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Distribution of Mercury in Soil, Water, and Vegetable Fern in a Former Gold Mining Area – Evidence from Nagan Raya Regency, Aceh Province, Indonesia

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ABSTRACT

The mercury contamination associated with the former intense illegal gold mining activities is suspected in the watershed of Krueng Cot Satu, Nagan Raya Regency, Aceh Province, Indonesia. The aim of this study was to evaluate the mercury contamination residue in the water, soil, and vegetable fern (*Pityrogramma calometanos* (L)) The samples were collected from locations in the already closed artisanal gold mining sites. The sampling locations were purposively determined by considering their closeness to the previous gold mining activities sites. The content of mercury was analyzed using flow injection for atomic spectroscopy – atomic absorption spectroscopy. The method used was validated by linearity, Limit of Detection (LoD), Limit of Quantification (LoQ), Relative Standard Deviation (RSD), and recovery. The validation test showed that this method is well linear, sensitive, accurate, and precise with a correlation coefficient, LoD, LoQ, RSD and recovery of 0.9999, 0.0477 μg/L, 0.1447 μg/L, 2.96% and 95–105%, respectively. Herein, it was found that the concentrations of mercury contents in the water samples were below the detectable range. However, a high range of mercury concentration of 0.236 – 0.328 μg/g was found in soil, with the highest concentration obtained in the sample collected from the riverbank. The fern sample collected near the riverbank contained mercury in all its parts and concentrated in the root (0.408 μg/g in the leaves, 0.276 μg/g – stalks, and 9.994 μg/g – roots). Meanwhile, the absence of mercury contamination was obtained in the leaves and stalks of the fern samples collected far from the riverbank. The roots, however, were detected with mercury contamination with the highest concentration reaching 27.660 μg/g. Despite its disappearance in the water, mercury contamination residue from the former artisanal gold mining activities still could be traced in the soil and heavy metal accumulating plant – *P. calometanos* (L).

Keywords: Aceh, Artisanal gold mining, mercury, ferns, *Pityrogramma calometanos.*

INTRODUCTION

The presence of heavy metals in environment is yet still a common problem to this date. The heavy metal could originate from natural activities, such as volcanoes [Mandon et.al, 2019], hot spring manifestations [Idroes et.al., 2019a], erosion [Fang et al, 2016], geysers [Ciesielczuk, 2013], and fumarole [Idroes et.al, 2019]. Moreover, anthropogenic activities also contribute to the heavy metal pollution, including industries [Hoang et al., 2021], mining [Wahidah, 2019], or agriculture [Seiler and Berendonk, 2012]. Dynamic environmental system causes the widespread heavy metal pollution from one ecosystem to another. The heavy metals present in the soil could be transferred to plants, animals, and human. Heavy metal could have a detrimental effect on human health due to its ability of interacting and accumulated in human body via protein [Suhartono, 2018], blood, tissues [Fazio, 2014] or even bones [Suhartono, 2019]. Furthermore, heavy metal contamination could be transported by water and contaminate the aquatic ecosystem, including the sediment and aquatic organisms [Liu, 2014].

Illegal gold mining in Indonesia, as reported by others, has caused a mercury pollution [Rozo, 2020], followed by the degradation of environmental quality [Spiegel et al., 2018]. Due to its sensitive nature, the illegal gold mining and processing have become a primary source of severe heavy metal pollution that is significant and controllable. Gold is separated from its ore using mercury [Torkaman et al., 2021]. Usually, the processing site is located near the watershed [González-Valoys et al., 2022]. It is ascribed to the fact that the processing requires a lot of water, and in addition, its location near the watershed would ease the waste discharge [Saniewska et al., 2022]. The waste effluent is discharged directly to the river; meanwhile, the solid waste is stacked around the site without proper management. Aceh is a Province with many illegal gold mining practices that are located in Nagan Raya Regency [Zulkarnaini, 2019]. On the basis of authors' observation on the site, the gold processing sites tend to be located near the community residence. At the moment, the illegal gold mining and processing sites have been identified and closed by the government, but its heavy metal residue still impacts the surrounding environments [Neto & Soares, 2021].

