Assessment of Heavy Metal Contamination in Sediments in Sungai Pinang River Basin

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Abstract: A study was conducted to analyse the type and concentration of heavy metals found present in sediments samples in the Sungai Pinang river basin, Penang. River sediment samples were collected at both upstream and downstream along the main river channel of Sungai Pinang and its five tributaries including Sungai Air Terjun, Sungai Air Hitam, Sungai Air Putih, Sungai Dondang and Sungai Jelutong. A total of 19 sampling points were selected throughout the entire catchment area where nine points located at the main river of Sungai Pinang and the other 10 points located at the downstream and upstream of each tributaries. A total of 97 samples were obtained four times from January to February. Two types of analysis were applied to measure the heavy metal concentrations namely X-Ray Fluorescence (XRF) and Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES). For the XRF test, the selected compounds analysis containing heavy metals that was carried out were K₂O, Na₂O, CaO, MgO, MnO, Fe₂O₃, Al₂O₃, TiO₂ and SiO₂ while for the ICP-OES, specific heavy metals analysis were determined to test the presence of As, Ca, Cd, Co, Cr, Mg, Fe, Ni, Pb and Zn. The highest concentration of K₂O, Fe₂O₃, Al₂O₃ and SiO₂ at the main river were 2.80%, 3.39%, 18.52% and 74.18% respectively. The highest concentration of Ca, Cr, Zn, Mg and Fe recorded at main river are 35.9576, 17.0467, 3.3633mg/kg at SD (US) and 7.1662mg/kg at SAT (US). It can be concluded, the pollutants in the sediments of Sungai Pinang are contributed by the urban housing area, commercial and agricultural area.

1. Introduction
Vast advancement of human activities continues driving urban population growth and urban expansion due to economic globalization [1]. Rapid urbanization and industrialization are the two fundamental factors among many anthropogenic causes induced the environmental degradation in term of heavy metals contamination in soils [2]. Effortlessly, heavy metals able to be transported to the soil through wet and dry deposition by the atmosphere within a distance and dispersed up to several kilometres, away from its sources [3]. Existence of heavy metals can be toxic and exposure to high levels of heavy metals will cause accumulation of the elements in the sediments and deteriorate river nearby [4]. Besides, the polluted water was consumed by the plants, then released as gases into the atmosphere which will affect public human health [5]. It can inhibit the biodegradation of organic contaminants and disturb the food-chain [6].
Sungai Pinang is classified as one of the seven most contaminated river in Malaysia (Department of Environment, Malaysia). It once classified as Class VI, based on the Interim National Water Quality Standards (INQWS). The accumulation of heavy metals in soils resulting from the intensified urbanization and anthropogenic activities severely exhibit the toxicity of the soil to aquatic organisms leading to the deterioration of Sungai Pinang. The presence of chemical organic and inorganic pollutants such as polycyclic aromatic hydrocarbons (PAHs) and heavy metals are said to be the secondary source of pollution [7], where a significant high concentrations of heavy metals namely Cd, Cr, Zn and Pb were found in Sungai Pinang which might pose a threat to the aquatic ecosystems [7]. Therefore, the determination of total concentration of the heavy metals in soils surrounding the river of Sungai Pinang is a primary importance.

The accuracy in determining the heavy metals concentration had been a significant challenge encompasses the complexity of the involved matrices, the identification of reliable and fast methods for this application [8-10]. Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) has been the methods in soil contamination studies due to the utility, sensitivity and reliability of the equipment. This method able to rapidly analyse the heavy metals contents, after the pre-treatment process, in many types of contaminated soils which possess a very low detection limits for most of elements. The pre-treatment process, acid digestion, is time-consumed and presumably induced cross contamination of samples [10]. However, speedy pollution monitoring in the sense of urgency of detection is required to protect the public human health [11-12]. Therefore, X-Ray Fluorescence (XRF) technique emerged as the method befitting the vital criteria namely quick decision making in urgent situation, less involvement of sample handling and transporting, chain-of-custody documentation, less expensive per sample allowing more complete and denser sampling [11-12]. Furthermore, XRF technique is considered as efficient analysis with a very sensitive non-destructive and minimal sample preparation method for rapid and multi-element detection [10,13]. The objective of this study is to assess the presence of heavy metals and the concentration levels in the river sediment samples in the Sungai Pinang river basin and its five tributaries using ICP-OES and XRF methods.

