises a substantial portion of estuarine sediment delivered from both marine and terrestrial sources, such as dead marine organisms, by-products of living biomass, plant litter, and soil organic matter (Agah et al., 2016). Negative correlation between organic matter and sand percentage, while positive correlation between organic matter with silt and clay were observed. The higher organic matter corresponded to a higher clay content and indicated that organic matter was enriched in fine-grained marine sediments. High level of heavy metals is associated with organic matter because fine-grained particles provide additional sorption sites with higher specific surface area in sediments (Fan et al., 2014). Metals Cr, Cu, and Zn were influenced by grain size and organic matter. However, metals Pb, Cd, As, and Hg did not show distinct correlation with most parameters.

2.6 Environmental Indices for Heavy Metals

The ecological indices for heavy metals include potential ecological risk index (PERI), pollution load index (PLI), geoaccumulation index (Igeo), and enrichment factor (EF). Potential ecological risk index (PERI) is based on toxic-response factors or numerical toxicity units obtained from natural metal concentrations in soil, igneous rocks, freshwater, sediment, and fauna or flora and a few normalisation procedures (Hakanson, 1980; Abrahim & Parker, 2008; Guo et al., 2010; Kabir et al., 2011). Further manipulations provided toxicity units used to convert sediment metal concentrations into potential ‘ecological risk’. PERI index groups ecological risk into four classifications: low (RI < 150); moderate (RI 150 - 300); high (RI 300 - 600); and very high (RI > 600).

Pollution load index (PLI) is an antilog minus the concentration of a contaminant minus an uncontaminated baseline value divided by a threshold minimum value related with degradation, or an alteration in the quality of the estuary minus the baseline. Baselines (uncontaminated) and thresholds (minimum concentration associated with degradation) are expressed for each pollutant (Wilson & Jeffrey, 1987; Banu et al., 2013). PLI data are combined for pollutant mixtures and normalised to the number of pollutants. PLI is indexed from 0 (unpolluted) to 5 or 10 (highly polluted).

Geoaccumulation index (Igeo), developed by Muller (1969), evaluates ecological pollution through contrasting the disparities concerning preindustrial and current concentrations. Igeo was initially constructed to evaluate the pollution level of soil and sediment (Loska et al., 2004). This approach has been broadly utilised in European trace metal research (Ji et al., 2018).

Enrichment expresses the scale of human-caused alterations and determines the level of pollution from the pristine condition. Anthropogenic enrichment, also known as mean or maximum enrichment, is the natural background abundance in relation to the current sedimentary metal concentration. Furthermore, sediment quality is the capacity of sediment to uphold a healthy benthic population. Background and enrichment do not consider toxicity or biological effects (Chapman & Wang, 2001). Although these metrics are unrelated and derived from distinct criteria and methodologies, they are frequently sum up as a single indicator. For example, priority index merges PLI, PERI, and Igeo, while the PLI merges enrichment and sediment quality guidelines (Caeiro et al., 2005; Kabir et al., 2011). Additionally, background concentration of metals and its derivation have substantial consequences in the assessment of anthropogenic alteration to an aquatic ecosystem through contamination indices or factors.

Dissimilar terrestrial land usage influence soil contamination in varying extents (Lee et al., 2006). The pollution in agricultural areas is relatively less than that in manufacturing areas. However, agricultural soil is mostly contaminated with Hg and Cd (Wei & Yang, 2010). As a result, it is essential to elucidate heavy metal contamination based on land use pattern. Consequently, the heavy metal pollution in sediment varies by proximal anthropogenic land use as it can change the physical and chemical properties of the upstream and downstream sediment metal composition (Liu et al., 2007; Zheng & Zhang, 2011).

In general, this chapter discusses the possible sources of the heavy metals in surface sediment, toxicity, and bioavailability of heavy metals where it is capable of accumulating in each level in food chain and eventually affects the human and environment as well as ecological system. Most of the studies in Malaysia were carried out to determine the impacts of anthropogenic activities on fish resources, water, and human in terms of ergonomics aspects. This study aimed to determine the distribution and heavy metal contamination in surface sediment of selected sites at Kelantan river and nearshore areas. Contamination status of heavy metals into riverine and nearshore environment were examined and ecological risk status were examined using appropriate tools as described in this chapter. The following chapter discusses the methodologies in identifying the heavy metals concentrations, grain size distribution, pH value, total organic matter content in surface sediment at selected sites of Kelantan river and nearshore areas.

2.7 Geography, Climate Setting and Oceanography of study area

Peninsular Malaysia is situated at the junction of the South China Sea (SCS) and the Indian Ocean. Its covers an area approximately 131 554 km2 where the north side is adjacent to southern Thailand, east side borders the southern part of SCS, and the west and south sides are connected to the Straits of Malacca (Sanusis et al., 2017). It can be structurally split into three north-south extensional geological belts namely the western, central, and eastern belts (Waipan Ng et al., 2015). The western belt is incorporated from Sibumasu Terrane while, the eastern and central belts are considered as single tectonic plate, Sukhothai Arc. The collision between the two plates along with the Bentong-Raub suture zone occurred in the Late Triassic (Metcalfe, 2016). The composition of bedrock of Peninsular Malaysia are consists of Paleozoic-Mesozoic granite, granodiorite, and Paleozoic sedimentary rocks. which is dominated by mudstone, sandstone, limestone and andesitic-rhyolitic volcanic rocks.

The Kelantan River is one of the largest rivers on the Peninsula and Kelantan River Delta receives the sediment supply form the Kelantan River that covers an area of approximately 1879 km^2 (Wu et al., 2019). The temperature ranges between 21°c to 35°c at this area and experienced an equatorial climate throughout of the year. This region is affected by two types of wind known as Southwest Monsoon that occurs during late May to September and Northeast Monsoon that happened during November to March. The speed and direction of the wind in the South China Sea region impacted by these prevailing monsoon seasons throughout the year. As such, Malaysian Meteorological Department reported that during the southwest monsoon, the wind from southwest blows at a speed about 15 knots whereas, the wind blows from east(northeast), at a speed about 10 to 20 knots and may reach up to 30 knots sometimes during the northeast monsoon.

The sources of Kelantan River sediments are mainly derived from hinterland areas which are marked by three mountains namely Gunung Cintawasa in the southeast at an elevation of 1185 m, Gunung Stong in the eastern region at an elevation of 1422 m and Gunung Korbu (elevation 2183 m) in the south. Sungai Lebir, Sungai Nenggiri and Sungai Galas are the three main rivers located at the upper reaches of the Kelantan River that drained the sediments from these areas. The Nenggiri River flows over the granite and metamorphic rocks that supplied quartz and feldspar minerals whereas, the Galas River and Lebir River flowed over argilite sedimentary rocks, limestone and volcanic sandstone and caused the various composition of grain sizes and minerals. The distribution of sediments also influenced by the intense weathering and erosion activities from various sources at the offshore areas of Kelantan River (Radzir et. al.,2018).

The characteristics of sediment at Kelantan Delta interpreted as recently deposited sediments where the Kelantan River act as a major contributor. The sediments indicated that it could have sourced from various types of rocks such as sedimentary rock, metamorphic rocks, and also granitic rocks. The sediment deposited at river mouth of

Kelantan River were further by littoral drift process where it caused the mixing of fine sediments at the delta (Gao et al., 2019). The sedimentary process at this area indicates that sediment with bigger dimension and heavier deposited in shorter distances from the origin unlike the fine sediments which is further carried away to the continental shelf due to the energy degree and gravity attraction. Geological and geographical conditions of the area also caused turbulent flow known as hyperpycnal flow to occur in front of the Kelantan River mouth. This phenomenon happened when the medium to coarse sediments transported in water was heavier than the energy flow, which caused the sediments to fall to the bottom and continue with the rolling movement on the seabed (Parsons et al., 2001). The changes of sediment distribution reflected the impact from the integration of wave energy degree, water turbulence and the hydrodynamic processes that occurred during the transportation process. The implication of these integration processes generated the variable of sediments facies, demonstrated by the coarse grain size deposited in the delta areas (see Figure 2.1).

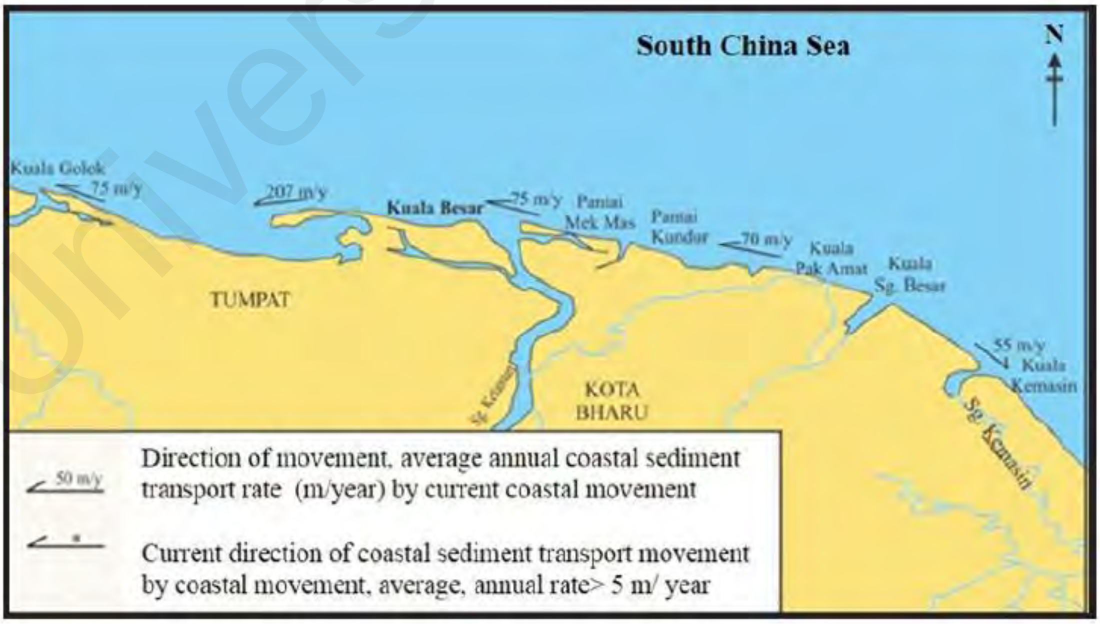

Figure 2.1: Illustration of coastal currents movements along the Kelantan Delta and nearshore area (Photo sourced from Radzir et al., 2018).

CHAPTER 3: METHODOLOGY

3.0 Introduction

This study aims to determine the sediment quality at Kelantan river and its nearshore area by assessing heavy metals contamination level at study area. This chapter discusses the study area, in-situ samples collection, preservation, and laboratory analytical methods. The environmental indexes that have been used in this study also discussed in this chapter. In order to achieve research objectives, sampling and analytical techniques are conducted very carefully to minimise sampling errors and biases.

3.1 Study Area

The Kelantan River (latitudes 4° 40' to 6° 12' North, longitudes 101° 20' to 102° 20' East) situated in North East Peninsular Malaysia spans a considerable region of the state of Kelantan (Figure 3.1) and pours into south-west of the South China Sea across the capital city of Kelantan, Kota Bharu, with populace of over 300 thousand in 2010 based on a decade report by the Malaysian Department of Statistics (Milliman & Farnsworth, 2011). The Kelantan River is 25 m deep and 248 km long, with a catchment area of 140 km and 150 km in breadth and length, respectively (Radzir et al., 2016). The mean yearly sediment load and run-off from the river to the sea are 2.5 × 106 t and 1.8 x 1010 m^3, respectively (Milliman & Farnsworth, 2011). The catchment zone encompasses limestone hills, mountains, lowland jungle, and tropical jungle (Basarudin, et al., 2014). Primary tropical jungle chiefly traverses upstream whereas agricultural plantation traverses downstream and midstream. Throughout the dry period from February to September, the South West wind encourages calm sea conditions and eases showers. Throughout the rainy spell from October to January, the North East monsoon spreads wind waves and rainfall (Adnan & Atkinson, 2011).

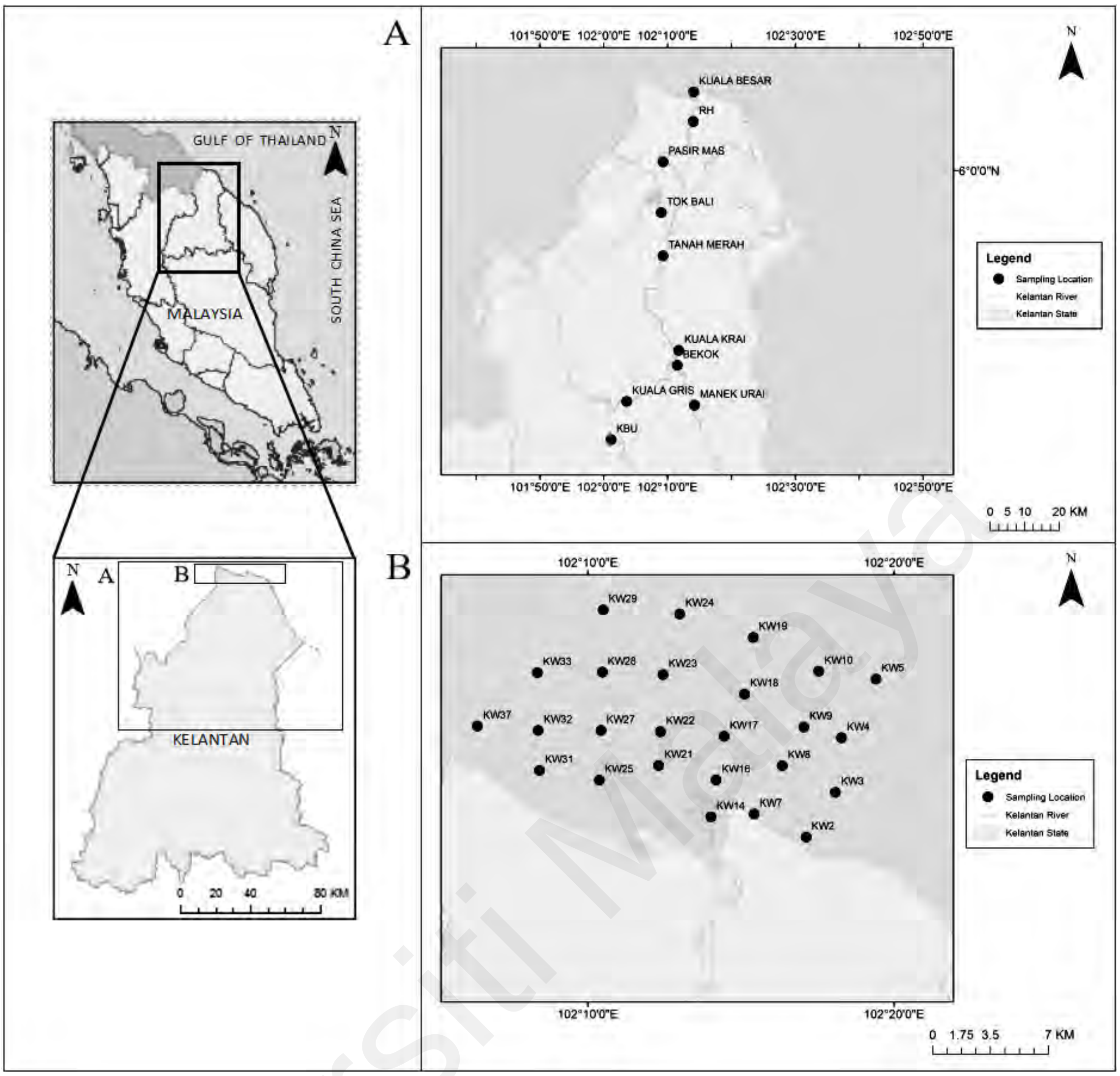

Figure 3.1: Sampling stations along the river and estuary of Kelantan River.

3.2 Collection of Samples

Site inspection was executed to examine adjacent human-based activities and river discharges. In August 2016, surface sediments collected from 10 and 25 selected sites of Kelantan River and its nearshore area. The surface sediments were collected using Van Veen grab sampler that captured approximately top 10 cm of surface sediments (Figure 3.2). Surface sediment samples were collected along the rivers, estuary, and area adjacent to it, nearshore Kuala Besar to represents both marine and land based heavy metals in the study area. The GPS data was recorded, and water quality parameters were measured in-situ at each sampling location using proper hydrolab or measuring equipment. The surface sediment samples were kept and sealed by using zip lock plastic bag which then, transferred to an ice box filled with ice cubes to the lab in order to avoid any microbial degradation of organic compounds during transportation and will be stored at - 20 ºC in freezer before further analytical processes.

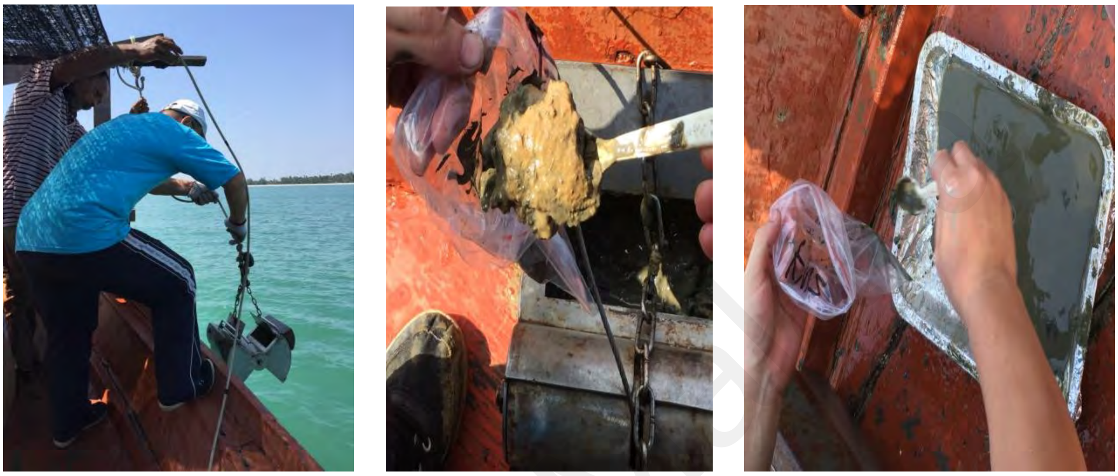

Figure 3.2: Surface sediment samples were captured using Van Veen grab sampler.

3.3 Digestion of Samples

The metal contents in digested sediment samples were analyzed by referred to the published methods (Chen, et al., 2001). Surface sediment samples were allowed to oven dried at 40 °C until a constant dry weight reached. A clean dry pestle and ceramic mortar were used to ground the oven dried sediment samples for homogenization purpose. Then, the homogenized sediment sample were allowed to sieve through a 63 µm mesh sized stainless steel sieve. Any unwanted debris in the form of gravel, coarse debris and others were removed manually by hand using stainless steel or plastic forceps before proceeding further analysis (Kamaruzzaman, et al., 2011).