Land reclamation of the heavy metal contaminated location is required to be carried out to recover the ecosystem, especially nearby the community living area and watershed area [Niu et al., 2021]. This is owing to the fact that human activities and river flows could increase the spread of the heavy metal contamination. One of the land reclamation approaches is by utilizing plant-based biosorbents [Li et al., 2015; Zhao et al., 2018; Makarova et al., 2022]. Nonetheless, the method tends to face challenges in determining the type of suitable plant for the specific climate and geographical condition of the intended reclamation sites [Narayanasamy, Sundaram, & Vo, 2022]. Indeed, there is another alternative of using a biobased adsorbent, but it could not be an option due to its high-price production [Rahmi, et al., 2022; Rahmi, Lelifajri, et al., 2022].

Among many biosorbent plants, the one that is massively abundant in the watershed is vegetable fern. This devision plant is commonly found in the wild with humid condition, such as river banks [Nagalingum et al., 2008]. A previous report suggested that the devision plant of Pteridophyta could absorb excessive contaminants, either organic or inorganic [Alizadeh et al., 2022]. In Aceh itself, ferns could be found thriving along the watershed. The plant is commonly used by the village communities as vegetable. Vegetable ferns have become one of local delicacies, known as *paku teu peuleumak* (translated as coconut milk-based soup of vegetable ferns). Therefore, it is crucial to perform the analysis of the mercury content in the vegetable ferns growing in the area of former illegal gold mining. Other than investigating its potential as mercury biosorbent, the analysis is required to map the mercury pollution on the plant (leaves, stems, and roots) which is useful when processing the vegetable as food ingredient.

MATERIALS AND METHODS

Materials

The chemicals used in this research were Argon gas, HNO_3, HCl, and $Hg(NO_3)_2$ – for standard solution. All chemicals were analytical grade and procured from Merck (Selangor, Malaysia). The water, soil, and vegetable fern samples were collected from the locations described in the following sections.

Sampling techniques

The sampling locations of this research were former illegal gold mining and processing sites located near the watershed of Krueng Cot Satu in Panton Bayam Sub-District, Nagan Raya Regency, Aceh Province, Indonesia. The sampling was carried out on 21st March 2021.

Determination of sampling points

Three sampling points for soil and fern samples were determined purposively in three different locations which were formerly used as gold mining and processing sites, labeled as point I, II, and III. The distribution of the locations was presented in a map (Figure 1). Sampling point 3 was located in