2. Materials and methods

2.1. Study Site
The study was conducted at Sungai Pinang which located at the north-east of the Penang Island (5.4056°N, 100.3232°E) and its five tributaries. The total area and distance of Sungai Pinang river basin are 50.97 km$^2$ and of 3.1 km respectively [14-15]. Very dense population with various development such as industrial, housing area, commercial, slaughter house and others were observed along Sungai Pinang [14,16]. The river basin was polluted with many types of point sources and non-point sources as a result of a highly developed area. The results obtain may have some changes due to the river low-tide condition.

2.2. Sampling and Sample Preparation
A total of 97 samples were obtained four times within January to February. There were nine sampling points at the main river of Sungai Pinang namely Point 1, Point 2, Point 3, Point 4, Point 5, Point 6, Point 7 Point 8 And Point 9. At the tributaries, a measurement was taken at the upstream (US) and downstream (DS) of each tributary named; Sungai Air Terjun (SAT), Sungai Air Putih (SAP), Sungai Air Hitam (SAH), Sungai Dondang (SD) and Sungai Jelutong (SJ). All the sampling points were marked and identified using Global Positioning System (GPS) as shown in Table 1. To maintain the chemical and biological properties of the samples collected, the samples were stored in cool storage with temperature of 4 °C [17].
ed and ± 0.5 g of cooled samples and spectroflux powder were weighed. Then, the samples were placed on 0.45 µm membrane paper. Finally, the samples were cooled in a dessicator to a room temperature.

2.2.1. Digestion of Samples (EPA Method 3050B)

Acid digestion by Environmental Protection Agency (EPA) Method 3050B is required for identification of heavy metals by ICP-OES method. 1 g of sample was weighted in 250 ml of digestion flask before the sample was heated up to 95 °C with 10 ml of 50% HNO₃ without boiling. Then, the sample was left cooled to room temperature. The process was continued with repeated addition of 65% HNO₃ to reflux the sample until no brown fumes were observed in the sample. Next, the solution was allowed to evaporate until the volume reduces to 5 ml. After cooling the solution, 10 ml of 30% H₂O₂ was added slowly. Digestate solution was filtered using 0.45 µm membrane paper. The solution was diluted to 100 ml using deionized water. The solution samples were stored at 4 °C for ICP-OES analysis [18].

2.3. Sample Analysis

2.3.1. Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

The test was performed using Inductively Coupled Plasma (ICP) Varian 715ES to determine the minor and major metal elements present in the soil sample. For each element, the detection limit is different which is in the range of 0.05 to 500 µg/L. ICP-OES working with 2 ml of conical quartz glass nebulizer, cyclone spray chamber and torch made up of quartz glass. The digested sample can be analysed using ICP Varian 715ES equipment.

2.3.2 X-Ray Fluorescence (XRF)

In this test, the sample was converted to fused-glass bead type. Firstly, 1-2 g of samples was placed into a clean and dry Petri dish using spatula and oven-dried for one hour at 105 °C. Afterwards, the samples were cooled in the dessicator. Then, ± 0.5 g of cooled samples and spectroflux powder were weighted and using spatula the sample was mixed in the crucibles for few minutes until it is homogenous. The sample-spectroflux were placed into the fused-glass bead automatic machine. The sample-spectroflux were heated to temperature of 1100 °C for 25 minutes. Concurrently, the samples were moved in a few directions to make it homogenous. After 25 minutes, the machine automatically moved the samples to Pt-Au crucibles and started the cooling process. The fused-glass bead was inserted into the Panalytical Axios Max equipment.