The sediment samples were digested in a fume hood using a conventional aqua regia digestion method in this study (Chen, et al., 2001). About 0.5 g of homogenized sediment samples weighed and transferred into a 250 ml glass beaker for the digestion. A 12 mL combination of ultra-pure acids with the ratio 3:1 (37 % HCl: 70 % HNO3) added to the sediment samples in the beaker. Then the beakers were covered by using watch glasses to avoid losses of volatile elements (Sastre et al., 2002). Then, the beakers were heated at 110 °C for 3 hours on a hotplate and was brought up to near 2 - 3 ml remaining in the beaker. After completed the digestion process, the digested samples were allowed to cool down at room temperature. Once the samples cooled down at room temperature, the digestate that produced at the end of digestion were filtered by using Whatman no. 42 filter paper in a volumetric flask. Then, the solution was transferred into a polypropylene test tube and diluted up to 50 ml using Ultrapure water and stored for further analysis.

3.4 Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-OES)

The samples were analyzed for metals concentration (As, Cd, Cr, Cu, Mn, Ni, Pb, and Zn) by using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) at the Hydrogeology Lab, Department of Geology, University of Malaya, and the concentration of metals in sediments then were calculated by several methods and calculations. The similar procedures were carried out for a blank sample and also for the standard samples (Ashraf, et al.,2011). To assess the impurity, a blank solution that does only contain reagent were prepared. The detection limit of the ICP-OES instrument for all the analysed metals in this study ranged based on the following terminology: Cd, Cr, Cu, Mn, Ni and Zn (< 0.1 parts per billion), As and Pb (1 - 10 parts per billion). A recovery test was conducted to ensure the accuracy of the results (Ogunkunle, et al., 2014). In this recovery test, in order to compare the measured values with the certified values, a Buffalo River Sediment Reference Material (SRM 8704) of sediments was used. The equation that has been used to calculate the final results of heavy metal concentration is as follows:

$$C = \frac{\mathrm{A \times V \times DF}}{W} \tag{3.1}$$

C: Actual concentration (mg/kg)

A : The measured concentration of the sample (mg L^{-1})

V : Final volume of the collected sample (L)

DF : Dilution Factor

W : Dry weight of the sample (kg)

3.5 Physicochemical Characterisation

3.5.1 Particle size analysis (PSA) / Textural Analysis

Particle size analysis is an established laboratory procedure in determining the particle size distribution of a soil or sediment. The grain size determinations in sediment were carried out using a Malvern Mastersizer, UK (model MSS Hydro2000MU) instrument at Geology Department, UM as described in detail by Jamil et al. (2010). Sediment comprises an accumulation of various shapes and sizes of particles and the objective of the grain size analysis is to group them according to the range of sizes in order to determine the relative proportion by weight in percentage. Laser diffraction method is an appropriate method for samples containing more than 10 % of fine sediments. The particle size analysis was carried out and modified based on method proposed by Folk (1980). The method involves the soaking of the sediment samples in a 2 % Calgon solution for about 15 to 20 hours at least to disperse the particles in the sediments. Before starting the analysis, a Calgon solution was prepared. In order to prepare a 2 % of Calgon solution, about 20 g of sodium hexametaphosphate powder was weighed using laboratory weighing scale and measured. The measured sodium hexametaphosphate powder was dissolved in 1000 mL volumetric flask and was topped up to 1 L by using ultra-pure water and was left overnight. After that, an approximately 3 g of dried sediment sample weighed and transferred into a 15 mL centrifuge tube. The prepared calgon solution was pipetted into the centrifuge tube that has sediment sample in it. The centrifuges tubes were shaken manually by hand and left overnight in a centrifuge tube racks for at least 24 hours to let the dispersion of the particle takes place. The samples were then allowed to analyze in particle size analyzer, PSA Malvern Mastersizer, UK (model MSS Hydro2000MU) to obtain grain size data (Figure 3.3). The characteristics and distribution as well as the texture of sediment grain size was classified according to Wentworth size class.

Figure 3.3: Preparations of sediment samples for particle size analysis using Malvern Mastersizer.

3.5.2 Loss on ignition (LOI)

Loss on ignition method (LOI) is commonly practiced and ubiquitous method which is used to determine the sediment properties such as water content, organic carbon, inorganic carbon, and minerogenic residue (Hoogsteen et al., 2018). This method is widely used due to instantaneous and cheapest property compared to all the other methods employed for determining some of those parameters (Gaidoumi et al., 2019; Ivezic et al., 2016; Dean, 1974 & Maher, 1998). The LOI provides a fast and inexpensive means of determining carbonate and organic contents of clay-poor calcareous sediments and rocks with precision and accuracy comparable to other, more sophisticated geochemical methods (Gaidoumi et al., 2019; Ivezic et al., 2016). Sediment is mostly made up of a mixture of organic material, clastic silicates, oxides (sand, silt, and clay portions), carbonates and water. The LOI method can be used for analyses that run a large number of samples since it is fast and inexpensive (Dean, 1974; Bengston & Enell, 1986). Despite of few uncertainties that could limit the precision of the method, this LOI method provides a rough indication of the organic matter and carbonate contents and a good correlation with the organic carbon content. Studies shows that Loss-on-ignition (LOI) is an accurate measurement of primary organic content of sediments, if the fine fraction is present in low percentages. However, there are number of factors that influenced the LOI results such as sample size, exposure time, position of the sample in the furnace. Sediment composition seems to be the main cause, which determines the value of correction factor between loss-on-ignition and total organic carbon. If an absolute organic carbon determination is to be done, loss on-ignition data must be sustained by organic carbon determination. At the ignition of 475°C for 3 hours, the conversion of organic matter into gases occur resulting in no mass change in the mineral matter (Plater et al., 2015). At a high temperature ranges between ~200 ºC and 500ºC, the organic matter is oxidized to carbon dioxide and combustion of organic matter took place (Mustoe, 2016). The evolution of carbon dioxide from carbonate mineral forms occurs at temperatures between approximately 700 ºC and 900 ºC which known as organic carbon combustion. The loss on ignition corresponds to the mass difference between before and after calcination and represents the organic matter content (Mustoe, 2016; Munroe, 2019). Studies have refined the LOI method for obtaining a more accurate determination of organic matter content (Touch et al., 2017). Studies refined that the LOI method to provide a more accurate determination of organic matter content in terrestrial soils (Salehi et al., 2011). It is known as a simple and economical method where it practices a dry combustion method, that determine direct the amount of organic matter since for past few decades (Cambardella et al., 2001; Konen et al., 2002).

In the determination of organic matter content through the loss-on-ignition (LOI) method involves the heated destruction of all the organic matter in the sediment (Gustitus et al., 2021). The sediment samples were oven dried with the temperature of 40 °C and grounded by using pestle and mortar before allowed to pass through 2 mm mesh size sieve in order to remove coarser particles. About 5 g of oven dried sediment sample was weighed using laboratory weighing scale and an empty ceramic crucible or any other similar vessel's weight also were recorded down. Then, the oven dried sediment sample was placed in that ceramic crucible and the weight of ceramic crucible together with the sediment samples were also recorded as W_1. The sediment samples in crucibles were placed in a furnace at a temperature of 500 °C for 4 hours. Before removing the crucibles from the furnace (after 4 hours), the furnace door was opened to allow some of the heat to disseminate and the crucibles were carefully removed with long handled tongs and were placed onto an asbestos mat. When they have cooled down slightly, the samples were placed in a desiccator and allowed to cool down fully and re-weighed. The weight of the samples was recorded as W_2. The calculations of organic matter content were determined by measuring the differences between the initials and final weight of sediment sample divided by the initial weight times by 100 % (Equation 3.2). All weight should be corrected for moisture or water content prior to organic matter content calculation. The equation used to calculate organic matter content percentage is as shown below (Dra et al., 2019 & Sultan, 2009).

$$LOI = ((W_1 - W_2)/W_1) * 100 \tag{3.2}$$

In short, the total organic matter content (OM) was estimated using the loss on ignition method (LOI) as shown in the above equation. This LOI method was chosen based on simplicity, cost efficiency and less labor intensity (Abubakar, et al., 2018). This method requires the dry combustion of organic matter and consecutive estimation of mass loss due to loss on ignition (Barille´-Boyer, et al., 2003).

3.5.3 pH

The pH is degree the concentration of hydrogen ion in the water. It is very essential to measure the pH in a enclosed system at constant temperature in order to avoid the contamination by atmospheric carbon dioxide. The pH value of sediment samples was evaluated manually according to McLean (1982) as the following steps. A 10 g of sediment was added with 25 ml of distilled water in a glass beaker, according to solid/liquid ratios with 1:2.5 ratio. The glass beaker was then covered with a plastic film and placed in an orbital shaker for 4 hours at 175 rpm. A digital electrode pH meter (Model WTW ph 330) used to read the pH reading as shown in Figure 3.4 (Bing et al., 2016).

Figure 3.4: Image showing sediment being analysed with digital electrode pH meter (Model WTW ph 330).

3.6 Quality Control/Quality Assurance (QA/QC)

To make sure that there was no contamination in the process, the blank solution was prepared in each batch of the experiment. All the sediment samples for the analytical determination of As, Cd, Cr, Cu, Mn, Ni, Pb and Zn concentration were analyzed with reagent blank at an interval of every 10 samples to get rid of equipment drift and to make the reading of instrument at zero. Replicates of samples also were analyzed to estimate the precision of the analytical techniques.

All the glasswares that used for the digestion process were cleaned under running tap water and soaked in 10 % (v/v) of Nitric acid (65 % HNO3 Sigma-Aldrich, Darmstadt, Germany) for about 24 hours in order to minimise contamination from impurities. After that, the glasswares were rinsed with distilled water followed by 0.5 % (w/v) Potassium permanganate (KMnO4). Then, the distilled water was used to rinse all the glasswares prior to dry it in an electrical drier (Yap, et al., 2002).

The Inductively coupled plasma atomic emission spectroscopy (ICP-OES) was calibrated using internal standards for all the heavy metals (As, Cd, Cr, Cu, Mn, Ni, Pb, Zn) to obtain a calibration curve. The standard solutions of each metal were prepared from 1000 mg/L (BDH Spectrosol®) stock solution and multiple level calibration standard was developed. The precision and accuracy of the experimental method was also checked through the calibration curve that produced by the standard solution.

The Certified Reference Material (CRM) is known as a reference material that has been certified by a standard procedure in which it established the traceability to an accurate recognition of the unit where the property values are stated and also for each certified value comes with an uncertainty statement expressed as a stated level of confidence. The property value of CRMs is commonly determined within a known uncertainty limits by measuring the sample representative for the whole batches. The analysis accuracy was compared to the Buffalo River Sediment Reference Material (SRM 8704) sourced from the National Institute of Standards and Technology (US) to compare the quality of total acid digested sediment and the percentage ranged from 82.60 % to 101.78 % (Table 3.1).

Table 3.1. Recovery test using Buffalo River Sediment Reference Material (SRM 8704).

Element	Analyzed value (mg/kg)	Certified value (mg/kg)	Recovery (%)
Pb	152.67	150	101.78
Cd	2.43	2.94	82.60
Mn	452.72	544	83.22
Cu	77.70	87	89.31
Zn	350.99	408	86.03
As	14.98	17	88.12
Ni	37.28	42.9	86.89
Cr	101.17	122	82.92

3.7 Environmental Indices

3.7.1 Pollution load index (PLI)

Pollution load index (PLI) is an approach that provides an understanding on the quantity of polluting component at the studied environment, and it is an easier yet a comparative method for assessing a site's quality (Mohamed et al., 2014). An integrated approach of pollution load index (PLI) of the eight metals were calculated to evaluate the sediment quality (Tomlinson et al., 1980). Contamination factor is expressed as the ratio of mean of measured concentration with the average shale concentration given by Turekian & Wedepohl (1961), which is employed as global standard reference for uncontaminated sediment (Equation 3.3). The PLI for each sampling site is the n th root of n number multiplying the contamination factors, *CF* (Equation 3.4) altogether. The PLI value for each single site was calculated as follows:

$$CF = C_{metal} / C_{background}, \tag{3.3}$$

$$\text{PLI} = n\sqrt{(\text{“}CF\ 1\ \times\ CF\ 2\ \times\ CF\ 3\ \times\ \ldots\ldots\ldots\ CF\ n\text{”})}, \tag{3.4}$$

Where;

CF = Contamination factor

C_{metal} = Mean metal concentration at contaminated site

$C_{background}$ = Metal average shale concentration (Turekian and Wedepohl, 1961)

n = number of metals

The overall toxicity status of the sample and the results of the contribution of the eight metals are assessed by the PLI method. The contamination factor is the ratio of the measured concentration to natural abundance of a given metal which monitored the pollution of one single metal over a period time. (Turekian & Wedepohl,1961; Loska et al., 1997). The PLI value are classified into two categories where PLI value > 1 indicates that the area is polluted condition and < 1 indicates no polluted condition (Tomilson et al., 1980).

3.7.2 Potential ecological risk index (PERI)

To assess the effect of ecorisk by the multiple metals in sediments from the Kelantan river and its nearshore area, the potential ecological risk was calculated. The Potential Ecological Risk Index (PERI) was originally developed by Hakanson (1980) which is widely being used in ecological risk assessment of heavy metals in sediments. It is also used to evaluate aquatic pollution control which has been widely applied in assessing heavy metal contamination of sediment samples (Hong et al., 2004). The Potential Ecological Risk Index, PERI (Equation 3.6) was evaluated by considering the content and the toxic response factors of trace elements in the sediment samples in order to assess the potential ecological risk of an environment (Kusin et al., 2016). According to this methodology, the potential ecological risk index (PERI) was defined using the following equation:

$$C^i_f = C^i_{surface} / C^i_{reference} \tag{3.5}$$

Where;

C^i_f = pollution coefficient of a heavy metal

$C^i_{surface}$ = present concentration of heavy metal in the sediment

$C^i_{reference}$ = concentration of heavy metal in reference natural background (Turekian &Wedephol,1961

$$PER = \sum_{i=}^{n} \mathrm{E}^{\mathrm{i}}_r \;\; ; E^i_r = C^i_f \times T^i_f, \tag{3.6}$$

Where;

E^i_r = potential ecological risk index value of a single heavy metal pollution

C^i_f = pollution coefficient of a heavy metal

T^i_f = response coefficient for toxicity of a single heavy metal

The potential ecological risk index that used for the study area ranges based on the following terminology: low ecological risk (PER < 150), moderate ecological risk (150 ≤ PER < 300), considerable ecological risk (300 ≤ PER < 600), and very high ecological risk (PER ≥ 600).

3.7.3 Geo-accumulation index (Igeo)

The geoaccumulation index (Igeo) is a common method to estimate the enrichment of metal concentrations above background or baseline concentrations that been proposed by Muller, 1969. The Igeo method also known as a quantitative approach in measuring the heavy metal contamination in sediment. The basic principle of this geoaccumulation index (Igeo) is that it works as a single metal approach that quantify metal pollution in sediments when the concentration of heavy metal is 1.5 or more higher than their lithogenic background values (Gaur et al., 2005).

Geoaccumulation index (Igeo) assesses current heavy metal concentrations against pre-industrial concentrations according to Muller (1969) based on classification by Huu et al. (2010). The equation to determine the Ige o value was as follows:

$$\text{Igeo} = \log_2 [Cn / 1.5 * Bn] \tag{3.7}$$

Where;

C*n* = measured concentration of element *n* in the sediment

Bn = geochemical background value element n in average crust (Turekian & Wedepohl, 1961)

The factor 1.5 was used to compensate potential variations which might be attributed to lithogenic effects in the sediments (Equation 3.7). The calculated Igeo values were classified according to the description of sediment quality of Igeo classification table by Muller, 1979 to deduce the pollution status of the sampling sites at the Kelantan river and nearshore areas (Table 3.2).

Table 3.2: Description of sediment quality Igeo classification (Müller, 1979).

Geoaccumulation index (Igeo)	Class	Quality of Sediment
< 0	0	Uncontaminated
0 - 1	1	Uncontaminated to moderately contaminated
1 - 2	2	Moderately contaminated
2 - 3	3	Moderately to highly contaminated
3 - 4	4	Highly contaminated
4 - 5	5	Highly to very highly contaminated
> 5	6	Very highly contaminated

3.7.4 Enrichment factor (EF)

Enrichment Factor (EF) is commonly used in the literature as a way of identifying and quantifying human interference with global element cycles. The concept of normalizing element concentrations to an average crustal value which are discussed in detail by Wang et al., (2017). The EF calculated to diminish the metal variability affiliated with variations in mud/sand ratios. EF also act as a convenient tool to plot a geochemical trend over a large geographic area which possibly have substantial variations in the mud to sand ratios. The EF values were calculated for each element to estimate the anthropogenic impact on heavy metals in sediment. In this study, Al was used for normalization in order to eliminate the grain size effect (Wang et al., 2017). The EF values of the heavy metals analyzed in this study were calculated using the following equation as shown in Equation 3.8 (Sutherland, 2000):

$$EF = \frac{\left({}^{Me}/_{Al}\right)\ sample}{\left({}^{Me}/_{Al}\right)\ reference} \tag{3.8}$$

Where;

$(Me/Al)_{sample}$ = ratio of the metal (Me) to Al in samples of interest

$(Me/Al)_{rreference}$ = ratio of metal and Al concentrations of background

The EF values were divided into different categories of contamination. The classification of EF values are as follows: EF < 2, deficiency to minimal enrichment; EF = 2 - 5, moderate enrichment; EF = 5 - 20, significant enrichment; EF = 20 - 40, very high enrichment; and EF > 40, extremely high enrichment.

3.8 Correlation of Physicochemical Characteristics and Heavy Metals

Statistics analysis was performed using IBM-SPSS software version 20 (International Business Machines Corporation, USA). The Pearson test was performed to identify significant correlation between heavy metal concentration, organic matter content, pH value, as well as percentages of clay, silt, and sand. The spatial distribution of heavy metal concentration and physicochemical properties of the sediment were performed by using software ArcGIS 10.3 by using the Inverse Distance Weighted (IDW) method (Zhu et al., 2018).

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Physicochemical Characterisation

4.1.1 Particle size analysis (PSA)

Table 4.1 shows the results of all physicochemical analysis (pH, LOI, content of clay, silt and sand). The sediment grain size and texture analysis along the Galas River (Kuala Gris and KBU) and Lebir River (Manek Urai and Bekok), the riverbed sediment is mainly composed by silt. The sand content gradually decreased from upstream to the downstream in overall (Figure 4.1a). Similarly, the overall clay content reduced moderately from the upstream to downstream and found to be peaked at the Kuala Krai and Bekok stations which is the confluence of both Galas and Lebir river. On the other hand, the overall silt content elevated gradually and the highest was recorded at the Tanah Merah station that contributed 70% to the total sediment composition at that area.