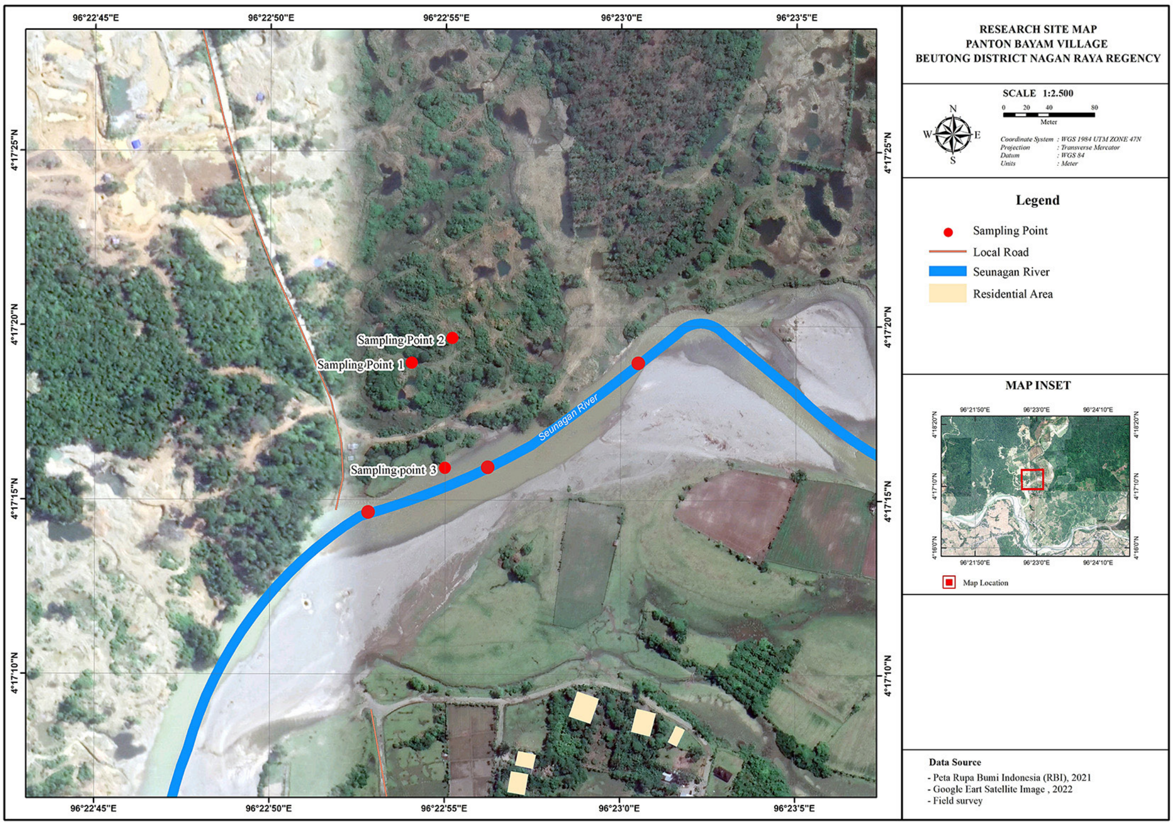

Figure 1. Sampling point of soil and fern plant in former illegal gold mining in Krueng Cot Satu, in Panton Bayam Sub-District, Nagan Raya Regency, Aceh Province, Indonesia