3. Results and Discussion

3.1. Main River of Sungai Pinang

Based on the Table 2 obtained for the main river of Sungai Pinang, the highest concentration of SiO₂ was recorded at Point 2 (Lorong Kulit) with the value of 74.18%. The high percentage of SiO₂ may be due to the existence of silica. Silica has numerous health impacts such as kidney damage, rheumatoid arthritis and others [19]. The highest concentration of Al₂O₃ was recorded at Point 9 (Fishery Area) with the value of 18.52%. Point 9 is located near the boat activities and fisheries industry, which may release fuel into the river. Also, the street food shop at the area disposed their runoff directly into the river. During the sampling work, the sample taken was near the bauxite where it is known as the main source of Al₂O₃ given the higher percentage of Al₂O₃.

Furthermore, the highest level of K₂O was recorded at Point 2 with the percentage of 2.80. The presence of K₂O may imposed health issues such as cough, shortness of breath and sore throat. Besides, Fe₂O₃ also present with the highest value of 3.39% at point 9. The high concentration of Fe₂O₃ were

### Table 1. Coordinate of sample points along Sg. Pinang

| Point | Location | Latitude     | Longitude    | Point | Location | Latitude     | Longitude    |
|-------|----------|--------------|--------------|-------|----------|--------------|--------------|
| 1     | SgPinang | 5.411811     | 100.309035   | 6     | SgPinang | 5.407817     | 100.326087   |
| 2     | SgPinang | 5.411209     | 100.309820   | 7     | SgPinang | 5.405086     | 100.325449   |
| 3     | SgPinang | 5.410824     | 100.313192   | 8     | SgPinang | 5.404376     | 100.326320   |
| 4     | SgPinang | 5.410176     | 100.316128   | 9     | SgPinang | 5.404086     | 100.329742   |
| 5     | SgPinang | 5.408217     | 100.322856   |       |          |              |              |
recorded due to the possibilities of rusting of anchor arrow boat when continuously exposed to salt water given rusting of the metal was the main source of Fe₂O₃. Longer exposure to Fe₂O₃ may cause cough and shortness of breath.

Table 2. The percentage of heavy metals using XRF for Sungai Pinang sediment samples (%).

| Sampling Points | Concentration of heavy metals using XRF for Sungai Pinang sediment (%) |
|-----------------|-----------------------------------------------------------------------|
| P1 (Jalan Air Hitam) | SiO₂ 72.78 TiO₂ 0.24 Al₂O₃ 10.36 Fe₂O₃ 1.26 MnO 0.06 MgO 0.16 CaO 0.21 Na₂O 2.64 K₂O 87.85 |
| P2 (Lorong Kulit)  | SiO₂ 74.18 TiO₂ 0.20 Al₂O₃ 9.10 Fe₂O₃ 1.11 MnO 0.06 MgO 0.17 CaO 0.21 Na₂O 2.80 K₂O 87.99 |
| P3 (Jalan Perak)   | SiO₂ 73.27 TiO₂ 0.23 Al₂O₃ 9.76 Fe₂O₃ 1.39 MnO 0.01 MgO 0.07 CaO 0.14 Na₂O 0.15 K₂O 2.16 |
| P4 (Jalan Patani)  | SiO₂ 73.73 TiO₂ 0.23 Al₂O₃ 8.97 Fe₂O₃ 1.25 MnO 0.01 MgO 0.14 CaO 0.12 Na₂O 0.26 K₂O 87.00 |
| P5 (Taman Sri Pinang) | SiO₂ 67.75 TiO₂ 0.23 Al₂O₃ 8.61 Fe₂O₃ 2.97 MnO 0.01 MgO 0.37 CaO 0.70 Na₂O 1.22 K₂O 2.37 |
| P6 (Taman Lembah Sungai) | SiO₂ 60.98 TiO₂ 0.36 Al₂O₃ 13.86 Fe₂O₃ 2.82 MnO 0.02 MgO 0.62 CaO 0.30 Na₂O 0.66 K₂O 2.17 |
| P7 (SJK(T) Sg Pinang) | SiO₂ 69.53 TiO₂ 0.25 Al₂O₃ 11.20 Fe₂O₃ 2.25 MnO 0.01 MgO 0.17 CaO 0.26 Na₂O 0.40 K₂O 2.36 |
| P8 (Masjid Maqbul, Jln Jelutong) | SiO₂ 64.94 TiO₂ 0.25 Al₂O₃ 12.85 Fe₂O₃ 1.82 MnO 0.01 MgO 0.24 CaO 0.19 Na₂O 0.63 K₂O 2.20 |
| P9 (Fishery Area)  | SiO₂ 52.66 TiO₂ 0.42 Al₂O₃ 18.52 Fe₂O₃ 3.39 MnO 0.02 MgO 0.74 CaO 1.23 Na₂O 2.55 K₂O 80.22 |