In general, the overall pattern for sediment composition were as follows: silt>sand>clay (Figure 4.1b). The surface sediments at the river channel are mainly dominated by silty clay type of sediments. On the other hand, the surface sediments at the nearshore area were mainly consisted of silt type of sediment. The average percentage of silt is 72.11 % from overall sediment composition, followed by sand and clay with average percentage of 34.67 % and 17.34 % respectively (Figure 4.2). The highest sand content was present at the KW33 station and the lowest was found at the KW2 station. The sediments that near to shore (station KW2 & KW7) were mainly consists of sand and the clay content increased overall with increasing distance from the shore. The texture distribution patterns showed that most of surficial sediments at the nearshore were dominated by clayey silt and the sand content increased as the distance increased from the shore. Regions with denser human population, such as towns, urban districts (Tanah Merah), the capital city (RH, Pasir Mas), and along the Lebir River, harboured higher clay content thus comprised the silty clay type of sediment composition. Other areas along the Kelantan River typically consisted of the silt type of sediment composition. Areas near industrial, residential, fishery, agriculture or aquaculture activities have also been reported to contain relatively more clay than areas dominated by other activities (Sekabira et al., 2012). Furthermore, logging have been occurring at the upstream areas of the Galas River and Lebir River. Also, local rainfall typical of the tropical rainforest climate has an average annual rainfall of 2000 mm reaching 5000 mm during monsoon monsoon (Malaysian Meteorological Department, 2016). The logging activity could contribute to excessive silting and discolouration of the Kelantan River, which is exacerbated by frequent tropical showers (Chakravarty et al., 2012). Since heavy metals are absorbed onto clays and other fine-grained materials, the sediment of the Kelantan River that is abundant in clay and silt could become carriers of heavy metals (Ho et al., 2010).

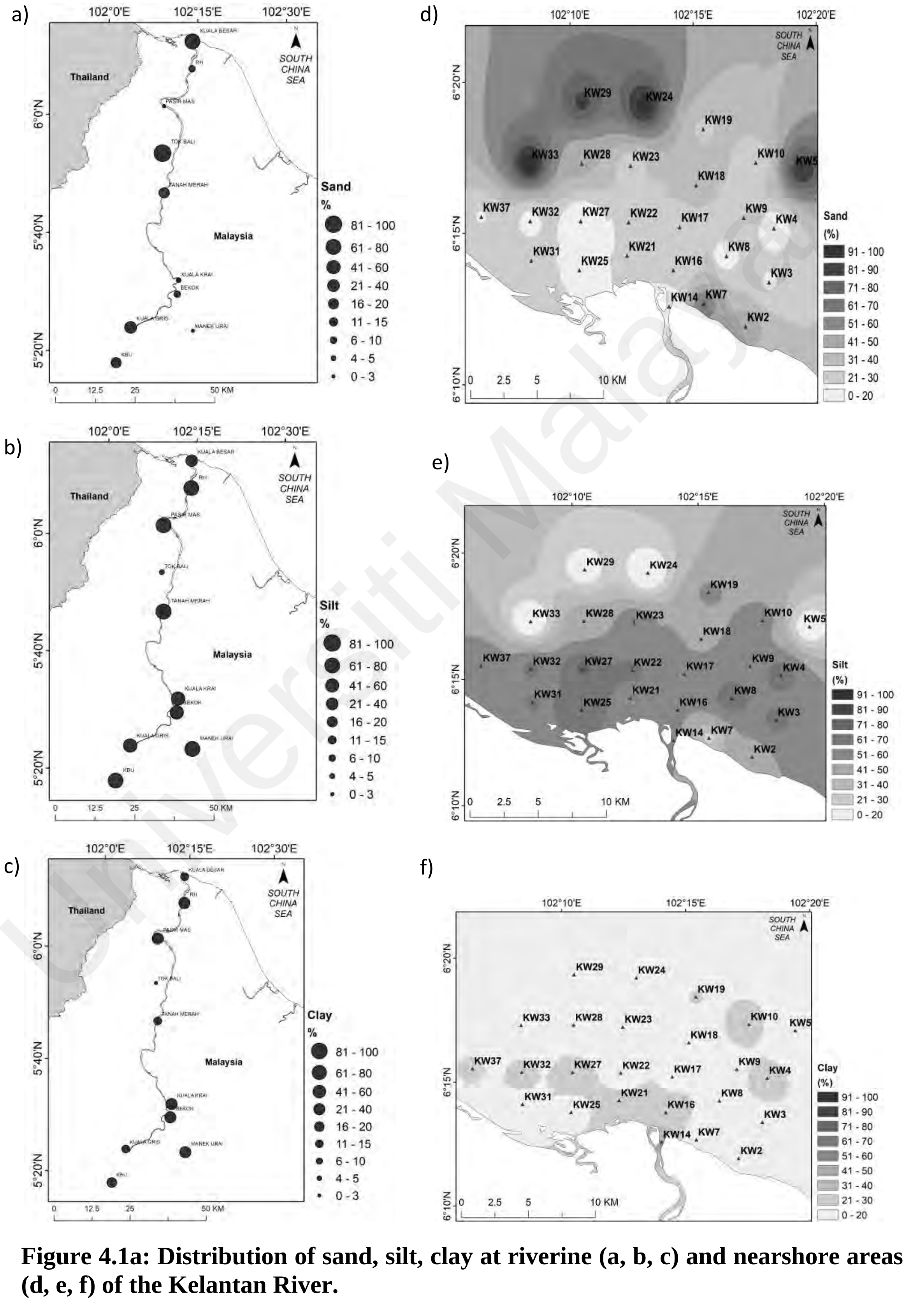

Figure 4.1a: Distribution of sand, silt, clay at riverine (a, b, c) and nearshore areas (d, e, f) of the Kelantan River.

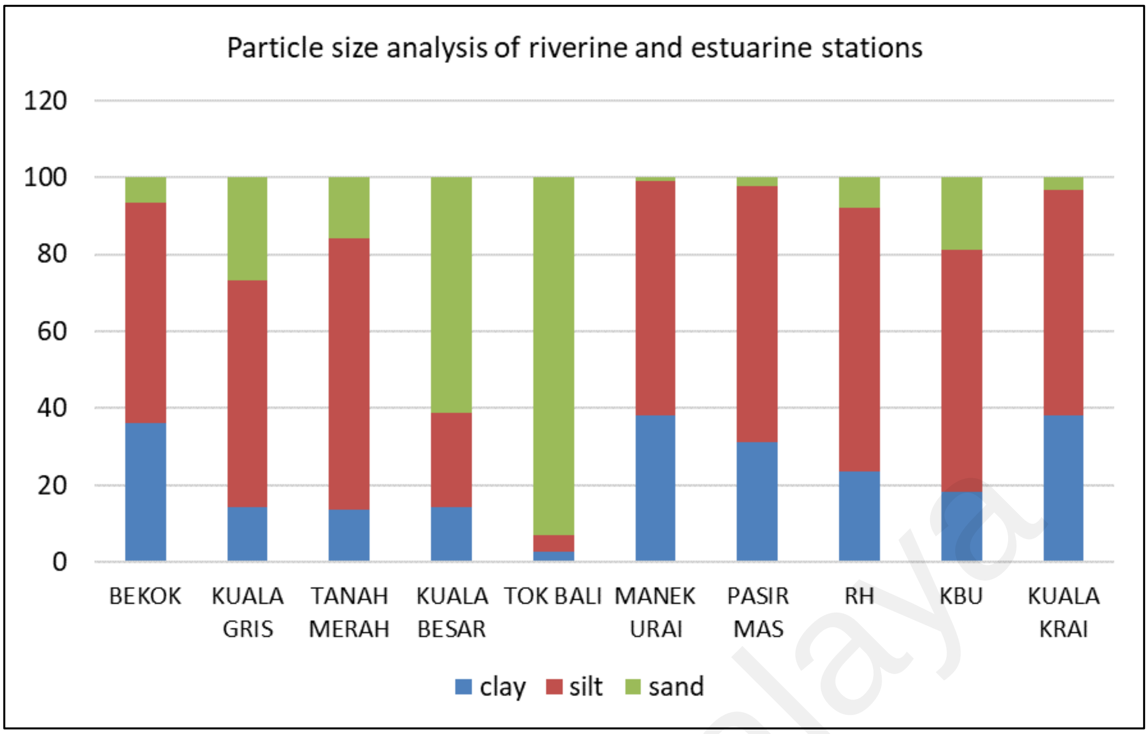

Figure 4.1b: Particle size analysis of surface sediments of riverine and estuarine stations.

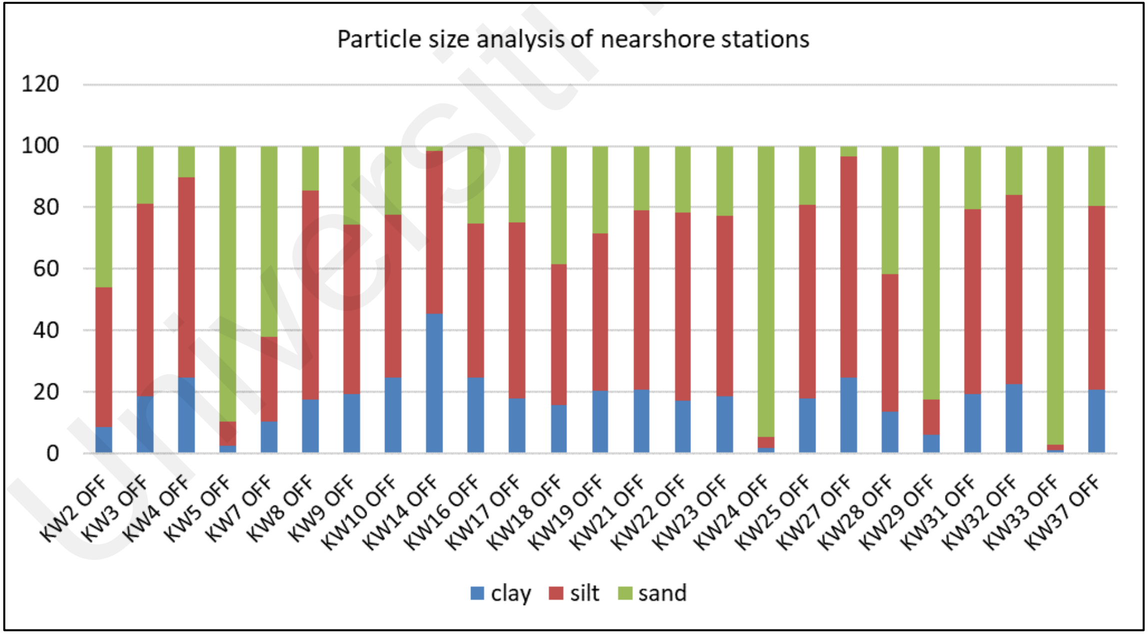

Figure 4.2: Particle size analysis of surface sediments of nearshore stations.

4.1.2 Loss on ignition (LOI)

The LOI computed in this study represents the organic matter content, which can be utilised for qualitative evaluation of the depositional nature of sampling locations (Schumacher, 2002). The organic matter content in surficial sediment ranged from 0.17 % to 1.95 % with an average of 0.94 % at the Kelantan river and ranged from 0.16 % to 3.50 % with an average of 1.84 % at the nearshore. The organic matter content decreased gradually from the upstream of the river to the downstream and the enrichment occur from the estuary (Kuala Besar station) to nearshore stations KW25, KW32 and KW37 with 3.36 %, 3.11 % and 3.50 % of organic matter content (Figure 2d and 4d). High organic depositions were detected in surface sediments at the capital city, confluence, upstream areas of both tributaries, and adjacent shelf areas and Wang et al. in 2017 also detected higher organic matter content along the adjacent shelf areas. Sediment rich in organic matter content formed due to aggregation of organic compounds onto the clay minerals in the water columns that deposited in an environment with low oxygen tension (Premuzic, et al.,1982; Hedges et al.,1997). Biological processes and microorganisms include bacteria, fungi, protozoans, plankton, decaying remains of organisms, fecal matter and also marine and terrestrial plant debris, or, from a biochemical standpoint, proteins, carbohydrates, lipids and pigments results in formation of in situ biological component (Luthy et al.,1997).

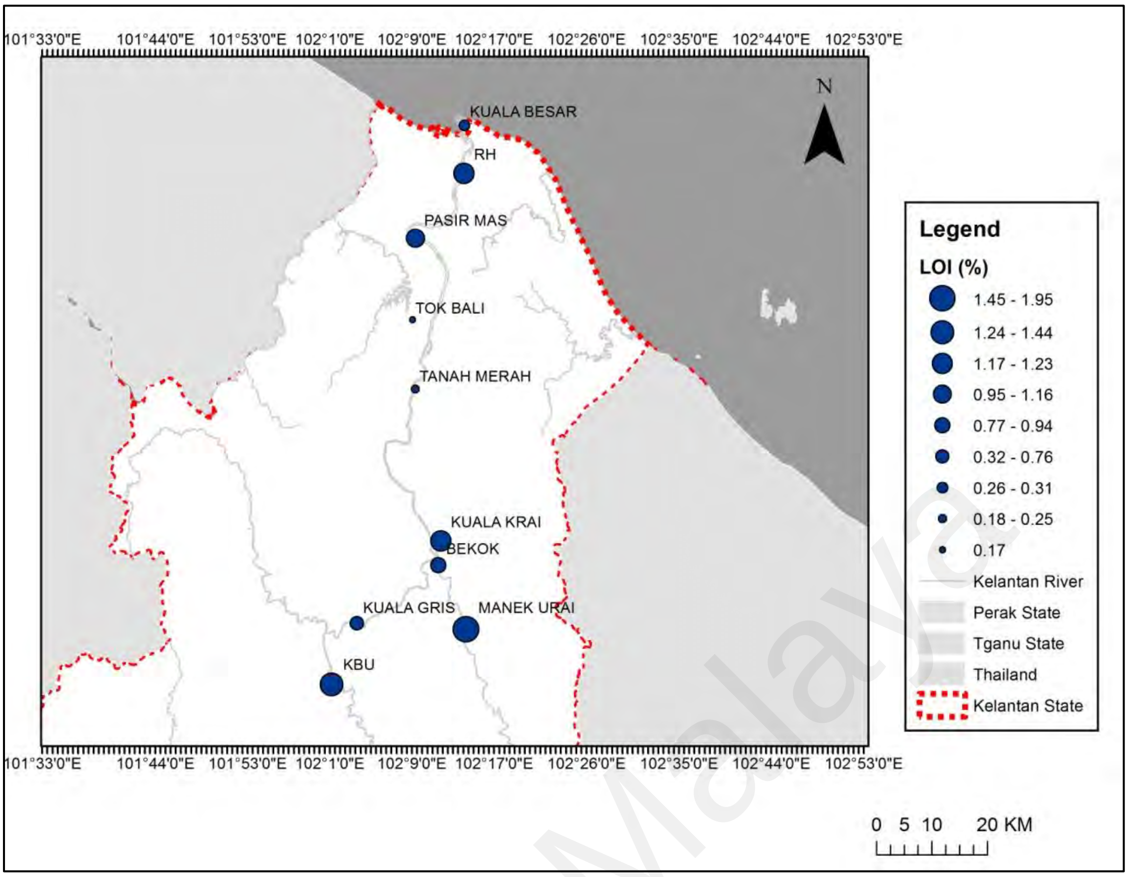

Figure 4.3: Loss on ignition (LOI) of surface sediments of riverine and estuarine stations.

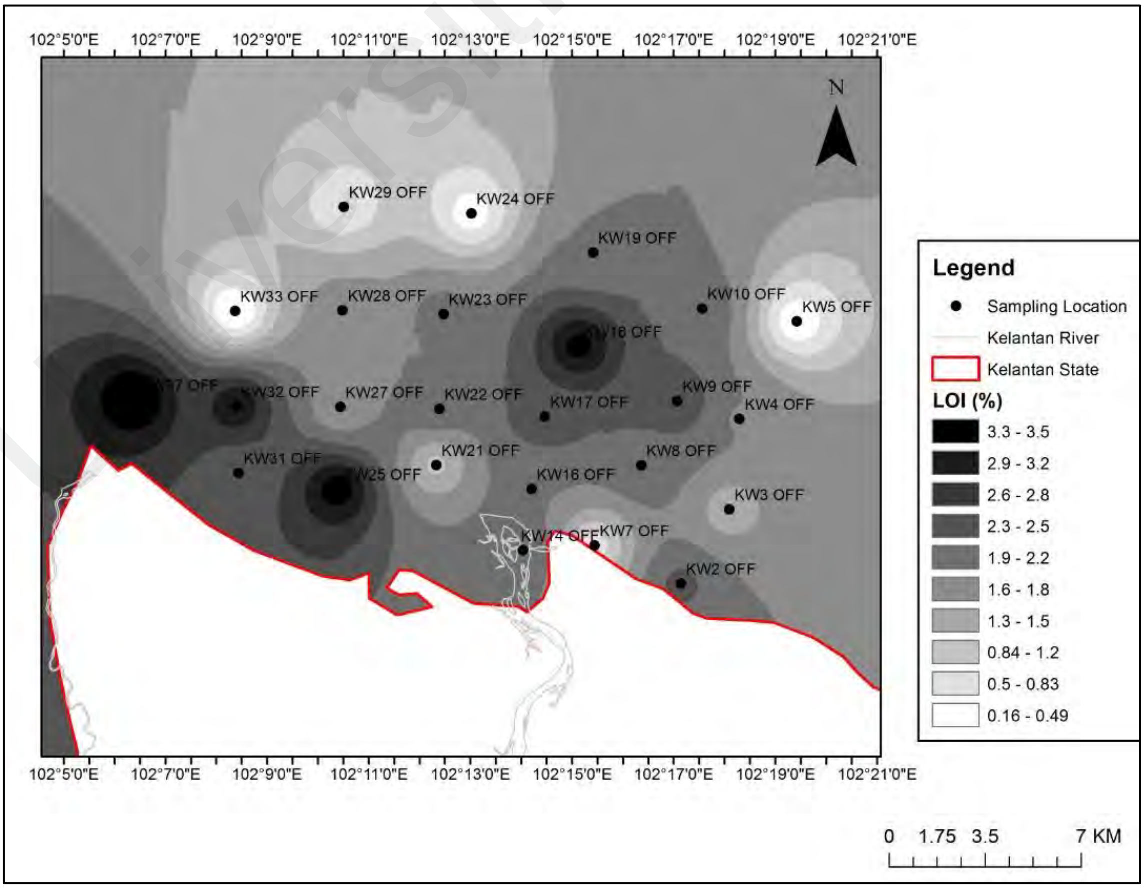

Figure 4.4: Loss on ignition (LOI) of surface sediments of nearshore stations.