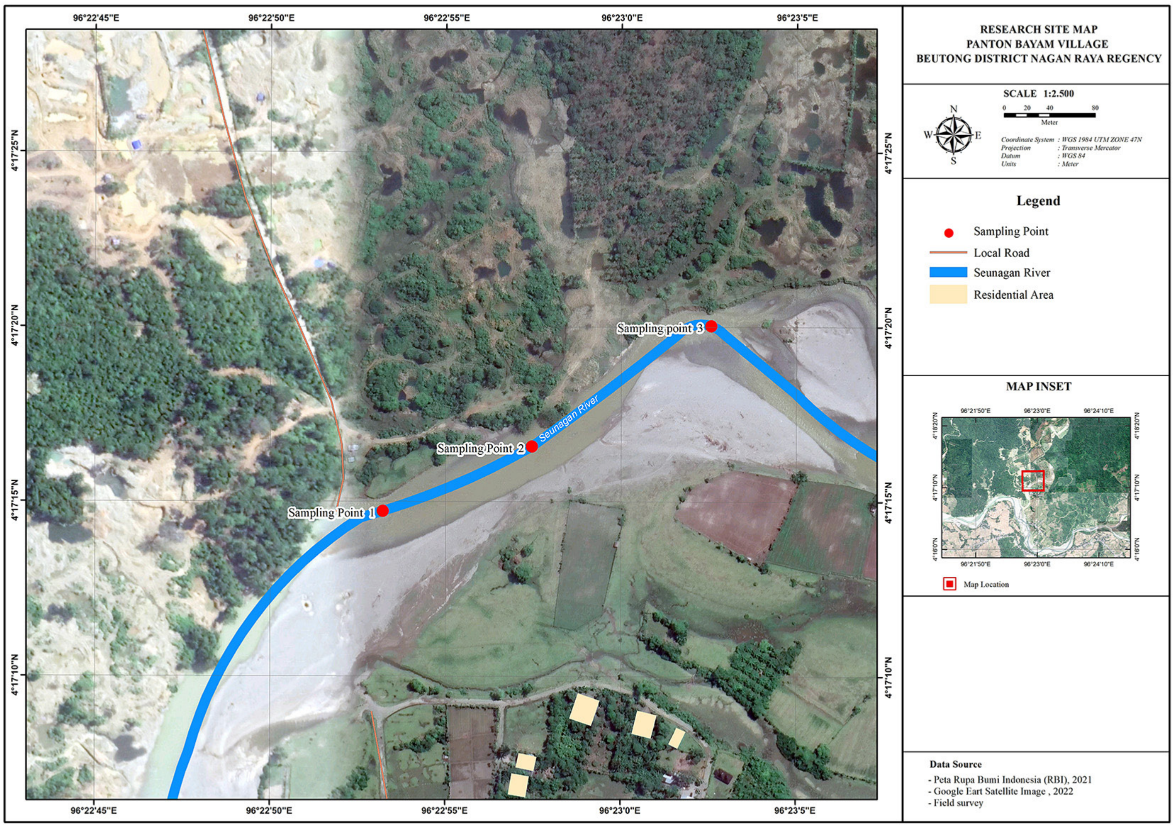

Figure 2. Sampling point of soil and fern plant in former illegal gold mining in Krueng Cot Satu, in Panton Bayam Sub-District, Nagan Raya Regency, Aceh Province, Indonesia

the riverbank of Krueng Cot Satu, which was assumed as a final point of the mercury-contaminated wastewater flow before entering the river. Three sampling points for water sample were also determined purposively in the river flowing through the former illegal gold mining (Figure 2).

Soil sample collection

The sediment samples were collected using stainless steel scoop in the predetermined locations of former gold mining and processing. The samples were stored in a plastic container, labeled according to the locations (for example SS-01 for soil sample from sampling point 1), and transported to the laboratory.

Fern sample collection

Several ferns grew in the three predetermined sampling points (1, 2, and 3) were randomly chosen and pulled out using stainless steel scoop from the soil. The samples were collected without considering the age criteria. The plants were placed in a polyethylene bag with soil before being transported to the laboratory. Taxonomic examination was carried out in the Laboratory of Biology, Universitas Syiah Kuala according to the published guideline [Sundra, 2016]. It is then known that the species of the plant was *Pityrogramma calometanos* (L) with its detailed taxonomic identities presented in Table 1.

Water sample collection

The water samples were collected using a plastic scoop and stored in a polyethylene bottle, which was priorly washed with HNO_3 (once) and the water sample (three times). All samples were preserved by reducing their pH level to pH<2 with HNO_3 and stored in a refrigerator at 4°C ± 2°C [SNI, 2021]. Each of the water samples was labeled according to the sampling point (for example WS-01 for the water sample collected from sampling point 1). All samples were transported to the laboratory for mercury content determination using flow injection for atomic spectroscopy – atomic absorption spectroscopy (FIAS-AAS).

Table 1. Taxonomic identity of the collected fern

Taxonomic ranks	Name
Kingdom	Plantae
Sub kingdom	Tracheobionta
Division	Pteridophyta
Class	Filicopsida
Sub class	Polypoditae
Order	Polypodiaes
Family	Pteridaceae
Genus	*Pityrogramma* Link
Species	*Pityrogramma calometanos* (L) Link

Sample preparation

Soil sample preparation

Plastic debris and leaves were separated from the soil sample, and then air-dried at room temperature. The dried sample was crushed homogenously and sieved (100 mesh). The sample was weighed at 3 g and inserted into a vessel, added with 25 mL distilled water, and rigorously shaken. Into the mixture, 10 mL HNO_3 and 5 mL HCl were added. The vessel container was sealed and adjusted with a vessel spanner before being inserted into a digestion microwave, run for 15 minutes. The obtained liquid was filtered and dissolved in distilled water up to 100 mL.

