Research Article

Heavy metals and arsenic concentrations in water, agricultural soil, and rice in Ngan Son district, Bac Kan province, Vietnam

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Abstract

Due to the fast development in many industries such as metallurgical, mining, and chemical sectors in Vietnam, many issues related to environmental contamination have been raised. This study was conducted to briefly investigate the heavy metal levels in water, agricultural soil, and rice in Thuong Quan commune, Ngan Son district, Bac Kan province, Vietnam. The analytical results of 06 elements such as Arsenic (As), Cadmium (Cd), Copper (Cu), Lead (Pb), Nickel (Ni), and Zinc (Zn) showed the current status of agricultural soil, water, and rice quality in the vicinity of study area. For surface water and domestic water samples, the average level of six elements did not exceed the Vietnam standard. While the As levels in agricultural soil were 15.6 - 27.2 mg/kg which exceeded the Vietnam standard for agricultural soil. On the other hand, the total Arsenic (tAs) levels in rice were 0.18 - 0.40 mg/kg which not exceeded the Vietnam standard for polished rice. Throughout this study, tAs is the only element that needs to be further investigated from the relatively high concentration in agricultural soil samples. There were seven out of eight rice samples which had the As level exceeded the Codex standard for polished rice, the inorganic Arsenic (iAs) is also needed to be analyzed for further conclusion.

***Keywords**: ICP-MS, heavy metals, water, agricultural soil, rice.*

1. INTRODUCTION

Rice plays an important role in national food security and political stability in Vietnam and has a direct impact on social security because it is consumed by nearly 89 million of the total population. It is an important source of income for more than 60 million people living in agricultural and rural areas. Rice is the country's main crop, accounting for more than 90% of total cereal production in 2009 reached 38.89 million tons, about 14 million tons higher than in 1995 (General Statistics Office, 2009).

Based on the survey of Viet Nam's Ministry of Health (MOH) in 2004, the total daily food consumption was 388 g/person. About 376 g of rice are equivalent to 135 kg/person/year. The Vietnamese need about 2,400 kcal/person/day, of which 1,400 kcal is sourced from rice. It was also found that cereals remain the food group that provides the majority of calories in the diets of the Vietnamese [16]. Cereals, in which rice makes up the largest share, account for about 30% of expenditure but contribute more than 65% of a calorie per capita daily.

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The rice cultivated in the area near the mining site or using contaminated water for irrigation may have a negative influence on human health. Numerous researches on the impact of heavy metal contaminated soil and water on the quality of rice were conducted in Vietnam [4, 10, 11]. Heavy metals contamination was reported in agricultural land, food crops due to the discharge and dispersion of mine wastes [12]. The mining sector increases pollution, especially As in soil which can transfer to some plants such as rice, the tea plant, and cabbage. Ingestion of heavy metals through food and drinking water is a major exposure source for humans. Therefore, growing human foods in heavy metal contaminated media could lead to bioaccumulation of these elements in the human food chains from where these elements ultimately reach the human body [1]. Non-essential heavy metals (Cd, Pb, and Mercury, Hg) and metalloids (As, etc.) may be toxic even at significantly low concentrations. Essential heavy metals are required in trace levels in the human body but may become toxic beyond certain limits or threshold concentrations. For some elements, the window of essentiality and toxicity is narrow. Heavy metals have been reported to be carcinogenic, mutagenic, and teratogenic [6].

Because of these potential impacts on human health, this study was conducted to investigate the heavy metal (As, Cd, Cu, Pb, Ni, and Zn) concentrations in agricultural soil, water, and rice in the vicinity of Ngan Son district in Bac Kan province, Vietnam.

2. MATERIALS AND METHODS

2.1. Study area

Thuong Quan commune is in Ngan Son district, Bac Kan province, Vietnam. In this commune, there are numerous small and scattered mines. Based on the Planning for exploitation, mining, and using mineral in Bac Kan province, the period 2013 - 2020, mining activities are going to be expanded. Accordingly, a Lead-Zinc (Pb-Zn) refining factory with a capacity of 5,000 tons per year is going to be built in Ngan Son district [2]. The study area and location of sampling sites were shown in Figure 1 below.