4.1.3 pH

Neutral to mild acidic pH were discovered at the estuary, capital city, midstream, confluence, and tributaries, whereas alkaline pH were recorded at coastal marine areas and Tanah Merah, which is a midstream urban district (Figure 4.5 and 4.6). The pH values from 6.24 to 8.72 for Kelantan river with a mean pH of 7.0 whereas ranged from 6.80 to 8.39 with a mean 7.84 for nearshore areas indicating acidic to neutral nature of Kelantan river environment. The observed pattern can be explained since the pH in saltwater environments is usually mildly alkaline but acidic in anthropogenically populated riverine regions (Kulthanan et al., 2013). The acidic pH was caused by naturally occurring silicate minerals. Overall, the pH values were below the acceptable limits (6.5 - 8.5) for drinking and other domestic purposes (WHO, 2011).

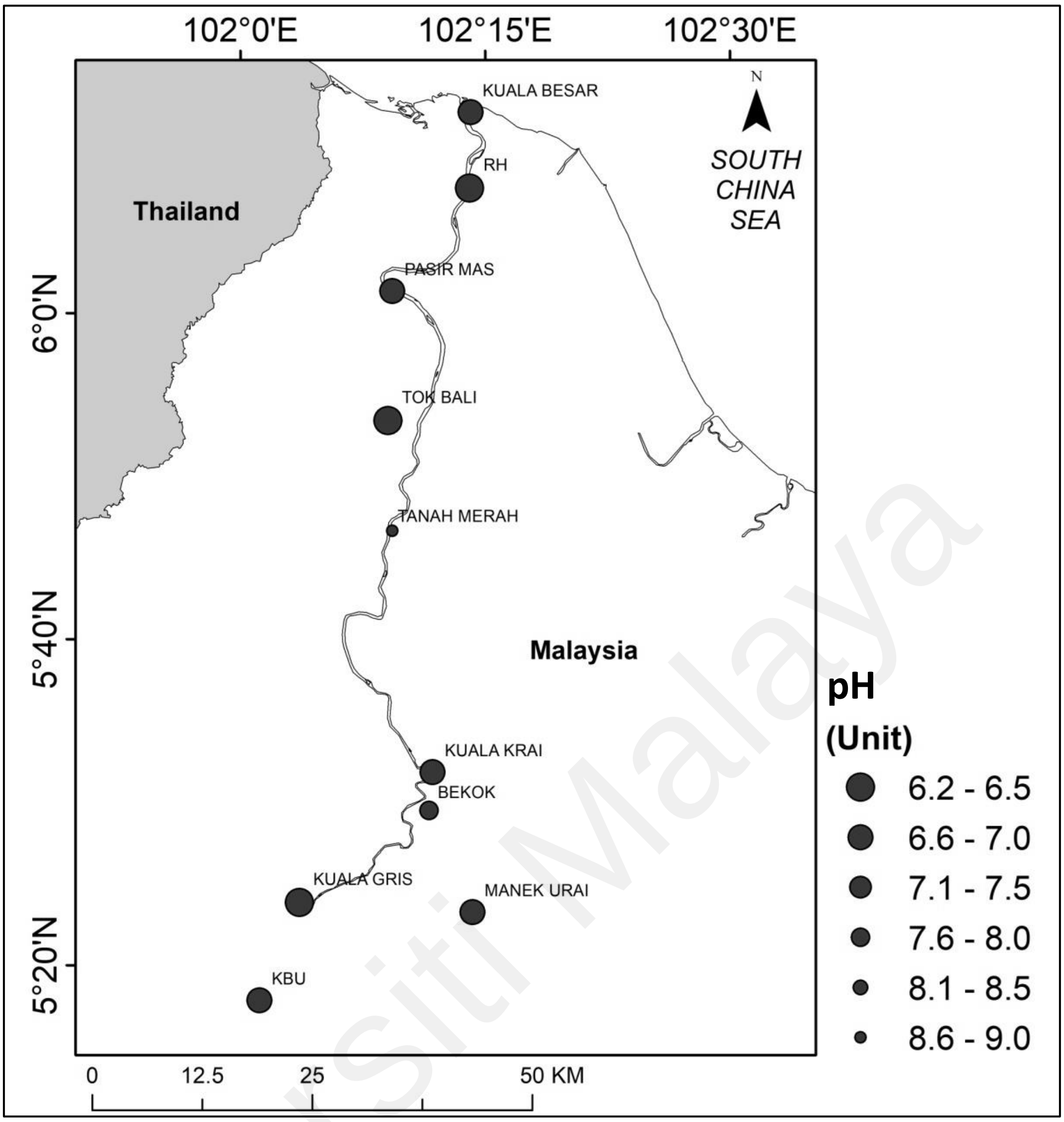

Figure 4.5: The pH of surface sediments of riverine and estuarine stations.

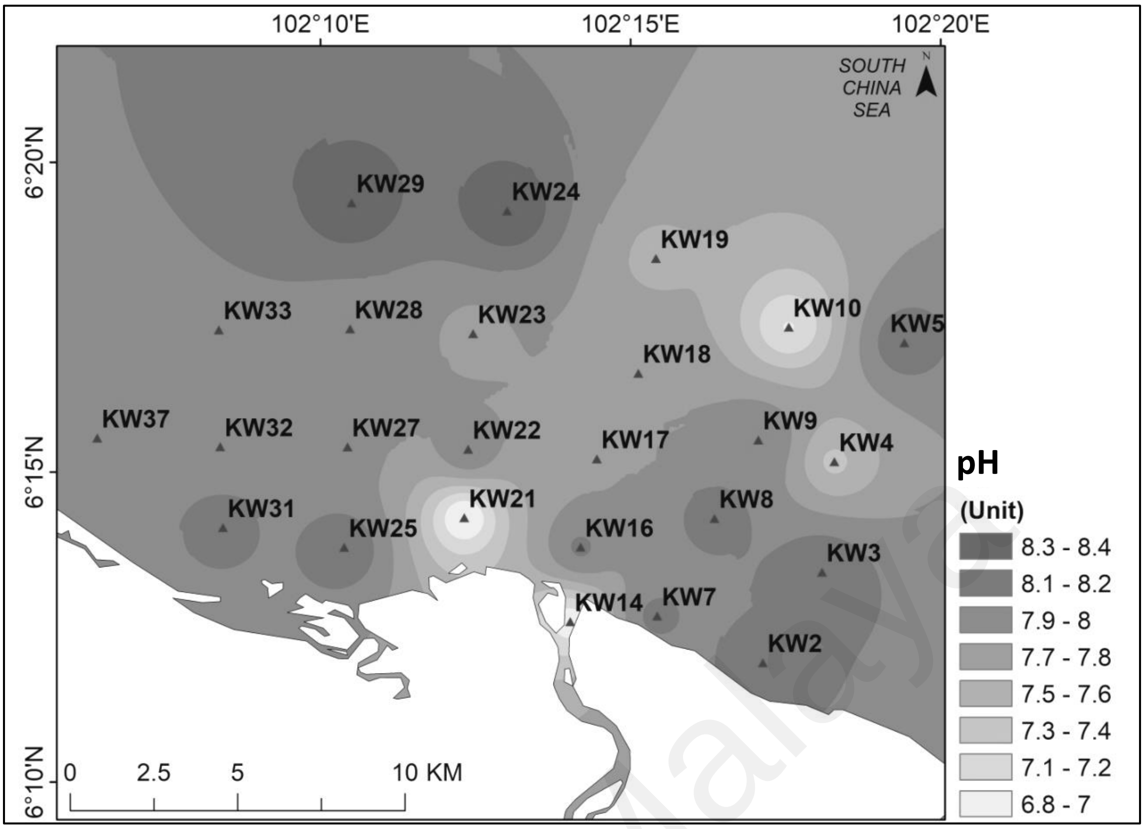

Figure 4.6: The pH of surface sediments of nearshore stations.

Table 4.1: Physicochemical results of all sampling stations at Kelantan river and nearshore area.

			Percentage %		
Station	**pH**	**LOI (%)**	**Clay**	**Silt**	**Sand**
BEKOK	7.53	0.94	36.11	57.22	6.67
KUALA GRIS	6.49	0.76	14.27	59.07	26.67
TANAH MERAH	8.72	0.25	13.52	70.62	15.86
KUALA BESAR	6.95	0.31	14.30	24.67	61.03
TOK BALI	6.24	0.17	2.55	4.30	93.15
MANEK URAI	6.92	1.95	38.08	61.17	0.76
PASIR MAS	6.93	1.16	31.35	66.45	2.20
RH	6.32	1.22	23.72	68.33	7.94
KBU	6.93	1.44	18.21	63.08	18.71
KUALA KRAI	6.99	1.23	38.29	58.35	3.36
KW2 OFF	8.13	2.24	8.60	45.54	45.86
KW3 OFF	8.19	1.31	18.78	62.47	18.74
KW4 OFF	7.36	1.81	24.69	65.13	10.18
KW5 OFF	8.12	0.26	2.37	7.83	89.80
KW7 OFF	8.05	0.67	10.26	27.70	62.05

Table 4.1, continued.

KW8 OFF	8.12	2.09	17.56	67.85	14.60
KW9 OFF	8.00	2.48	19.23	55.37	25.40
KW10 OFF	6.99	2.07	24.64	53.16	22.20
KW14 OFF	6.89	2.14	45.39	52.94	1.67
KW16 OFF	8.03	1.85	24.52	50.21	25.27
KW17 OFF	7.79	2.44	17.79	57.26	24.95
KW18 OFF	7.74	3.28	15.58	46.14	38.28
KW19 OFF	7.54	1.85	20.35	51.16	28.49
KW21 OFF	6.80	1.09	20.70	58.55	20.75
KW22 OFF	7.97	2.01	17.07	61.37	21.56
KW23 OFF	7.74	1.96	18.73	58.45	22.82
KW24 OFF	8.39	0.34	1.70	3.59	94.71
KW25 OFF	8.16	3.36	17.91	63.03	19.06
KW27 OFF	7.85	1.52	24.63	72.11	3.26
KW28 OFF	7.90	1.64	13.52	44.78	41.70
KW29 OFF	8.37	0.56	5.96	11.55	82.49
KW31 OFF	8.06	2.31	19.17	60.28	20.55
KW32 OFF	7.94	3.11	22.57	61.58	15.85
KW33 OFF	8.00	0.16	0.98	2.02	97.00
KW37 OFF	7.86	3.50	20.89	59.61	19.51

4.2 Heavy Metal Concentrations

The average concentration of heavy metals in descending order at Kelantan river and nearshore surface sediment concentrations were as follows: Mn > Zn > Pb > Cr > Cu > As > Ni > Cd, and Mn > Pb > Zn > Cr > As > Cu > Ni > Cd (Figure 4.19 and 4.20).

Table 4.2: Nearshore and riverine concentrations of heavy metals As, Cd, Cr, Cu, Mn, Ni, Pb and Zn.

Stations	Concentration of heavy metals, mg/kg (ppm)							
	As	Cd	Cr	Cu	Mn	Ni	Pb	Zn
BEKOK	8.48	3.49	12.08	9.08	1187.86	4.79	20.86	28.35
KUALA GRIS	4.50	0.00	25.39	11.60	215.76	9.00	21.90	40.99
TANAH MERAH	2.30	0.80	12.58	5.79	287.94	5.29	41.33	23.56
KUALA BESAR	2.50	0.70	9.40	10.10	99.00	3.80	11.40	28.50
TOK BALI	3.79	0.40	2.30	1.40	20.66	1.20	5.89	12.28
MANEK URAI	8.10	4.20	14.49	12.10	610.16	3.60	18.09	28.29
PASIR MAS	11.59	0.70	24.98	14.69	451.45	6.99	24.48	40.16
RH	11.78	2.99	24.15	15.57	473.65	8.48	27.54	44.61
KBU	5.40	0.00	28.90	16.10	369.30	10.50	23.00	41.80
KUALA KRAI	15.40	-0.20	22.80	17.60	1355.73	6.00	31.59	40.99
KW2 OFF	11.02	0.00	20.35	8.04	454.13	6.75	87.97	37.53
KW3 OFF	10.72	13.41	21.25	9.63	395.13	7.35	56.41	41.31
KW4 OFF	12.79	5.30	25.48	16.59	313.55	8.19	74.34	40.17
KW5 OFF	6.86	1.09	6.57	1.39	131.32	2.98	34.32	13.13
KW7 OFF	6.28	2.29	14.65	4.78	402.03	5.28	52.12	36.18
KW8 OFF	10.18	3.49	18.16	8.88	322.06	7.98	36.73	32.44
KW9 OFF	14.70	0.90	25.80	15.80	350.10	9.40	53.70	48.50

Table 4.2, continued.

Stations	As	Cd	Cr	Cu	Mn	Ni	Pb	Zn
KW10 OFF	15.98	35.56	30.97	20.48	258.34	10.69	64.64	51.95
KW14 OFF	16.08	0.10	31.16	21.77	235.22	8.19	55.83	42.25
KW16 OFF	22.36	0.10	33.25	20.07	648.46	10.48	56.81	45.73
KW17 OFF	15.41	15.31	139.76	26.64	474.06	17.40	66.60	45.53
KW18 OFF	16.27	11.08	32.24	20.56	347.50	11.38	84.73	45.71
KW19 OFF	17.00	3.40	33.29	21.80	389.72	13.80	72.29	54.49
KW21 OFF	17.69	0.10	42.37	17.29	401.48	13.19	78.04	42.67
KW22 OFF	17.00	0.00	25.29	12.60	527.49	8.90	55.79	47.09
KW23 OFF	13.10	0.50	25.60	11.80	528.40	9.40	57.00	45.10
KW24 OFF	5.38	3.49	10.76	4.68	195.20	6.38	77.72	16.44
KW25 OFF	19.53	0.10	25.10	12.95	681.31	8.77	58.38	40.45
KW27 OFF	16.93	0.20	27.68	16.73	478.30	11.05	60.93	56.55
KW28 OFF	20.36	1.70	29.74	15.17	469.56	9.48	99.50	41.92
KW29 OFF	2.77	0.10	26.97	2.96	151.72	10.57	42.18	20.55
KW31 OFF	18.07	0.60	30.35	17.27	584.76	12.38	60.50	51.02
KW32 OFF	17.90	0.00	33.90	22.70	398.20	14.90	64.70	67.00
KW33 OFF	9.96	0.60	6.88	4.78	130.03	4.78	39.76	20.03
KW37 OFF	19.16	15.87	27.24	16.26	480.84	11.47	64.26	42.31

Table 4.3: Comparison of average heavy metal concentrations in previous and present studies.

Metal	Average concentration (ppm)	Year	Reference
Pb	20.82	1993	(Ahmad *et al.*, 2009)
	42.22	2014	(Wang *et al.*, 2017)
	50.89	2016	This study
Cd	1.82	1993	(Ahmad *et al.*, 2009)
	0.04	2014	(Wang *et al.*, 2017)
	3.66	2016	This study
Mn	394.00	1993	(Ahmad *et al.*, 2009)
	423.44	2016	This study
Cu	6.74	1993	(Ahmad *et al.*, 2009)
	16.74	2014	(Wang *et al.*, 2017)
	13.30	2016	This study
Zn	18.67	1993	(Ahmad *et al.*, 2009)
	47.63	2014	(Wang *et al.*, 2017)
	38.73	2016	This study
Ni	22.06	2014	(Wang *et al.*, 2017)
	8.59	2016	This study
Cr	56.74	2014	(Wang *et al.*, 2017)
	26.34	2016	This study
As	12.21	2016	This study

4.3 Heavy Metal Distribution

In this study, the highest Pb concentration, 99.50 mg/kg, was discovered at coastal station KW28 situated in front of the estuary (Figure 4.19 and 4.20). The elevated accretion of Pb at KW28 could be affected by the entrapment zone that is conceived from the mixing of saltwater and freshwater (Balachandran et al., 2005). Relatively high Pb level concentrations were identified at the compactly populated capital city, Tanah Merah and Kuala Krai urban districts, since oil spillage, sewage effluent, urban runoff, and boating that were observed at the studied areas could lead to elevated Pb concentrations in adjacent water bodies (Abu-Hilal et al., 1987). Automobile vehicles emissions, paint, pesticides, coal, and mining exploration are the sources of contamination of Pb in the ecosystem (Abbas et al., 2016). Pb has wide usage in many industries, for instance, it is used in manufacturing batteries, ammunition, metal products like pipes, solder and X-ray shielding devices. This had arisen health related concerns, thus, the usage has reduced in past years drastically. The high-level concentration of Pb at study area were observed at boating activities where it recorded a low pH value of sediment (Whitfield et al, 2014). By drinking water, the Pb could enter the human body and causes accumulation at the digestive tract and lungs which then spread to whole body by blood as a carrier. Fatal diseases as cancer, anaemia, nervous system damage, renal kidney diseases and mental retardation can occur in the presence of large quantity of Pb in drinking water (Razak, et al, 2015). Assessment of heavy metal concentrations in the Kelantan River from the present study with previous reports revealed an increasing concentration of Pb, implying the necessity for continuous monitoring of Pb (Table 4.3).

Besides that, relatively greater Mn concentrations were discovered at the confluence, Lebir River and nearshore areas closer to the estuary (Figure 4.15 and 4.16). The Mn concentrations in the Kelantan River was observably inputted from the Lebir River. The Mn concentrations in surficial sediment ranged from 20.66 mg/kg to 1355.73 mg/kg with an average of 507.15 mg/kg at the Kelantan river and ranged from 130.1 mg/kg to 681.31 mg/kg with an average of 389.96 mg/kg at the nearshore. The Mn influx could be caused by logging upstream, which released soil minerals and lessen the quantity of vegetation and trees that absorb Mn for growth. Contrastingly, relatively lower Mn concentration was discovered at the post-ait area. The lower Mn levels can be explained by Mn absorption by the mangrove geographical terrain on the ait, since Mn is an essential nutrient for plant growth (Janaki-Raman et al., 2007).

Furthermore, relatively greater Ni concentrations were discovered along the Lebir River, at the capital city and confluence, including the relatively farther coastal regions (Figure 4.17 and 4.18). The Ni concentrations in surficial sediment ranged from 1.2 mg/kg to 10.5 mg/kg with an average of 5.97 mg/kg at the Kelantan river and ranged from 2.98 mg/kg to 17.39 mg/kg with an average of 9.64 mg/kg at the nearshore. Nonetheless, the relatively constant Ni concentrations alongside the river revealed mostly lithogenic source of Ni metals. Nevertheless, the utilisation of paints, building materials, and agricultural chemicals at adjacent urban regions could aggravate Ni quantity in the environment if without continuous and regular observation of heavy metals (Kabata & Pendias, 1992).