Fern sample preparation

Each fern sample was cut according to its parts (leaves, stalks, and roots). Afterwards, the size of each part was reduced using a stainless-steel knife. The sample was air-dried at room temperature before crushed until homogenous. As much as 3 g sample powder was inserted into the vessel container, added with 25 mL distilled water, and rigorously shaken. Then, 15 mL solution of HNO_3:HCl (2:1) was added into the sample mixture, sealed, and adjusted in the vessel spanner in a Microwave Digestion System (run for 15 minutes). The liquid produced thereafter was filtered and dissolved in distilled water until the volume reached 100 mL.

Mercury analysis using FIAS-AAS

A sample in liquid form was injected into FIAS through autosampler. The determination of mercury content was performed at wavelength of 253.7 nm. Each sample was determined in triplicate [EPA, 2021].

Method validation

The parameters used for validation were linearity, precision, LoD, LoQ and recovery. The linearity is calculated by linear regression between the concentration and absorbance from five standard solution series. Precision is determined by standard deviation from three repition measurement of standard solution. LoD and LoQ are calculated by the acquisition of regression parameter from the calibration curve regression line. Recovery is determined measuring the known concentration of standard solution.

RESULTS AND DISCUSSION

Method validation

Linearity, limit of detection and limit of quantification

The standard solutions of Hg $(NO_3)_2$ were analyzed in concentration range of 0–50 µg/L. The absorbances obtained were plotted in linear regression using LINEST to obtain regression parameters (Table 2). The method used is well linear where the coefficient of determination obtained was 0.9999. On the basis of the standard deviaton of regression, the method used is highly sensitive, where the LoD and LoQ calculated were 0.0477 µg/L and 0.1447 µg/L, respectively.

Precision and recovery

The precision of method used was identified by testing of 10 µg/L standard solution in three repetitions. The result showed that the method was highly precise with % RSD of 2.96 % (Table 3). The accuracy of the method was determined by calculating the concentration of 10, 20 and 50 µg/L standard solution. The result showed that the method is highly accurate with the recovery in range of 95–105 % (Table 3).

Table 2. Linearity and uncertainty of calibration curve threshold

Parameters	Value
Slope	0.006197143
Intercept	0.000432143
Standar deviation of slope	0.000002395
Standard deviation of intercept	0.000065600
Coefficient of determination	0.999999701
Standard deviation of regression	0.000089600

Table 3. Recovery and relative standard deviation test

Recovery		% RSD
Standard solution test	% Recovery	
10	99.76	2.96
20	100.08	
50	96.78	

Mercury content in water

Identification of the mercury content in the river water of Kreung Cot Satu was performed to detect the source of pollution which possible affect the mercury content within the former gold mining area. The area was located at the riverbank which could have a contact with the water river when the water debit increases due to natural factors such as rain or flood. Initially, it was speculated that the mercury present in the river water might be attributed to many former gold mining sites surrounding the river, as reported by several studies [Aminah et al., 2021; Barron, 2019; Meilina and Ramli, 2021]. Moreover, the illegal gold processing is commonly known for its action of discharging the wastewater to the river [Basri and Prayudi, 2022]. Nonetheless, in this present study, the presence of mercury was not detected in all sampling points (Table 4). The absence of mercury could be influenced by geographical factor, phase concentraiton [Cui et al., 2021], salinity [Bełdowska et al., 2015], and the mixing of river and sea waters [Saniewska et al., 2022]. There is a possibility that the mercury has been carried to the ocean, as suggested by a previous research [Saniewska et al., 2022]. Hence, the mercury content left in the water sample is too small to be detected.

Mercury content in soil

The presence of mercury and its content level in soil samples have been presented in Table 5. The results revealed that all samples from the three sampling locations contained mercury ranging

Table 4. Content of mercury in water from Krueng Cot Satu River which shares the same location with former gold mining site

Coordinate	Water sample	
	Label	[Hg] (µg/L)
96° 22’ 53.439”E 4° 17’ 14.731”N	WS-01	Not detected
96° 22’ 57.684” E 4° 17’ 16.582” N	WS-02	Not detected
96° 23’ 2.791” E 4° 17’ 20.03E	WS-03	Not detected

Table 5. Content of mercury in the soil samples collected from Krueng Cot Satu River which shares the same location with former gold mining site