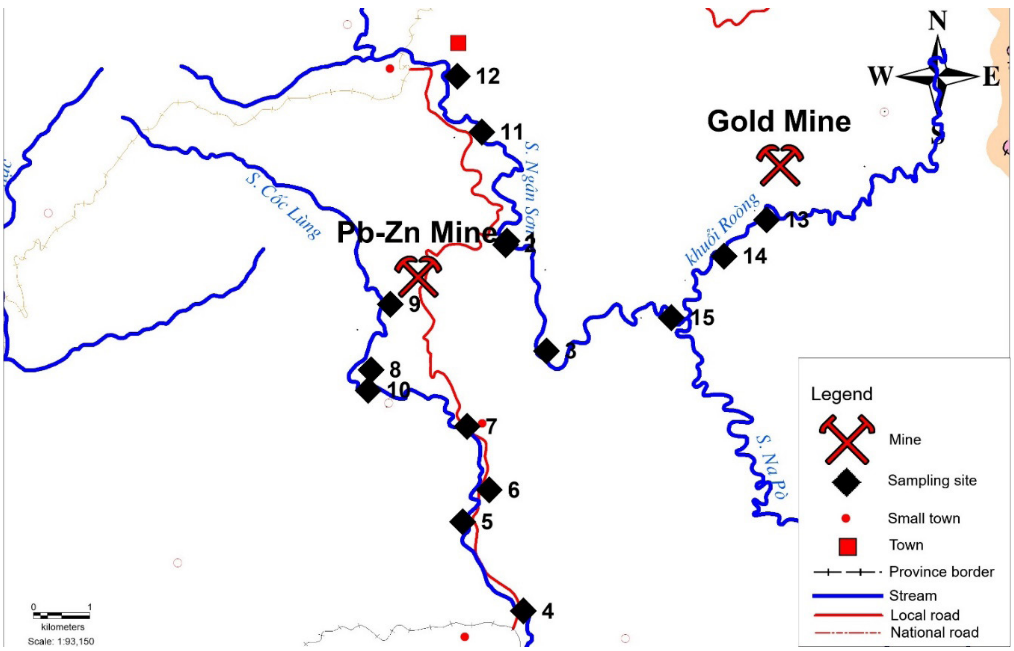

Figure 1. *Study map*

The local residents live in small villages scattered along the streams. The average annual rainfall, humidity, and temperature in Bac Kan province are 1,400 - 1,600 mm/year, 84%, and 20 - 22°C, respectively. The rainy season starts from May to October and the dry season starts from November to April.

2.2. Field sampling and sample preparation

Sampling was carried out in October 2019. The agricultural soil, surface water domestic water, and grain rice samples were collected along the streams running through the mining site in Thuong Quan commune. Because the study area is the mountainous terrain, streams run at the foot of the abysses so all the samples were collected based on the accessibility to areas near streams. In total, 15 surface water samples, five domestic water samples, eight agriculture soil samples, and eight rice samples were collected.

After the agricultural soil had been collected, they were separately packed in zipper bags in the field and transported to the laboratory. Samples were dried at 40°C until their weight was constant, sieved (100 mesh) to separate debris, and eventually kept in plastic bags (100 g). Surface water at depths 0 - 5 cm was collected from the stream which flows through the mining site. Domestic water was pumped from wells for 10 - 15 min before sample collection to flush out all retained water in the pipes. Water samples were filtered through disposable syringe membranes (0.45 µm), then transferred into 50 mL conical tubes, and kept in the dark at 5°C. Every water sample was acidified in the field with nitric acid. Grain rice sample was collected using clean disposable gloves, placed into individual plastic bags, and preserved using a handy cooler during sampling and transportation to the laboratory. In the laboratory, grain rice samples were separated into rice and husk, then thoroughly washed in de-ionized distilled water to remove dust particles adhered to the grain. After being dried in the oven at 40°C to a constant weight, the dry samples were ground to a fine powder using a miller.

2.3. Sample analyses

2.3.1. Equipment and Reagents

The main instruments were used in this study including a high-speed centrifuge (HANIL HA-1000-3, Republic of Korea); a heating block (Dry Thermo Unit DTU-2C); and Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Agilent 7500ce).

The reagents which were used for analysis in this study are the purity chemicals that meet the requirements for ICP-MS operation.