Moreover, relatively greater Cu concentrations were discovered at the upstream tributaries, confluence, riverine area, and capital city (Figure 4.13 and 4.14). The Cu concentrations in surficial sediment ranged from 1.4 mg/kg to 17.6 mg/kg with an average of 11.4 mg/kg at the Kelantan river and ranged from 1.39 mg/kg to 26.64 mg/kg with an average of 14.06 mg/kg at the nearshore. The mid-to-downstream agricultural activities and logging upstream along the Kelantan River could lead to the elevated Cu amounts. According to Shaari and co-workers (2015), Cu is released via forest fire, boating, weathering, soil exposure, and plant decomposition. Cu are discharged to environment through many ways as such chemical and pesticide industry, mining and metal piping. Drinking water consumption is a potential source of Cu into human body. However, in the presence of higher concentration it may be harmful (Abbas et al., 2016). An excessive amount of intake of Cu could impact human and animal's metabolism in altering the blood pressure level and respiratory rates, kidney and liver damage, convulsions, cramps, vomiting or even case death to animals in an aquatic environment (Mziray & Kimirei, 2016).

Also, negligible Cd was discovered alongside the Galas River, suggesting little lithogenic entry from weathering or natural erosion (Figure 4.9 and 4.10). The average Cd concentrations in surficial sediment at the Kelantan river and at nearshore ranged 1.31 mg/kg and 4.59 mg/kg. Greater Cd concentrations were uncovered alongside the capital city area and Lebir River, which are both moderately to heavily inhabited regions. The risen Cd concentrations could be caused by nearby human-based activities, such as boating, smelting, mining, and recycling of ores or metals (Miko et al., 2002; Ayres et al., 2003). The average Cd concentrations in surficial sediment at the Kelantan river and at nearshore ranged 1.31 mg/kg and 4.59 mg/kg. Anthropogenic activities and industries like steel and plastic industries, metal plating and electroplating, nickel-cadmium batteries, welding, fertilizers are sources of Cd being released to environment. Apart from this, soils and rocks including mineral fertilizers and coal may also contain Cd resulted from human caused activities and industrial discharges (Yoshimoto et al., 2016). The low pH value of sediment causes the heavy metals to be desorbed from the water into water that leads to a higher concentration of Cd. Similarly, Cd concentrations decreases as pH value increases along the river due to the ability of metal binding has been reduced (Saraee et al., 2011). The Cr metal levels were significant along the Galas River and capital city area. Elevated Cr levels indicates high release of industrial and urban discharge, including the untreated effluent of petroleum, textile, and agricultural industries (Mohiuddin et al. 2012). Waste composition that released from textiles industry, leather tanning, metal finishing and chromate preparation, and pigments for paint, paper, and rubber which consume the usage of Cr are likely to contaminate the drinking water by releasing the wastewater to the environment (Abbas et al., 2016). The As concentrations in surficial sediment ranged from 2.3 mg/kg to 15.40 mg/kg with an average of 7.38 mg/kg at the Kelantan river and ranged from 2.76 mg/kg to 22.36 mg/kg with an average of 14.13 mg/kg at the nearshore. Additionally, greater As concentrations were discovered at urban regions, which were the capital city, Kuala Krai urban district and Lebir sub-district (Figure 4.7 and 4.8). This discovery was suggestive of anthropogenic input, whereby arsenic-consuming industries included smelting, wood and glass manufacturing that were observed along the Kelantan River (Brooks 2010). As are naturally released to the water column from geological formation processes that occur in sedimentary rocks, weathered volcanic rocks and geothermal water. Introduction of As by human activities a=through mining activities, manufacturing, wood preservation, metallurgy and by usage of pesticides (Freije, 2015). Lastly, the elevated concentrations of Zn were discovered at the capital city area, confluence, and along the Galas River (Figure 4.21 and 4.22). Nearby man-made and natural geography could have caused to the raised Zn concentrations, namely agriculture and urbanisation (Ramessur & Ramjeawon, 2002). The coastal of heavy metals was predominantly concentrated up to a certain distance offshore from the estuary, which could be aided by a common zone of enhanced particle concentration and particle settling or deposition, in which the situation is also known as the null zone or entrapment zone (Kimmerer, 1992). Zinc is discharged to environment through industrial and activities such as wood pulp production, brass plating, zinc and brass metal works, steel works with galvanizing lines and ground or newsprint paper production. A combination sources of agricultural activities, groundwater intrusion and sediment entrainment also discharging the Zn to the environment (Jan & Edward, 2016). The permissible level of Zn for human consumption by MFA is 100 mg/kg wet weight and the concentration is not exceeded the limit for this study. Overall, the Zn concentrations in surficial sediment ranged from 12.28 mg/kg to 44.61 mg/kg with an average of 32.95 mg/kg at the Kelantan river and ranged from 13.13 mg/kg to 67.01 mg/kg with an average of 41.04 mg/kg at the nearshore.

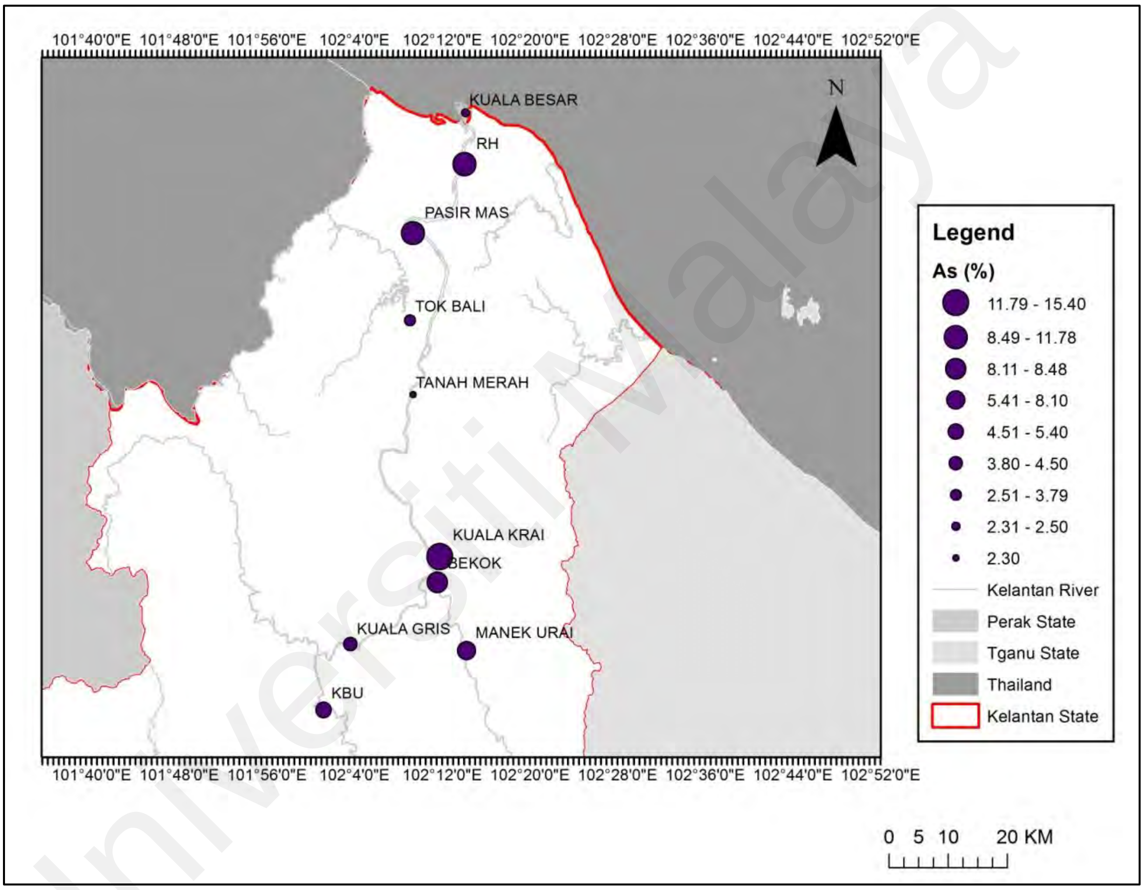

Figure 4.7: As distribution in surface sediments of riverine and estuarine stations.

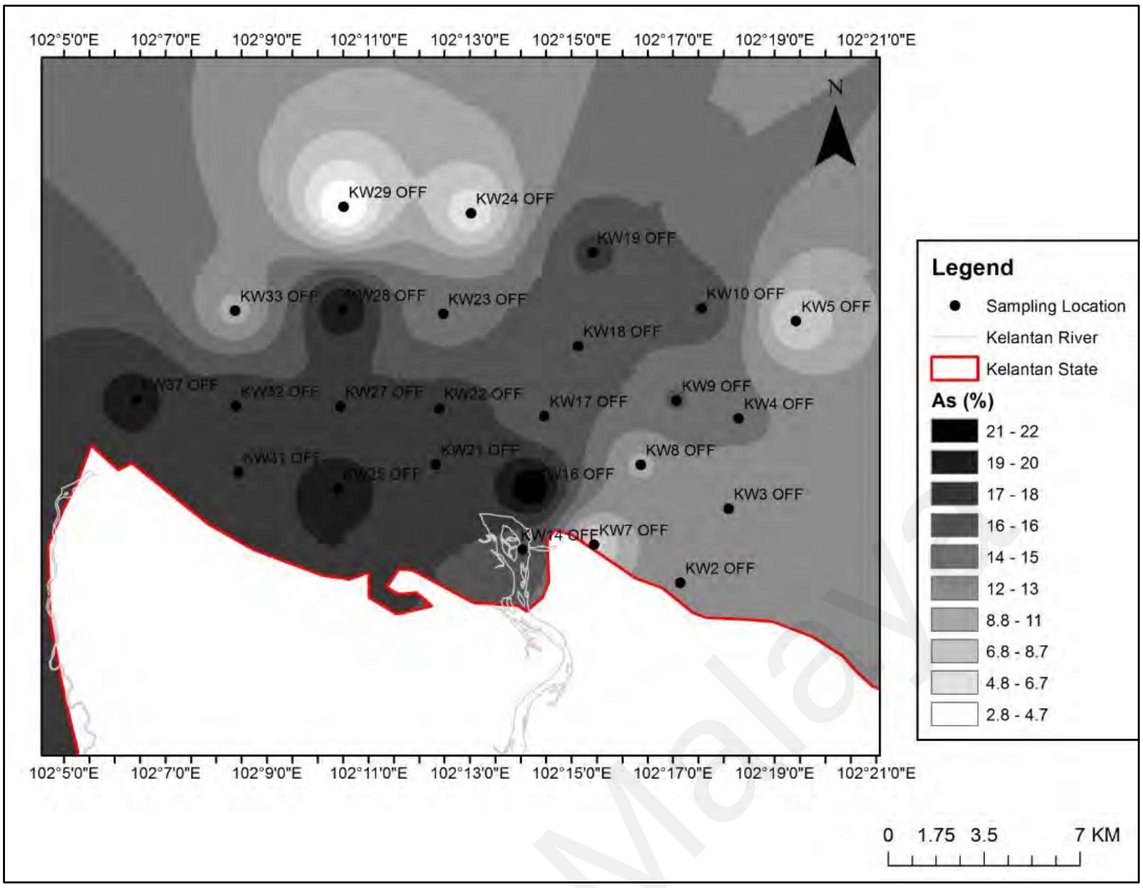

Figure 4.8: As distribution in surface sediments of nearshore stations.

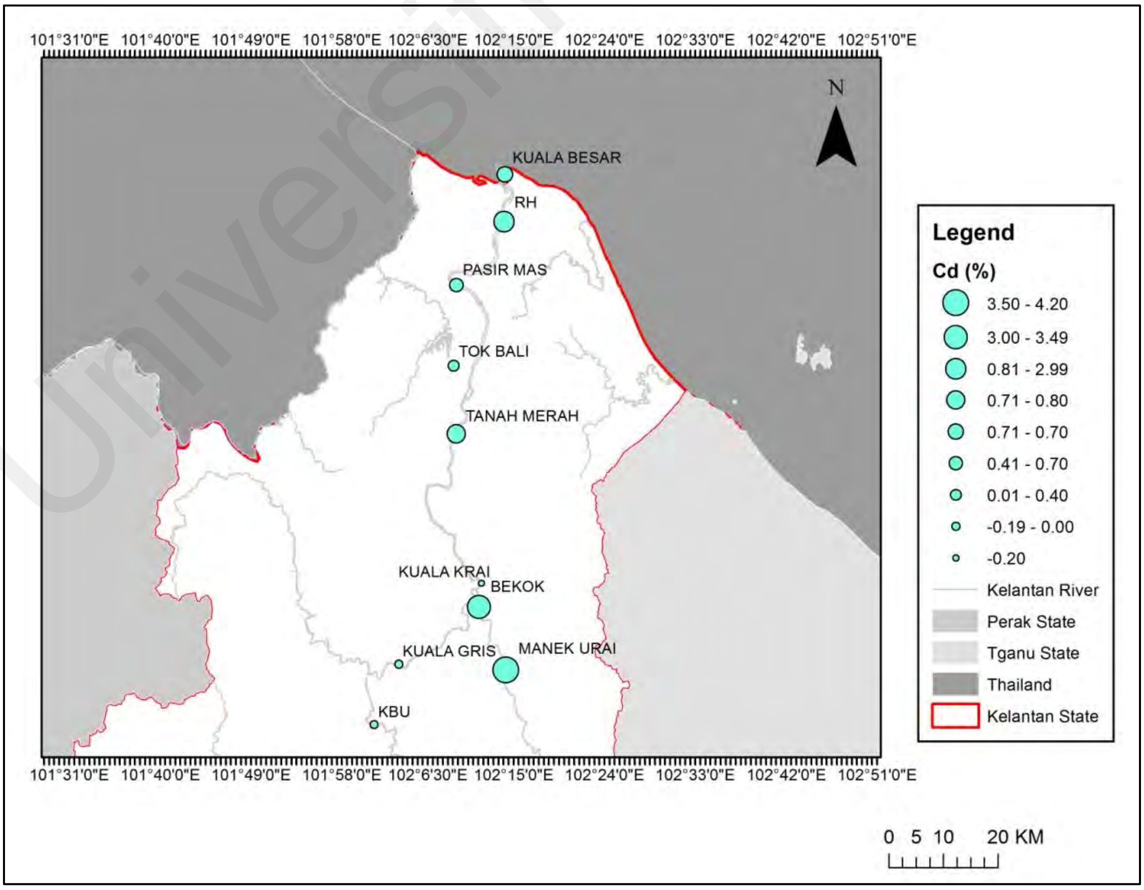

Figure 4.9: Cd distributions in surface sediments of riverine stations.

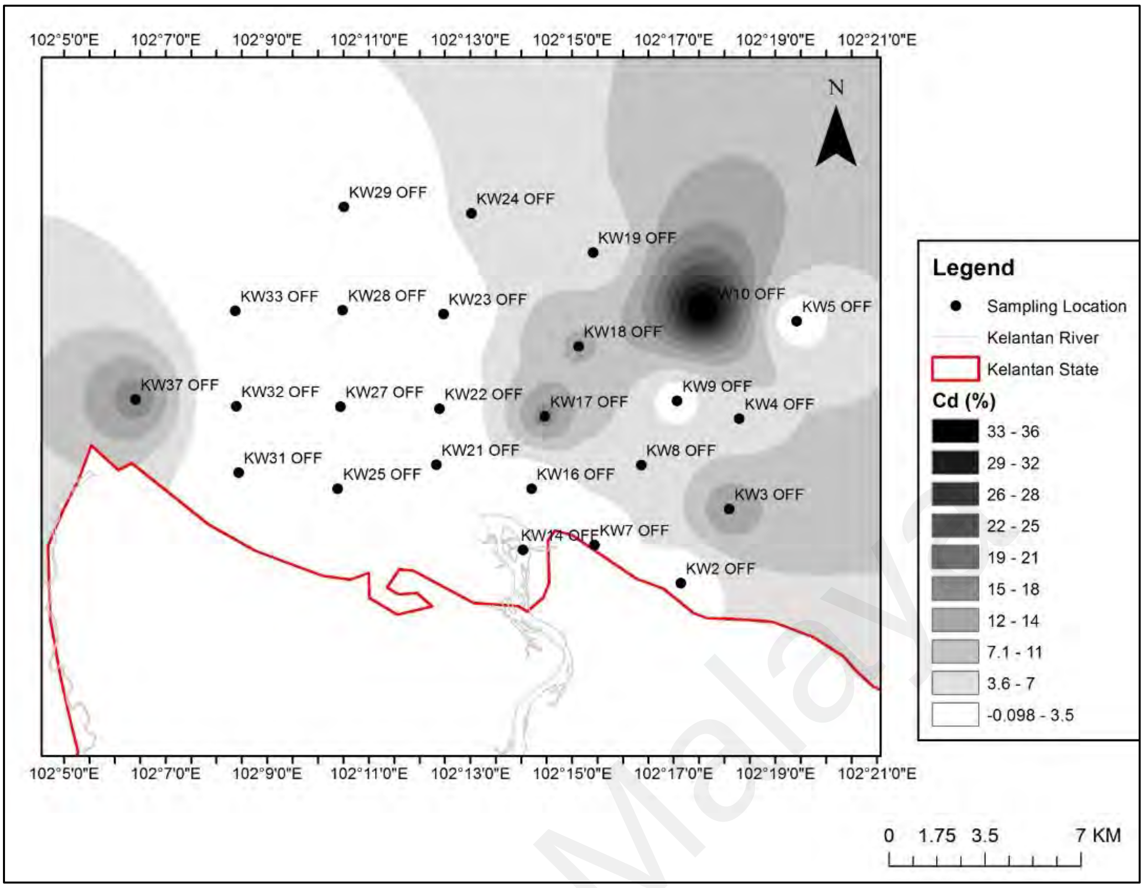

Figure 4.10: Cd distributions in surface sediments of nearshore stations.