Coordinate	Soil sample	
	Label	[Hg] (μg/g)
4°17'19.04"N 96°22'55.46"E	SS-01	0.271
4°17'19.42"N 96°22'56.46"E	SS-02	0.236
4°17'17.48"U 96°22'57.03"E	SS-03	0.328

between 0.236 and 0.328 μg/g. The highest mercury concentration (0.328 μg/g), was observed in sampling point 3, which was the location where the mercury entered the river. These data suggest that the mercury was carried from the land to the river by means of rain or erosion. The presence of mercury in the soil sample, and not in the water sample, indicates that the mercury was retained in the soil and possible distributed to somewhere else in the water. In a study, mercury could be reserved in the soil even for years [Zhou et al., 2015].

The mercury-containing soil is an indicator of food chain contamination, which attracts global attention [Fernandes et al., 2021]. The presence of mercury has been attributed to the antrophogenic activity, especially gold mining [Yoshimura et al., 2021]. Not only mercury, but also other heavy metals such as Cd, Cu, and Pb were found increased in area affected by artisanal gold mining [Nasir et al., 2021]. The people living near the contaminated locations could be exposed to mercury via wild vegetables grown therein, of which is vegetable fern which has been considered as local delicacies.

Mercury content in vegetable ferns

The contents of mercury in vegetable ferns (*P. calometanos* (L) Link) collected in this present study were presented in Table 6. The presence of mercury in the leaves and stalks of the ferns collected from sampling points 1 and 2 was not detected. Meanwhile, the sample from point 3 (riverbank) had mercury in all over its part. In contrast, the root parts of the ferns from all sample points (1, 2, and 3) were contaminated with mercury with concentrations ranging from 4.824 to 27.660 μg/L. As it can be seen, the presence of mercury in stalks and leaves is not correlated with the concentration of mercury in the root. However, the distribution of mercury in the parts of a fern could be associated with the mercury level in the soil, where the soil sample from point 3 had the highest concentration as compared with others. Therefore, it is not safe to consume the ferns that grow in the riverbank of Kreung Cot Satu. The representation image of mercury absorption by the fern collected from the riverbank area (point 3) and the area far from the riverbank (point 1 and 2) has been presented in Figure 3.

More importantly, it was noted that the smallest mercury concentration (4.824 μg/L) in the fern roots was more than 17 times higher than the concentration in the soil sample. Hence, it is stipulated that the mercury was absorbed and accumulated in the root of fern, proving its ability as biosorbent for mercury. Not only as biosorbent, the enrichment of heavy metal in a solid sample such as the root is beneficial for contamination the monitoring techniques using laser spectroscopy [Iqhrammullah et al., 2021; Nisah et al., 2022]. As suggested previously, plants act as direct receivers of heavy metal contaminant through water absorption [Pal & Sukul, 2022]. On the basis of the aforementioned study [Pal & Sukul, 2022], ferns absorb heavy metals along with the ability to accumulate and tolerate the heavy metal.

It is worth noting that *P. calometanos* is widely distributed in tropical region [Lianah et al.,2021], and usually grows near the ground water surface [Luthardt et al., 2021]. The plant itself has been used as integrative medicine for dysentery [Koniyo et al., 2019]. In addition, it has also been used by the locals to reduce the concentration of arsenic in the water [Koniyo et al., 2019].

Table 6. Mercury content in fern plant growing in former illegal gold mining

Parameter	Hg (μg/g)		
Sample	1	2	3
Location	4°17'19.04"N 96°22'55.46"E	4°17'19.42"N 96°22'56.46"E	4°17'17.48"U 96°22'57.03"E
Leaves	Not detected	Not detected	0.408
Stalks	Not detected	Not detected	0.276
Roots	4.824	27.660	9.994