Based on Table 3, the highest concentration of Ca, Cr, Pb and Zn was recorded at Point 5 with the value of 35.9576 mg/kg, 17.0469 mg/kg, 4.6901 mg/kg and 5.8392 mg/kg respectively. Point 5 located at high rise building and commercial area where the runoff from these activities gives a contribution to rising concentrations of Zn and Ca. A previous study found that the urbanization growth contribute to the increase of Ca concentration in sediment [20]. The highest concentration of As was recorded at Point 1 with the value of 0.8660 mg/kg. Besides, point 9 has the highest concentration of Mg and Fe with the concentration of 20.2590 mg/kg and 199.914 mg/kg respectively given the location is near the fisheries area. The permissible limit for Fe according to European Regulatory Standards (EURS) and World Health Organization (WHO) are 1500mg/kg and 50000mg/kg respectively, thus the Fe content can be considered at the safe level as it does not exceed the permissible limit.

Table 3. Concentration of heavy metals using ICP-OES for Sungai Pinang sediment (mg/kg).

| Sampling Points | Concentration of heavy metals using ICP-OES for Sungai Pinang sediment (mg/kg) |
|-----------------|--------------------------------------------------------------------------------|
| As  | Ca  | Cd  | Co  | Cr  |
| P1  | 0.8660 | 7.0339 | 0  | 0.0051 | 0.9487 |
| P2  | 0.0506 | 7.1209 | 0.2923 | 0.4094 | 0.9815 |
| P3  | 0  | 8.5463 | 0.0744 | 0  | 1.5122 |
| P4  | 0  | 20.2590 | 0.1673 | 0.1220 | 3.2122 |
| P5  | 0.6315 | 35.9576 | 0  | 0.6052 | 0.8591 |
| P6  | 0  | 16.7511 | 0.2219 | 0.1773 | 0  |
| P7  | 0.1873 | 11.7001 | 0.1301 | 0.2175 | 0.8841 |
| P8  | 0.6406 | 25.3269 | 0.8634 | 0  | 17.0469 |
| P9  | 0  | 21.7084 | 0  | 0  | 1.1206 |
Table 3. Concentration of heavy metals using ICP-OES for Sungai Pinang sediment (mg/kg) (continued).