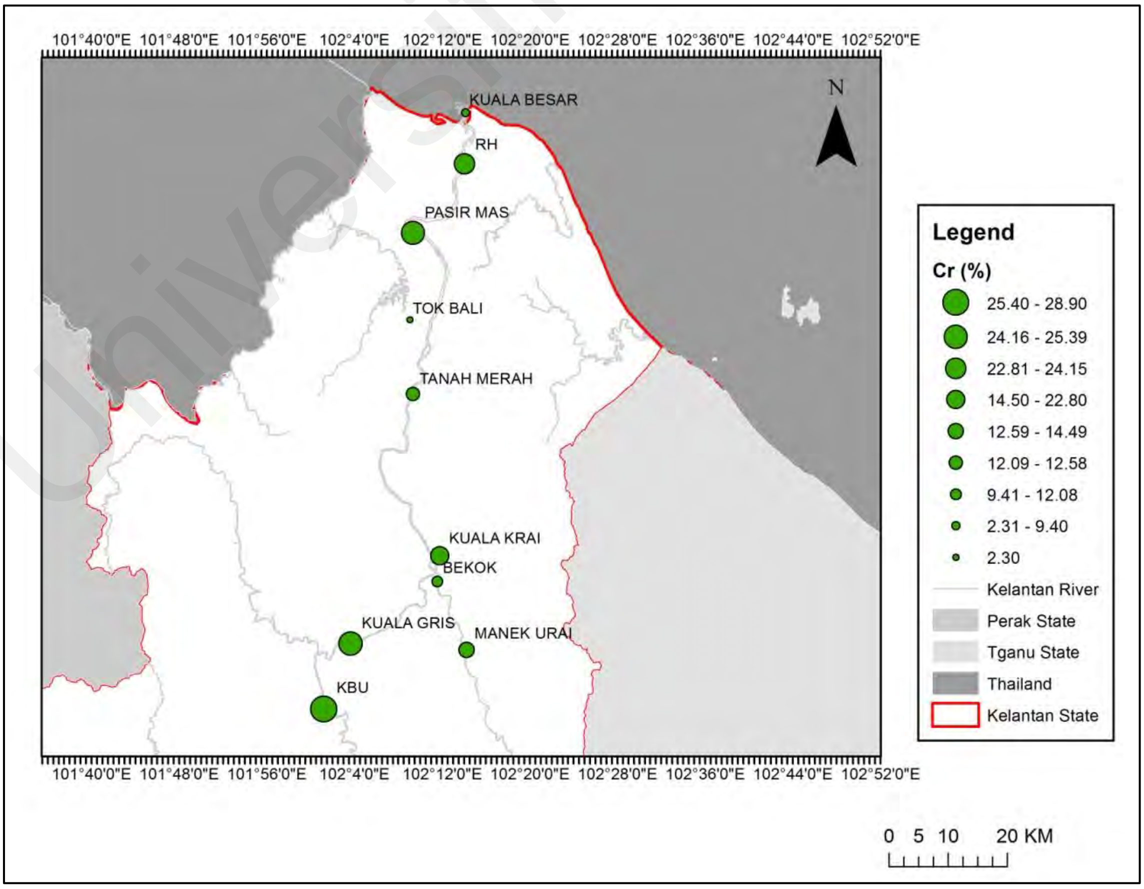

Figure 4.11: Cr distributions in surface sediments of riverine stations.

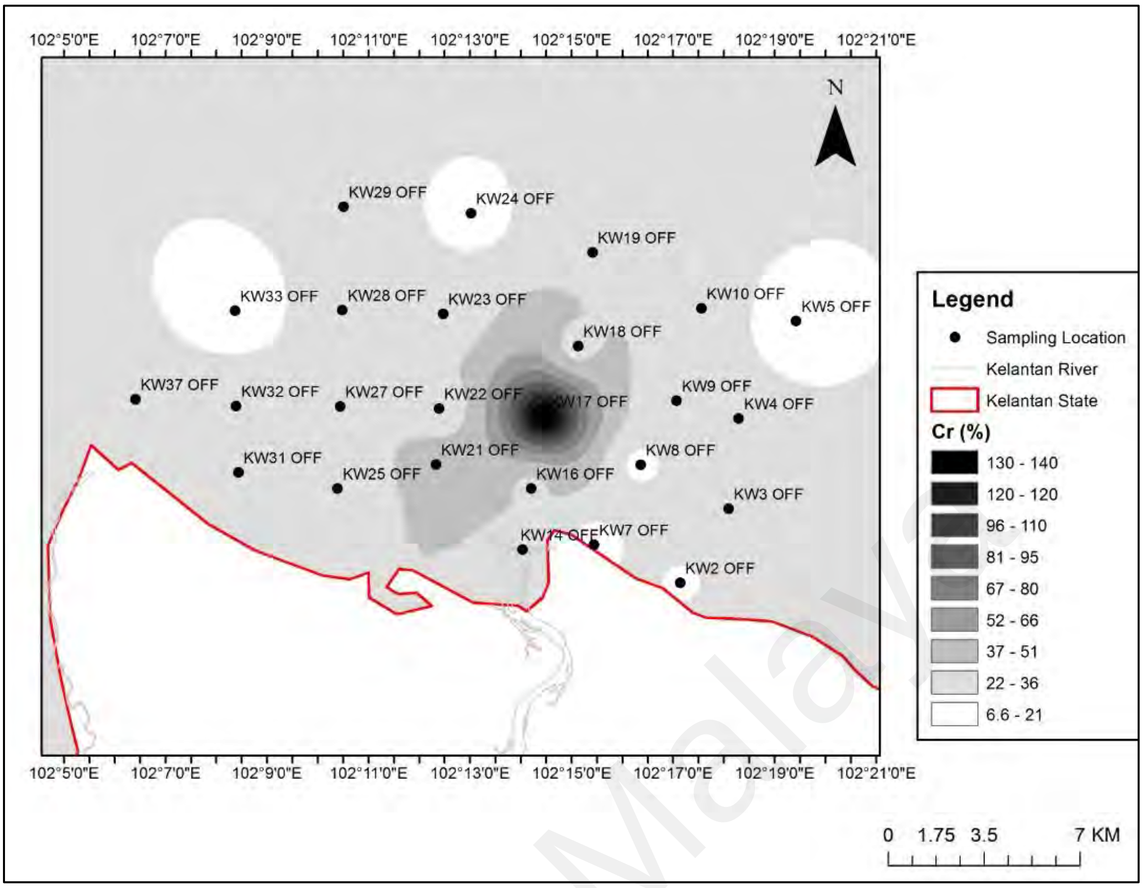

Figure 4.12: Cr distributions in surface sediments of nearshore stations.

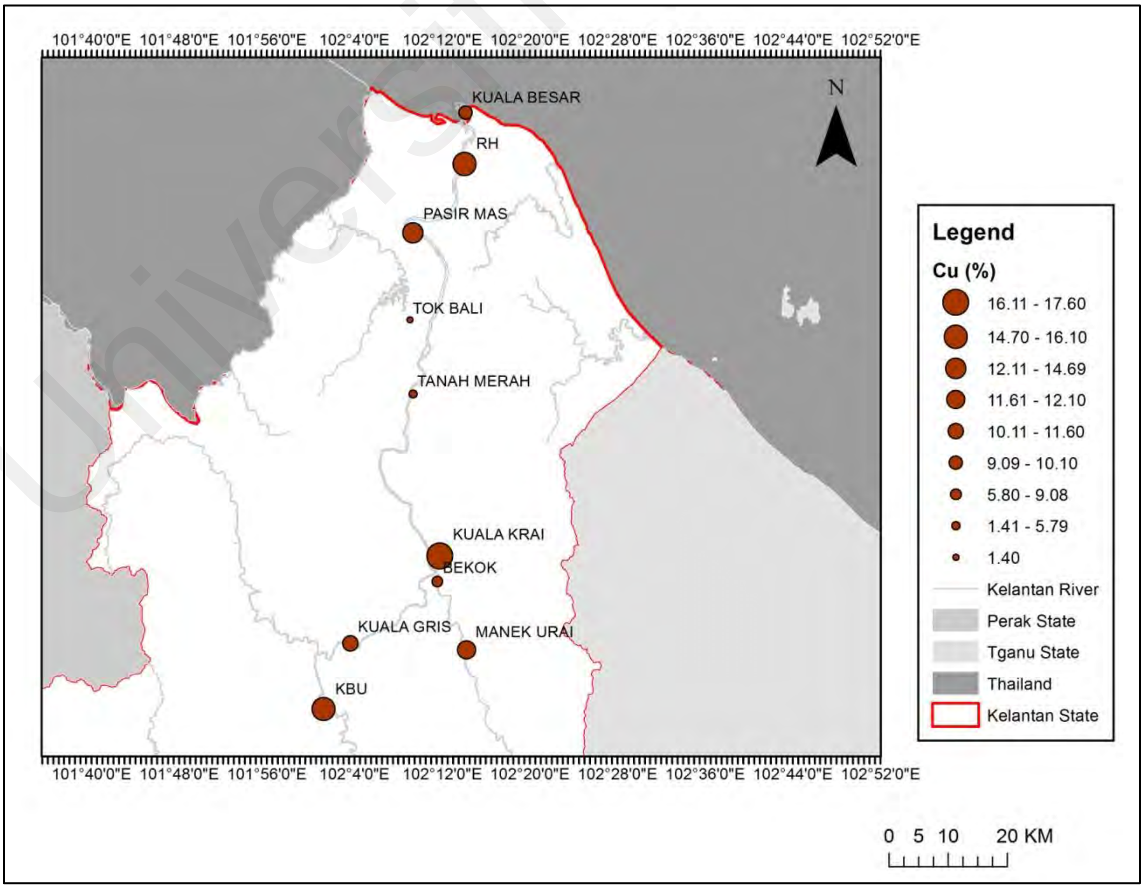

Figure 4.13: Cu distributions in surface sediments of riverine stations.

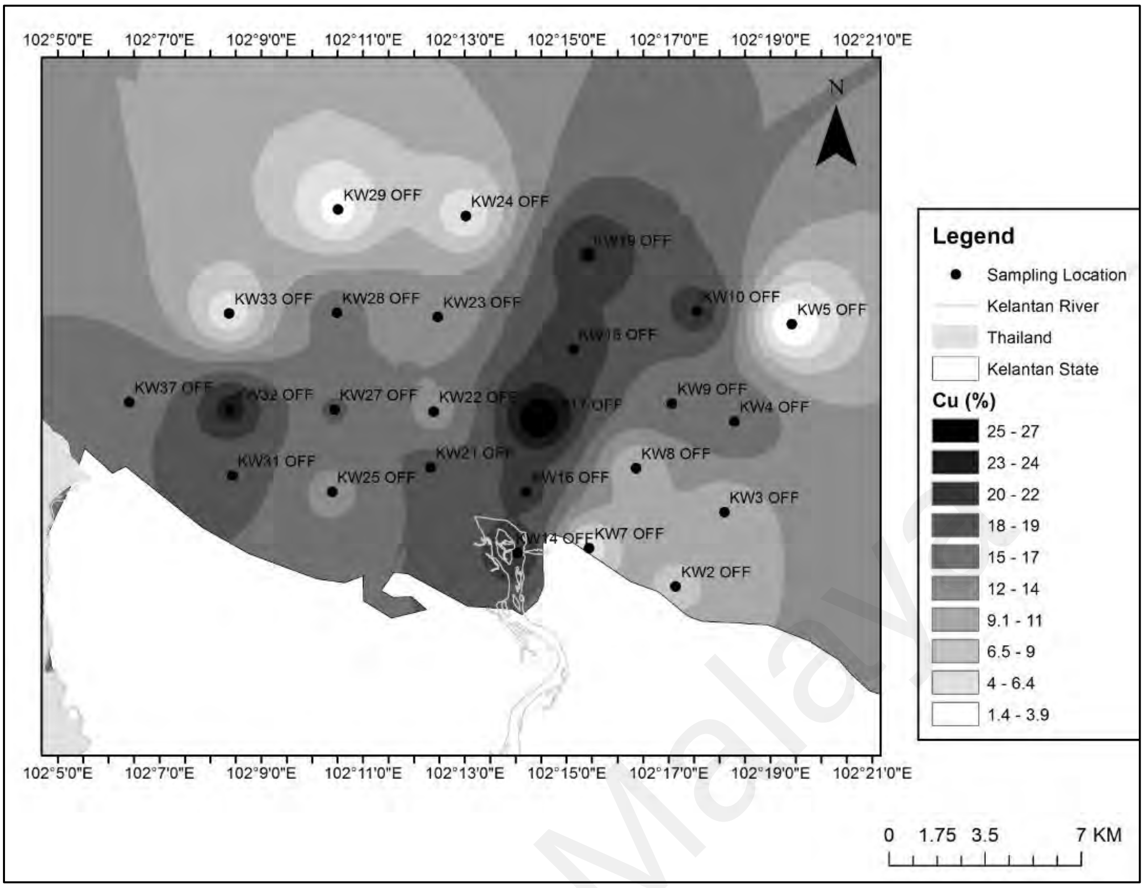

Figure 4.14: Cu distributions in surface sediments of nearshore stations.

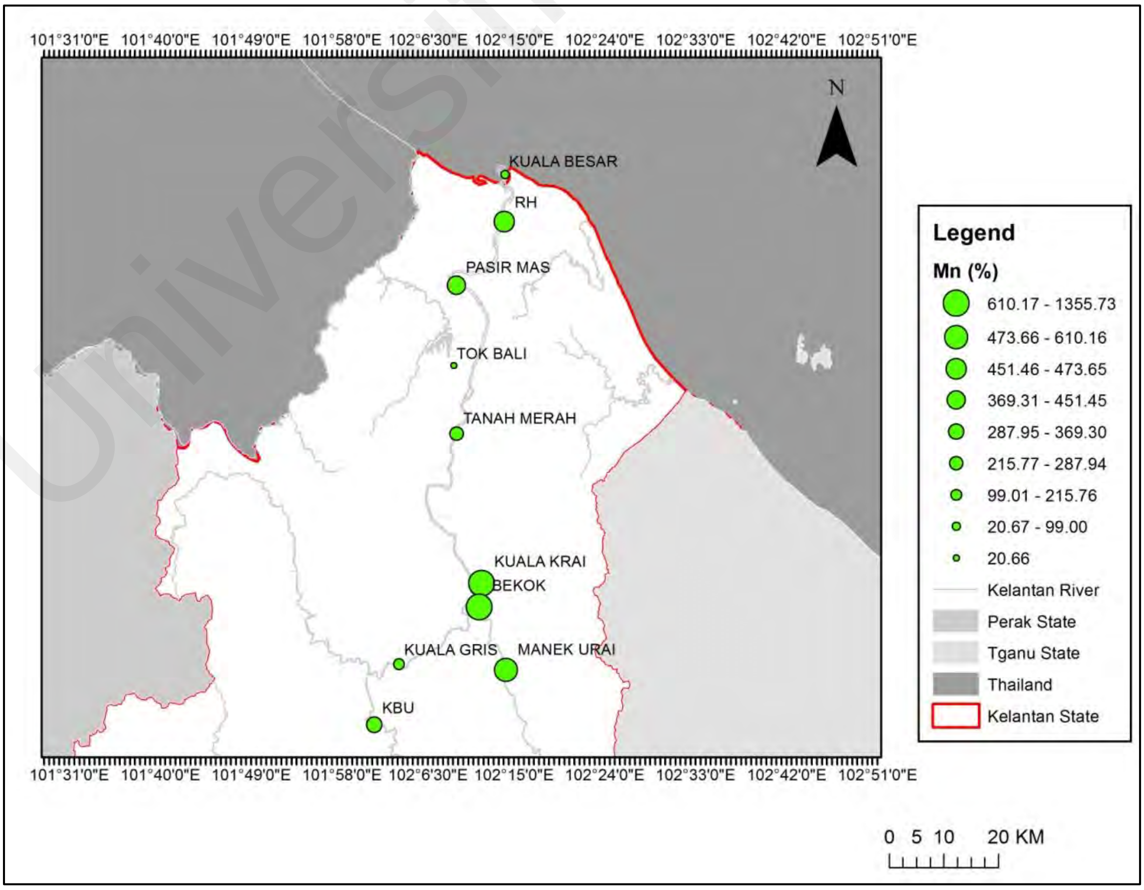

Figure 4.15: Mn distributions in surface sediments of riverine stations.

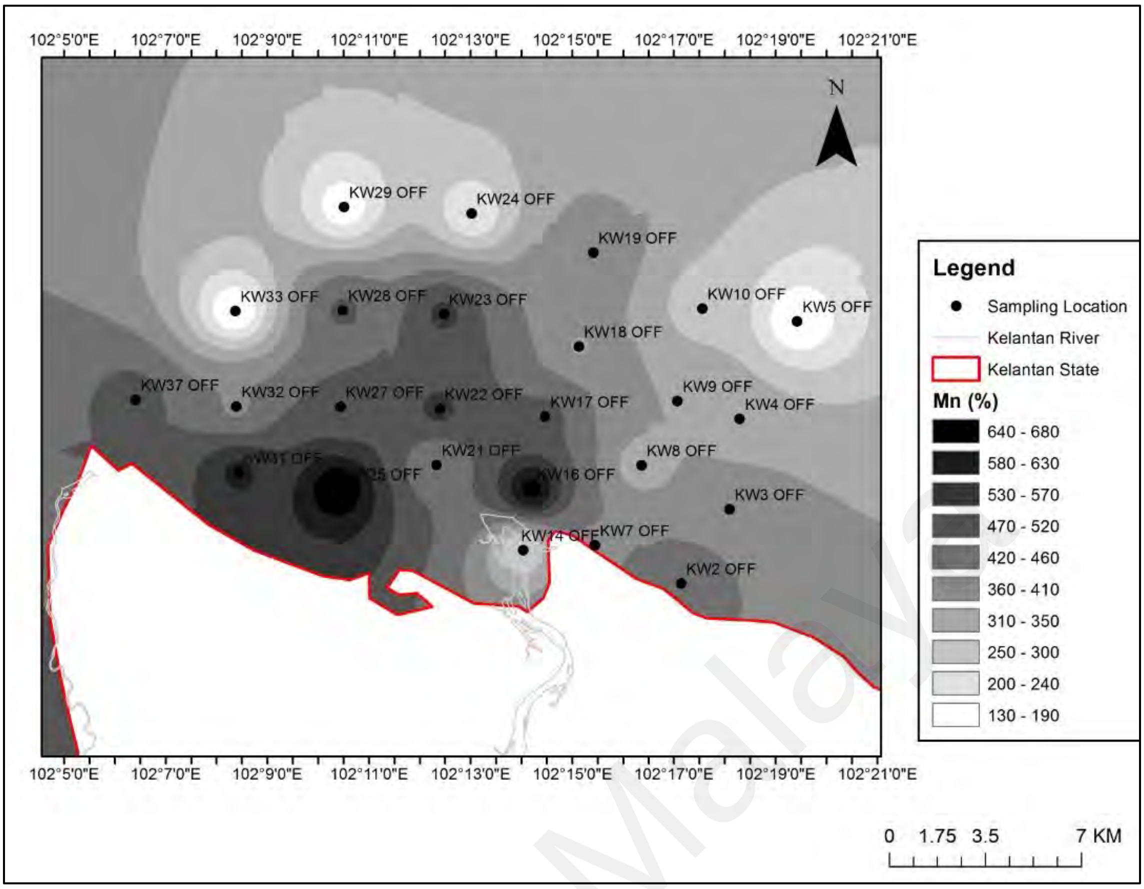

Figure 4.16: Mn distributions in surface sediments of nearshore stations.