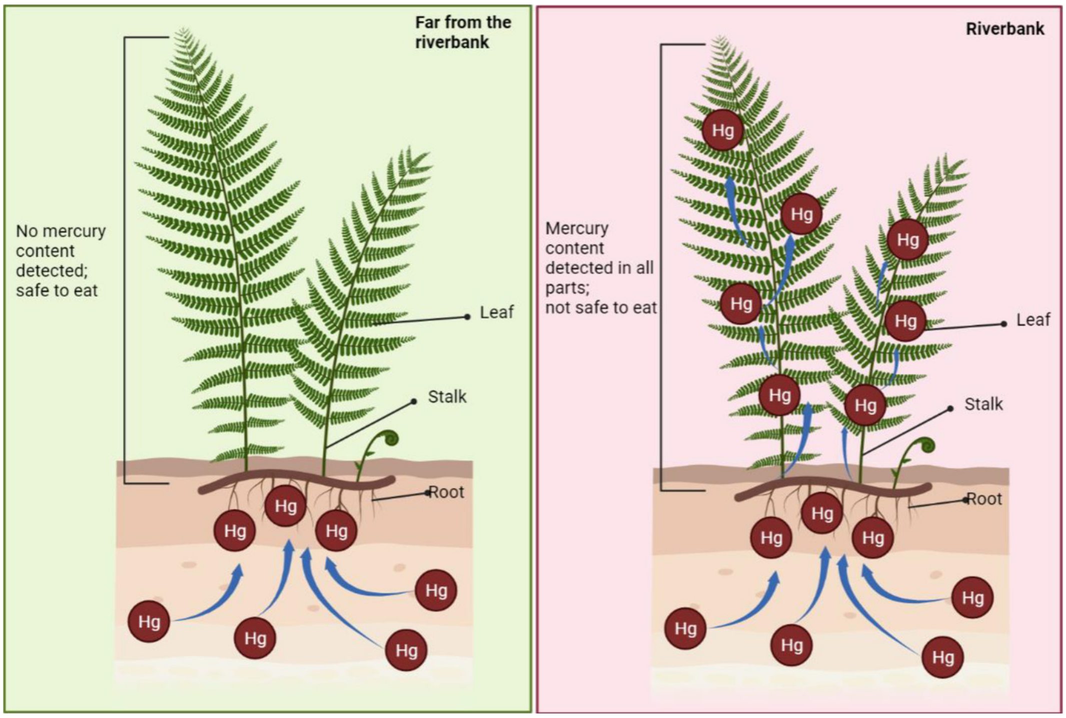

Figure 3. Representation of vegetable ferns absorbing Hg from the contaminated soil

The use of this plant for consumption should be taken carefully, by considering the level of heavy metal contamination in the place where the plants grow.

In the samples collected from sampling points far from the riverbank (point 1 and 2), the mercury was only detected in its root, while it was not detected in either the stalks or leaves. Hence, the stalks and leaves of a vegetable fern grow far from the riverbank is safe to eat. All parts of vegetable ferns collected from in the riverbank (point 3) contained mercury; hence, it is not safe to eat.

CONCLUSIONS

The residues of mercury from the former illegal gold mining and processing are still threatening the people living in Panton Bayam Sub-District, Nagan Raya Regency, Aceh Province, Indonesia. The determination of mercury content in this research was performed by means of the validated method. The validation test showed that this method is well linear, sensitive, accurate, and precise with a correlation coefficient, LoD, LoQ, RSD and recovery of 0.9999, 0.0477 μg/L, 0.1447 μg/L, 2.96 and 95–105%, respectively. Though the mercury is not found in water samples, its presence is still detected in high concentration range (0.236 – 0.328 μg/g) in the soil samples. The presence of mercury in soil could contaminate the wildly grown vegetables consumed by the locals, including vegetable ferns (*P. calometanos* (L)). For the vegetable ferns growing far from the riverbank, it is safe to consume their stalks and leaves but not the roots. However, for those that grow in the riverbank area, one should be cautious in terms of the Hg contamination in the leaves and stalks. Hence, the ban of fern consumption is recommended if it grows in the riverbank contaminated by high concentration of Hg. More investigations need to be carried out to fully understand the pattern of Hg distribution in the plant grown in contaminated soil in order to make more comprehensive regulation.

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