| Sampling Points | Concentration of heavy metals using ICP-OES for Sungai Pinang sediment (mg/kg) |
|-----------------|---------------------------------------------------------------------------------|
|                 | Mg  | Fe    | Ni    | Pb    | Zn    |
| P1              | 2.2760 | 67.8679 | 0.4821 | 0     | 2.5151 |
| P2              | 2.9580 | 56.6231 | 0.5760 | 3.0762 | 1.4502 |
| P3              | 2.7154 | 77.3047 | 0.2224 | 2.3735 | 1.7799 |
| P4              | 7.9108 | 85.7387 | 0     | 0     | 4.1868 |
| P5              | 18.3765 | 152.6620 | 1.7557 | 0.9848 | 5.8392 |
| P6              | 24.0283 | 133.0080 | 0     | 4.6901 | 2.1064 |
| P7              | 5.8456 | 71.6660 | 0     | 1.7364 | 3.6868 |
| P8              | 25.8065 | 0     | 0.5467 | 0.1064 | 4.4358 |
| P9              | 39.5626 | 199.9140 | 0     | 0     | 2.9388 |

3.2. Tributaries

Based on Table 4, SAH (DS) has the highest percentage of SiO$_2$ with the value of 93.44%. The concentration of K$_2$O can be seen higher at upstream of Sungai Dondang with the value of 3.06% because it located within housing area while the highest concentration of Fe$_2$O$_3$ with the value of 0.90% recorded at the downstream of Sungai Dondang. The highest concentration of Al$_2$O$_3$ was recorded at Sungai Air Terjun (downstream) with the value of 6.75%. Al$_2$O$_3$ is commonly used as the component of paints and varnishes and in the manufacture of alloys, ceramics, glass, electrical insulators and resistors. Other types of heavy metals analysed do not show any significant values as there only low level of concentration.

Table 4. The percentage of heavy metals using XRF for tributaries sediment (%).

| Sampling Points | Percentage of heavy metals using XRF for tributaries sediment (%) |
|-----------------|------------------------------------------------------------------|
|                 | SiO$_2$ | TiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$(t) | MnO | MgO | CaO | Na$_2$O | K$_2$O | Total |
| SAT(US)         | 87.59     | 0.22     | 6.62       | 0.80         | 0.01 | 0.09 | 0.09 | 0.25     | 1.77     | 97.45     |
| SAT(DS)         | 87.76     | 0.22     | 6.75       | 0.80         | 0.01 | 0.08 | 0.13 | 0.27     | 1.65     | 97.71     |
| SAP(US)         | 90.92     | 0.17     | 4.80       | 0.76         | 0.01 | 0.08 | 0.07 | 0.02     | 0.98     | 97.83     |
| SAP(DS)         | 92.55     | 0.15     | 3.62       | 0.66         | 0.01 | 0.09 | 0.07 | 0.02     | 0.89     | 98.08     |
| SAH(US)         | 87.98     | 0.12     | 6.51       | 0.89         | Bdl  | 0.07 | 0.08 | 0.03     | 0.66     | 96.35     |
| SAH(DS)         | 93.44     | 0.04     | 3.39       | 0.29         | Bdl  | 0.07 | 0.08 | 0.12     | 0.96     | 98.41     |
| SD(US)          | 86.77     | 0.10     | 6.37       | 0.70         | Bdl  | 0.12 | 0.14 | 0.51     | 3.06     | 97.81     |
| SD(DS)          | 87.52     | 0.18     | 6.49       | 0.90         | 0.01 | 0.09 | 0.09 | 0.11     | 1.68     | 97.11     |
| SJ(US)          | 90.57     | 0.08     | 4.32       | 0.52         | Bdl  | 0.12 | 0.25 | 0.64     | 1.79     | 98.34     |
| SJ(DS)          | 90.37     | 0.13     | 3.96       | 0.64         | Bdl  | 0.12 | 0.36 | 0.35     | 1.38     | 97.36     |