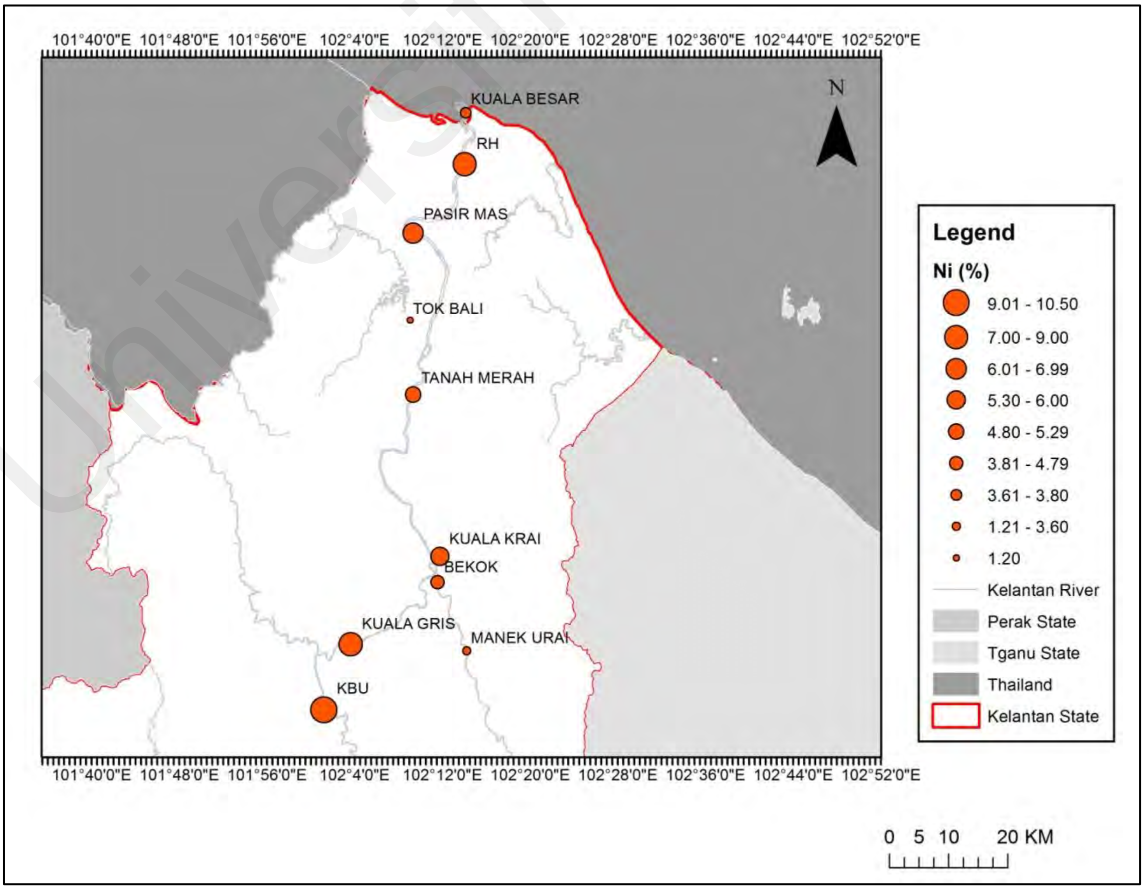

Figure 4.17: Ni distributions in surface sediments of riverine stations.

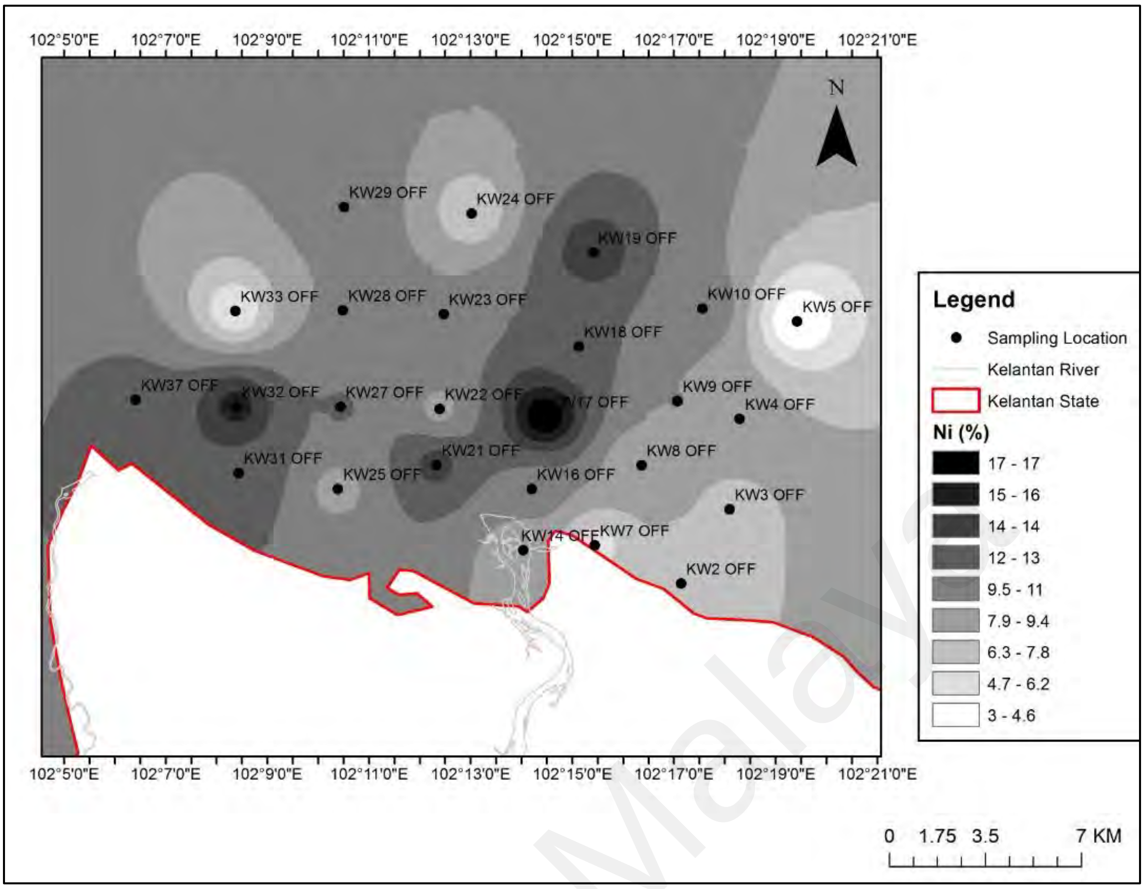

Figure 4.18: Ni distributions in surface sediments of nearshore stations.

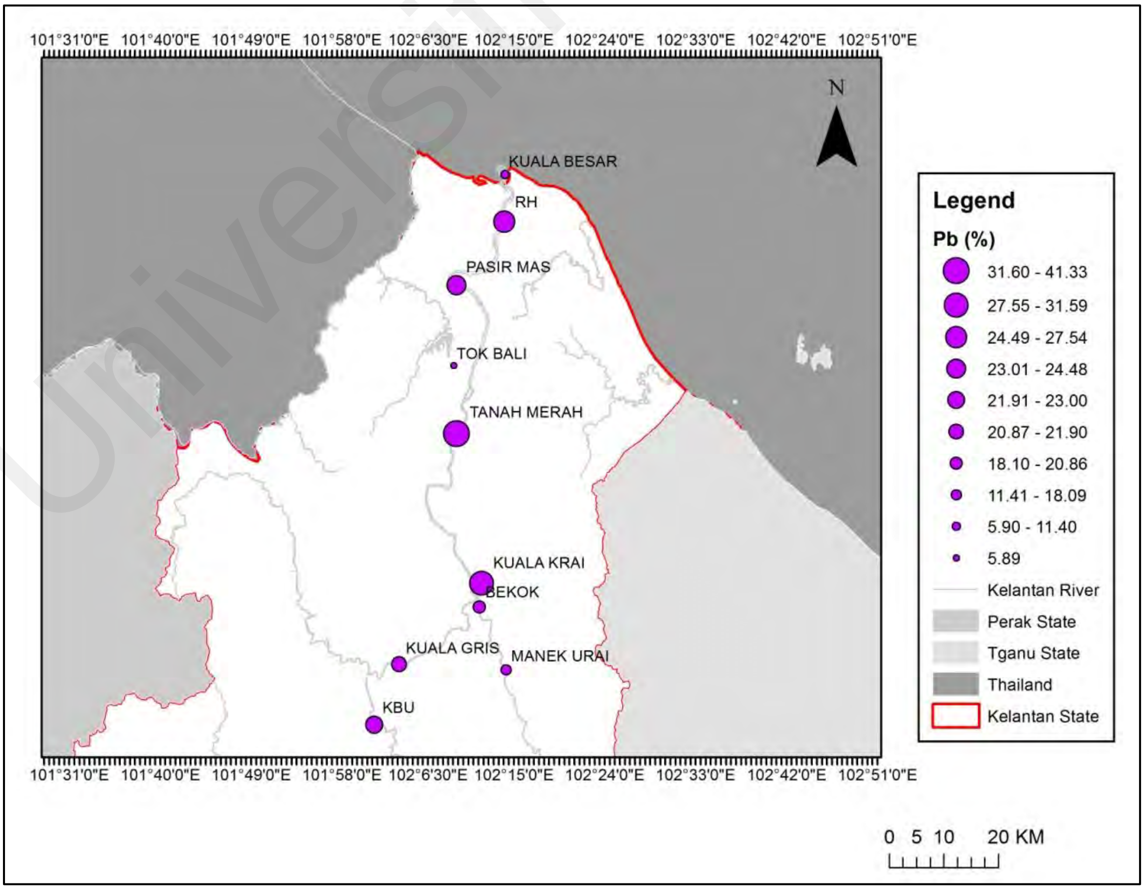

Figure 4.19: Pb distributions in surface sediments of riverine stations.

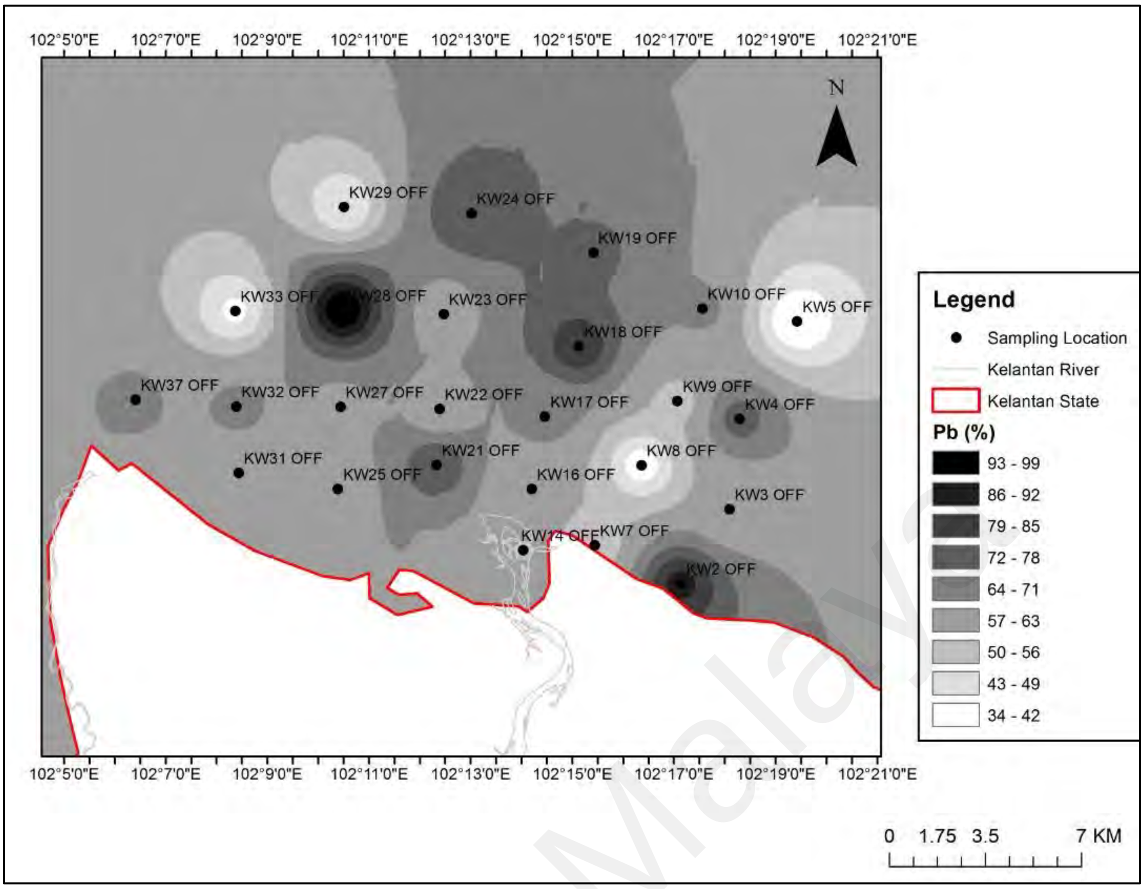

Figure 4.20: Pb distributions in surface sediments of nearshore stations.

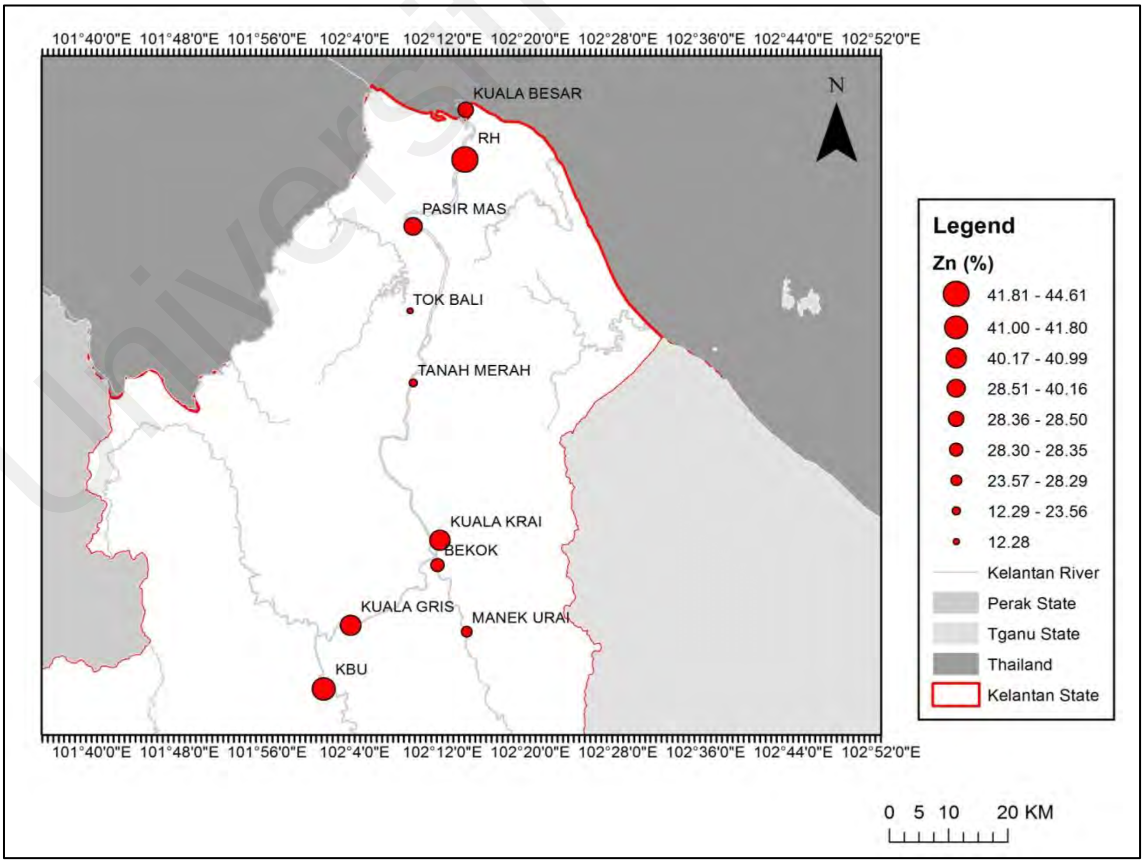

Figure 4.21: Zn distributions in surface sediments of riverine stations.

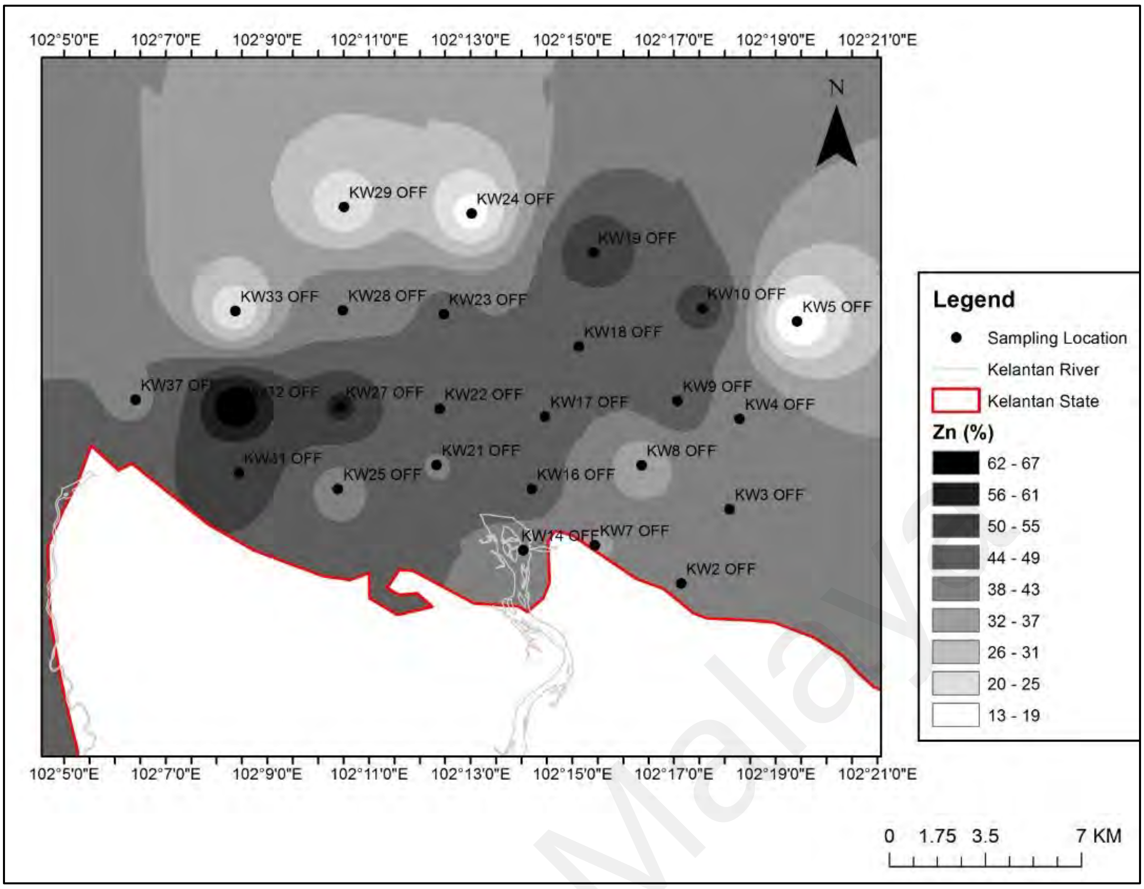

Figure 4.22: Zn distributions in surface sediments of nearshore stations.