Based on Table 5, the most significant heavy metals concentration present at the tributaries were Zn, Fe, Mg, Pb and Ca. The highest concentration of Zn, Fe, Mg and Pb were 1.8013mg/kg at Sungai Jelutong (downstream), 56.4778 mg/kg at Sungai Air Hitam (upstream), 3.3633mg/kg at Sungai Dondang (upstream) and 7.1662mg/kg at Sungai Air Terjun (upstream). The highest concentration of Ca was at SJ (US) with 34.8247 mg/kg as the sampling point was located within housing area. As the dumping of untreated wastes directly into the ditch or drain are the cause of increasing in the concentration of Ca. The population density within high rise was apparently higher than the landed houses. Thus, waste production of high-density population area is higher as compared to medium and low density. Besides, agricultural activity within the houses area could contribute to Ca available [20].
Table 5. The concentration of heavy metals using ICP-OES for tributaries sediment (mg/kg)

| Sampling Points | As    | Ca    | Cd    | Co    | Cr    |
|-----------------|-------|-------|-------|-------|-------|
| SAT(US)         | 0.3522| 2.2491| 0.0414| 0     | 0.4954|
| SAT(DS)         | **0.0312** | 13.6815 | 0.1334 | 0     | 0.4874|
| SAP(US)         | 0     | 1.6994| 0.1240| 0     | 0.6810|
| SAP(DS)         | **0.6406** | 4.7385 | 0.0754 | 0     | 0.5325|
| SAH(US)         | 0     | 5.1826| 0.1522| 0     | 0     |
| SAH(DS)         | 0.1667| 4.3302| 0     | 0     | 0.9929|
| SD(US)          | 0.1206| 6.1060| 0.0861| 0     | **0.1986** |
| SD(DS)          | 0     | 2.7353| **0.0062** | 0     | 1.2635|
| SJ(US)          | 0     | 34.8247| 0     | 0.1469 | 0.9828|
| SJ(DS)          | **0.5134** | 16.0435 | **0.1783** | **0.3032** | 0.3731|
| SAT(US)         | 3.3562| 43.0781| 0.1521| 7.1662| 0.9821|
| SAT(DS)         | 1.9559| 49.0656| 0     | 1.2127| 1.0144|
| SAP(US)         | 1.2370| 29.6240| 0     | 1.4186| **0.9122** |
| SAP(DS)         | 2.3930| 38.3782| 0     | 2.1356| 1.0738|
| SAH(US)         | **1.3864** | **56.4778** | 0.9675 | 0     | 1.0346|
| SAH(DS)         | 2.4911| 50.3205| 0     | **0.5338** | 1.0283|
| SD(US)          | **3.3633** | 36.5786| 0     | 1.0165| 0.9441|
| SD(DS)          | 1.8830| 41.6055| **1.6865** | 1.0290| 1.0290|
| SJ(US)          | 2.6648| **18.5342** | 0     | 1.4417| 1.8013|
| SJ(DS)          | 2.8509| 37.4075| 0     | 1.7619| 1.8013|

4. Conclusion

In conclusion, ICP-OES detected the presence of several types of heavy metals namely Zn, Pb, Ni, Fe, Mg, Cr, Co, Cd, Ca and As. Whilst, XRF test detected the presence of Na₂O, SiO₂, Al₂O₃, MgO, TiO₂, Fe₂O₃, CaO and K₂O. The highest concentration of K₂O, Fe₂O₃, Al₂O₃ and SiO₂ at the main river were 2.80%, 3.39%, 18.52%, and 74.18% respectively. The highest concentration of Ca, Cr, Zn, Mg and Fe recorded at main river are 35.9576, 17.0469, 5.8392, 39.5626 and 199.914mg/kg respectively. SAH (DS) has the highest percentage of SiO₂ with the value of 93.44% followed by Al2O3 with value of 6.75% at SAT(DS) and K₂O at SD (US) with the value of 3.06%. The highest concentration of Fe, Ca, Mg and Pb are 56.4778 mg/kg at SAT (US), 34.8247 mg/kg at SJ (US), 3.3633mg/kg at SD (US) and 7.1662mg/kg at SAT (US). The pollutants presence in the sediments of Sungai Pinang as well as its tributaries are resulting from the commercial, industrial, workshops, residential houses, street food stalls and others.

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