4.4 Correlation between Physicochemical Characteristics and Heavy Metals

The pH data showed a moderate negative correlation with a value of -0.389 against clay composition and a positive weak correlated with sand composition with a value of 0.223 (Table 4.4). Furthermore, in this study, the correlation analysis revealed a significant positive correlation between the total organic matter content and heavy metals indicating that it might control the distribution of the analyzed metals in the sediments. Also, the significant correlation between total organic matter content and As, Zn, Cu implied a contribution of heavy metals from domestic or agricultural sources. The results of correlation analysis showed that Mn, Cu, Zn and As correlated significantly with clay and silt (Table 4.4). The tendency of fine-grained sediments to adsorb much more heavy metal due to high specific surface area characteristics results in a higher concentration of heavy metals in sediments. This could suggest a natural origin of these components from weathering and erosion; the metals may have been contributed by agriculture, logging and land development. However, other heavy metals did not show any significant relationship with the finer-sized particles, suggesting that there might be other possible factors that determined their distribution.

One of the controlling factors of spatial distribution in the sediments is the organic matter content. The geochemical behaviour of heavy metals can be regulated by organic matter content via adsorption, desorption and complexation. Among the heavy metals, Mn had showed a comparable negative correlation with other metals. However, other metals exhibited significant positive correlation with each other. Data revealed that Mn may not derived from the same sources as other heavy metals but from limited industries, or heavily filtered and utilised by the surrounding geographical terrains, such as vegetation.

4.5 Environmental Indices

4.5.1 Pollution load index (PLI)

According to the PLI data, only four coastal stations nearer to the estuary or shoreline were classified as polluted (Table 4.5). The calculated PLI data classified those four nearshore stations namely KW10, KW17, KW18, and KW37 as polluted which have PLI value more than 1, (PLI > 1) were ranged from 1.01 to 1.37. The PLI gives some useful information on quality of the environment to the inhabitants. It also gives beneficial information on status of pollution of the area to the decision makers (Islam et al., 2015). This higher PLI values indicates heavy metal input might be due to urban activities and anthropogenic sources (Yen & Rohasliney, 2013). The overall PLI data showed that the surficial sediments unpolluted in the Kelantan river and the nearshore area. The PLI data for heavy metals varies a little in surficial sediment along the Kelantan river and recorded higher at nearshore. A relatively higher PLI values were recorded for heavy metals at the tributary upstream of both Galas and Lebir river (station Manek Urai and Bekok) and shows an increasing trend in PLI for heavy metals at the main river channel flowed from mountainous area. However, there is a gradual decrease pattern observed in PLI for heavy metals in the shelf environment starting from the estuarine area towards the offshore direction.

Table 4.4: Correlation coefficient between sand, silt, clay, total organic carbon, pH and metals.

	pH	LOI	Clay	Silt	Sand	Pb	Cd	Mn	Cu	Zn	As	Ni	Cr
pH	1												
LOI	0.15	1											
Clay	**-0.389***	**0.371***	1										
Silt	-0.115	**0.551****	**0.674****	1									
Sand	0.223	**-0.531****	**-0.847****	**-0.964****	1								
Pb	**0.470****	**0.500****	-0.110	0.111	-0.040	1							
Cd	-0.041	0.298	0.091	0.110	-0.112	0.239	1						
Mn	0.005	0.275	**0.607****	**0.477****	**0.563****	-0.011	-0.073	1					
Cu	-0.226	**0.680****	**0.607****	**0.611****	**-0.659****	**0.396***	0.308	0.300	1				
Zn	-0.060	**0.693****	**0.470****	**0.695****	**-0.670****	**0.444****	0.198	0.294	**0.839****	1			
As	0.104	**0.738****	**0.397***	**-0.449****	**-0.467****	**0.623****	0.185	**0.394***	**0.739****	**0.731****	1		
Ni	0.132	**0.614****	0.154	**0.439****	**-0.372***	**0.572****	0.260	0.074	**0.772****	**0.761****	**0.623****	1	
Cr	0.016	**0.398***	0.155	0.298	-0.270	**0.338***	0.317	0.109	**0.650****	**0.427***	**0.386***	**0.739****	1

**Correlation is significant at the 0.01 level (2-tailed).
*Correlation is significant at the 0.05 level (2-tailed).

Table 4.5: Potential ecological risk index (PERI) and pollution load index (PLI) of heavy metals in the Kelantan River.

Station	Potential ecological risk index (PERI)		Pollution load index (PLI)	
	Index	Risk classification	Index	Pollution classification
BEKOK	376.25	Considerable	0.53	No pollution
KUALA GRIS	36.97	Low	0.00	No pollution
TANAH MERAH	106.17	Low	0.32	No pollution
KUALA BESAR	85.99	Low	0.24	No pollution
TOK BALI	47.00	Low	0.09	No pollution
MANEK URAI	447.70	Considerable	0.50	No pollution
PASIR MAS	113.04	Moderate	0.52	No pollution
RH	342.88	Considerable	0.66	No pollution
KBU	42.24	Low	0.00	No pollution
KUALA KRAI	26.96	Low	0.00	No pollution
KW2	53.14	Low	0.00	No pollution
KW3	1386.73	Very high	0.76	No pollution
KW4	586.72	Considerable	0.77	No pollution
KW5	130.52	Low	0.23	No pollution
KW7	263.50	Moderate	0.47	No pollution
KW8	386.77	Considerable	0.56	No pollution
KW9	143.90	Low	0.64	No pollution
KW10	3619.78	Very high	1.09	Polluted
KW14	51.25	Low	0.00	No pollution
KW16	58.91	Low	0.00	No pollution
KW17	1704.35	Very high	1.37	Polluted
KW18	1177.73	Very high	1.01	Polluted
KW19	408.84	Considerable	0.92	No pollution
KW21	89.28	Low	0.58	No pollution
KW22	55.49	Low	0.00	No pollution

Table 4.5, continued.

KW23	103.03	Low	0.59	No pollution
KW24	384.47	Considerable	0.41	No pollution
KW25	67.99	Low	0.52	No pollution
KW27	79.67	Low	0.60	No pollution
KW28	243.31	Moderate	0.80	No pollution
KW29	51.02	Low	0.25	No pollution
KW31	123.34	Low	0.72	No pollution
KW32	68.64	Low	0.00	No pollution
KW33	85.51	Low	0.29	No pollution
KW37	1648.21	Very high	1.02	Polluted

Table 4.5a: Average Potential ecological risk ($E^r{}_i$) of single heavy metal in the Kelantan River and nearshore sediment.

Metal	The value of $E^r{}_i$ at Kelantan river	The value of $E^r{}_i$ at nearshore
Pb	5.65[a]	15.55 [a]
Cd	130.83[b]	459.50[c]
Mn	0.60[a]	0.46 [a]
Cu	1.27[a]	1.56 [a]
Zn	0.35 [a]	0.43 [a]
As	5.68[a]	10.88 [a]
Ni	0.44[a]	0.71 [a]
Cr	17.71[a]	29.79 [a]

[a] classified as low potential ecological risk

[b] classified as considerable ecological risk

[c] classified as very high ecological risk

4.5.2 Potential ecological risk index (PERI)

Potential ecological risk index provides an estimation on contamination degree of the sediment in the presence of metals other than major elements such as Fe, Al, and Mn. This index is estimated using the toxicity effect of the metals along with measured concentration of the sediment in comparison with heavy metals reference value in the earth crust (Diami et al., 2016). The range of potential ecological risk index (RI) in the study area were found to be between 26.96 and 3619.78 indicating that low to very high ecological risk in the metal contaminated surface sediment according to risk classification. The highest RI found at the five nearshore sampling sites (KW3, KW10, KW17, KW18, KW37) which is classified as having very high ecological risk was apparently due to Cd contribution (Table 4.5). A study showed that Cd contamination had caused a major freshwater lake in China to be in a moderate potential risk at national scale due to anthropogenic activities discharges (Cheng et al., 2015). Other sampling sites were found to be in the range of low to moderate ecological risk contamination. Individual of each single element (E^i_r) for other heavy metals (Cr, Pb,As, Cu,Ni, Mn) were lower than 40, suggesting sediments showed a low potential risk. However, Cd, showed the highest E^i_r with range value of 130.83, contributing 80.5 % at river and 459. 49, about 88.56 % at nearshore respectively (Table 4.5a). The sewage that discharged from cities along the east coast of Peninsular Malaysia had attributed higher concentration of Cd (Rezaee et at, 2011). The risk posed by individual heavy metal (E^i_r) at different sampling sites descended in the order of Cd > Cr > As > Pb > Cu > Mn > Ni > Zn for river and Cd > Cr > Pb > As > Cu > Ni > Mn > Zn for nearshore. Other metals such as Cr, As and Pb showed a mean E^i_r value of 17.71, 5.67, 5.65 at river and also 29.79, 10.87, 15.55 at nearshore respectively. The higher levels of Cr in estuary and coastal waters are due to industrial wastewater discharge into Kelantan river estuary delta in addition to its natural sources. The increment of Pb levels in surficial sediment from river to nearshore indicating the sources of it, which is originated from natural weathering and also from erosion of upstream materials. Furthermore, the release of the agricultural production activities into the river, urbanization at the plain area and industrialization activities at the estuary delta area also contributed to the elevated Pb levels in study area (Wang et al., 2017). The agriculture industry and the fertilizer used may also cause the As contamination in surficial sediments (Rezaee et at, 2011).

4.5.3 Geo-accumulation index (Igeo)

Among the studied metals, the Igeo values shows a decreasing order Cd > Pb > As > Mn > Zn > Cu > Cr > Ni for river that ranged from - 3.72 to 1.40 and Cd > Pb > As > Mn > Zn > Cr > Cu > Ni for nearshore that ranged from - 3.49 to 1.53. The Igeo value for all the heavy metals across the stations were lower than 1, indicating that those stations uncontaminated except for Cd, which showed moderate contamination with Igeo value of 1.40 at the riverine area. Similarly at the nearshore areas, the Igeo values revealed unpolluted status or minor level pollution for all the heavy metals except for Pb and Cd with the Igeo value of 1.01 and 1.53 respectively, indicating moderate contamination at the nearshore areas (Table 4.6). However, the Igeo for As, Mn, Zn, Cu, Cr, and Ni shows no significant pollution and were categorized as an uncontaminated status of sediment quality for both river and nearshore environment with minimal value (Igeo < 0).

As compared to other heavy metals, Cd were moderately highest at both riverine area and nearshore area. Station Manek Urai and KW10 were found to be moderately polluted with Cd along the riverine and nearshore area that marked Igeo value 3.22 and 6.30. Also, KW28 showed a moderate contamination of Pb at the nearshore environment with the value of 1.73 indicating a decreasing trend in Igeo values from landward to the seaward.

Table 4.6: Geo-accumulation index (Igeo) and enrichment factor (EF) of heavy metals in the Kelantan River.

Metal	Geo-accumulation index (Igeo)		Enrichment factor (EF)	
	River	Nearshore	River	Nearshore
As	-0.90 [g]	-0.60 [g]	7.55 [c]	7.11 [c]
Cd	1.40 [e]	1.53 [e]	69.86 [a]	93.54 [a]
Cr	-2.66 [g]	-2.44 [g]	2.17 [d]	1.95 [e]
Cu	-2.61 [g]	-2.53 [g]	3.02 [d]	1.70 [e]
Mn	-1.89 [g]	-1.84 [g]	7.28 [c]	3.01 [d]
Ni	-3.72 [g]	-3.49 [g]	1.02 [e]	0.96 [e]
Pb	-0.57 [g]	1.01 [e]	14.20 [c]	24.94 [b]
Zn	-1.98 [g]	-1.89 [g]	4.56 [d]	2.78 [d]

[a] classified as very high contamination (Igeo); extremely high enrichment (EF).
[b] classified as high to very high contamination (Igeo); very high enrichment (EF).
[c] classified as high contamination (Igeo); significant enrichment (EF).
[d] classified as moderate to high contamination (Igeo); moderate enrichment (EF).
[e] classified as moderate contamination (Igeo); deficiency to minimal enrichment (EF).
[f] classified as no to moderate contamination (Igeo).
[g] classified as no contamination (Igeo).

4.5.4 Enrichment factor (EF)

The average EF values of Cr, Zn, Cu, and Ni in the present study were found to be moderate to minimal enrichment compared to Pb, Cd, As and Mn where it was extreme to significant enrichment in surface sediment of nearshore and riverine regions. Cd had the highest values among of all studied metals and it has an extremely high enrichment with a mean value of 69.86 and 93.54 at the river and nearshore area respectively. Pb and As had significant enrichment with an average value of 14.20 and 7.55 at the riverine and greater average value of 24.94 and 7.11 at the nearshore area. The average EF values of 7.28 nd 3.01 at the river and nearshore for Mn indicated a significant enrichment at the study area. Zn, Cu and Cr exhibited moderate enrichment at the riverine (average value 3.02, 4.56 and 2.17) and Ni had a deficiency enrichment at the river with an average value of less than 2. However, at the nearshore area surface sediment were deficiently enriched with Cu, Ni, and Cr (average value 1.70, 0.96, and 1.95) but has been moderately enriched with Zn (average value 2.78). In general, the average EF values pattern for river were Cd >

Pb > As > Mn > Zn > Cu > Cr > Ni and nearshore were Cd > Pb > As > Mn > Zn > Cr > Cu > Ni.

Distinguishment between sources of the element whether it is potentially due to anthropogenic or natural input can be derived from EF calculation. EF value of 0 to 1.5 indicates that it may be originated from crust and origin is natural. Contrarily, EF > 1.5 suggested that it originated through anthropogenic input and EF values greater than 10 indicated that the metals were non-crusted resources (Ahamad et al, 2020). Study areas which is situated nearer to different industries have a higher EF values The rate of removal of metals from the sediment and magnitude of input for each metals in sediment potentially varies the EF values for different metals (Nowrouzi et al., 2014). Fishing and its associated activities including boats servicing and repairing also have an impact on high enrichment of Pb on a specific study area. Also, domestic and industrial effluent also contributed to enrichment of Ni, Pb, and Zn along the stream. The extreme enrichment of Cd at the riverine and nearshore may also resulted from sand mining, domestic origin, industrial and agricultural activities along the river. The usage of fertilizers especially for crops such as oil palm may contain monosodium and disodium methylarsonate can alter by decreasing the pH level in the sediment can eventually increases the levels or enrichment of metals in surface sediment at the study area (Kadhum et al., 2015). Thus, it can be deduced that Cd can be mainly originated from anthropogenic activities.

4.6 Summary

This chapter summarises the research findings of the study and explains the outcome of the results. Based on the findings of this study, Kelantan river had showed the deteriorating environmental quality in surface sediments at the river and its nearshore area in terms of heavy metal pollution and ecological risk assessment status. The surficial sediments experienced Cd and Pb pollutions, at KW10 and KW24 which marked highest concentrations compared to other sampling sites. The heavy metals are mainly originated from anthropogenic activities and total organic matter content can elevate the enrichment levels of heavy metals in surface sediments at the study area. Based on the spatial distribution of heavy metal contents and environmental risk assessment results, the enrichment of Kelantan river and its nearshore area is closely related to the total organic matter content and grains size distribution at of the surficial sediments.

CHAPTER 5: CONCLUSION

This study showed the deteriorating environmental quality in surface sediments of the Kelantan River and its nearshore area by analyzing heavy metal pollution level and ecological risk assessment status. Higher concentration of heavy metals in sediments were found at the nearshore area compared to riverine area. Overall, surface sediment was mainly dominated by silt type sediment, followed by sand and clay distribution at the study area. The surface sediment at the river channel are mainly comprises of silt clay. The texture distribution patterns showed that most of surficial sediments at the nearshore were dominated by clayey silt and the sand content increased as the distance increased from the shore.

High LOI and organic depositions were detected in surface sediments at the capital city, confluence, upstream areas of both tributaries, and adjacent shelf areas. Wang et al. (2017) also detected higher OM along the adjacent shelf areas. Mild acidic to neutral pH levels detected at the tributaries, confluence, midstream, capital city and estuary, whereas alkaline pH levels were detected at coastal marine areas and Tanah Merah, the midstream urban district. The observed trend was expected due to pH in saltwater habitats is typically mildly alkaline but acidic in anthropogenically populated riverine areas (Kulthanan et al., 2013).

Average values of EF and Igeo the contamination degree in river and nearshore was defined as extremely enriched for Cd, significant to very high enrichment for Pb, moderate to significant for Mn, Cu, Zn, As and moderate to minimal enrichment for Ni and Cr. PLI values showed that the level of overall sediment pollution as highest at four stations namely KW10, KW17, KW18, KW37. PERI values revealed that each single element has low potential ecological risk and Cd shows higher ecological risk. Organic matter content had positive correlation with clay and silt content and negative correlation with sand composition. Besides that, significant and positive correlation were observed between organic matter content and Pb, Cu, Zn, As, and Ni. In addition, strong relationships between Cu and Zn (r=0.83), Zn and Ni (r=0.76), Ni and Cr (r=0.73) suggested potential enrichment mechanisms and similar sources for these heavy metals. The heavy metal content in surficial sediments showed a decreasing pattern from the river to the nearshore. The heavy metals concentration observed to be peaked at the convergence of the river stream (K.Krai and Bekok) and also at the low-land plain areas (Pasir Mas and RH). The heavy metals in Kelantan river and nearshore surface sediment in a descending order of average concentrations were as follows: Mn > Zn > Pb > Cr > Cu > As > Ni > Cd, and Mn > Pb > Zn > Cr > As > Cu > Ni > Cd. The data of this study revealed that anthropogenic development has discharged metal contaminants into the surrounding river and sea. The most enriched heavy metal contaminants that entail urgent monitoring enforcement are As, Pb and Cd. The potential for heavy metal contamination to worsen in the future, by anthropogenic activities, domestic and industrial wastewater influx, calls for attention from the public and scientific community towards continuous quality analysis and remediation planning.

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LIST OF PUBLICATIONS AND PRESENTATION

List of Publication

1. **Pooveneswary, N.,** Bakar, A. F. A., Wei, B. C., Hassan, Lee, C.W., Jun, A.J., Hassan, M.H., Yunus, R.M. & Zakaria, M.P. (2019). Contamination status and ecological risk of heavy metals in surface sediment of Kelantan River and its nearshore area, Malaysia.*Water Science & Technology Water Supply*, 20 (1): 103–117. https://doi.org/10.2166/ws.2019.138

Conferences / Seminar

1. Poster presentation in "IOES HICoE Seminar 2017: Air-Ocean-Land Interactions ", 12 September 2017, Institute of Graduate Studies, University of Malaya, Malaysia.