- HNO_3 70% - type: AR (Daejung Chemicals and Metals Co., Ltd, Republic of Korea)

- HCl 35% - type: AR (Daejung Chemicals and Metals Co., Ltd, Republic of Korea)

- Reference Standard Materials: Rice flour (NIST-1568a, National Institute of Standards and Technology)

- Reference Standard Materials: Contaminated soil (BAM-U110, Federal Institute for Materials Research and Testing, Germany)

2.3.2. Sample preparation

The agricultural soil samples were digested using a modified aqua regia method [17]. Briefly, 0.50 g of the dried sample was accurately weighed into a 15 mL polyethylene tube. Exactly

3.75 mL of HCl (35%) and 1.25 mL of HNO_3 (70%) were added and the mixture was allowed to stand overnight. The next morning, the mixture in the polyethylene tubes was heated at 96 ± 3°C for an hour using a heating block. After cooling down to room temperature, the aliquot was centrifuged at 1.824 × g-force (3,000 rpm, rotor radius 181.3 mm) for 10 min then filtered using 0.45 µm filtration paper. The supernatant was transferred to a fresh tube and stored at 4°C. Since the supernatant was highly acidic and contained high concentrations of elements, dilution with deionized water (1: 1,000) was required before analysis. Similarly, acid digestion was performed for rice and husk samples. Briefly, 0.30 g of sample was weighed into a 50 mL polyethylene tube 5 mL of the concentrated HNO_3 (70%) was added to each tube. The tube was then capped and left in a hood at room temperature. After 48 h, 15 mL of deionized water was added. The mixture was, then, filtered and kept in a fresh tube. All filtrates were stored in the refrigerator at 4°C until analysis [17]. Six elements including As, Cd, Cu, Pb, Ni, and Zn in water, soil, sediment, husk, and rice samples were analyzed by ICP-MS.

Digestions of two replicate samples were conducted. The Standard Reference Materials were also treated in the same manner as the samples to verify the accuracy of the digestion methods. The recovery rates from the digestions met the requirements of the certified values as shown in Table 1.

Table 1. *Certified values (mg/kg) and recovery rate (%) of elements of various Standard Reference Materials (SRM)*

	Ni	*Cu*	*Zn*	*As*	*Cd*	*Pb*
BAM-U110 certified values	95.6 ± 4.0	262 ± 9	990 ± 40	13.0 ± 1.1	7.0 ± 0.4	185 ± 8
Recovery Rate	105	110	96	110	102	105
SRM 1568a certified values	-	-	19.4 ± 0.5	0.29 ± 0.03	0.022 ± 0.002	-
Recovery Rate	-	-	88	104	103	-

3. RESULTS AND DISCUSSION

3.1. Heavy metals concentration in water samples

3.1.1. Heavy metals concentration in surface water samples

The analysis results of surface water samples were listed in Table 2.

Table 2. *Heavy metal concentrations in surface water (µg/L)*

Sample ID	*Ni*	*Cu*	*Zn*	*As*	*Cd*	*Pb*
W1	0.10	ND	1.51	3.78	0.02	0.22
W2	0.17	ND	39.5	1.80	0.02	0.58
W3	0.20	ND	3.16	4.07	0.03	1.97
W4	0.27	1.20	2.51	2.93	0.03	2.22

W5	0.61	0.45	8.07	4.43	0.11	8.15
W6	0.29	ND	1.97	3.87	0.04	2.85
W8	0.25	0.02	7.64	7.85	0.10	9.27
W9	0.17	ND	8.45	7.50	0.13	6.20
W10	0.40	0.07	6.58	4.65	0.09	7.17
W11	0.30	ND	3.08	4.81	0.06	2.72
W12	0.19	ND	4.62	5.32	0.07	4.12
W13	1.51	0.15	8.93	32.2	0.06	18.5
W14	1.09	ND	6.50	27.0	0.04	11.2
W15	0.19	ND	1.69	3.51	0.03	1.20
Min	0.1	0.02	1.51	1.80	0.02	0.22
Max	1.51	1.2	39.5	32.2	0.13	18.5
Average	0.41	0.38	7.45	8.12	0.06	5.45
Standard*	**100**	**200**	**1,000**	**20**	**5**	**20**

"ND": Not detected

"*": Vietnam standard for surface water QCVN 08-MT:2015/BTNMT (Ministry of Natural Resources and Environment)

From Table 1, there were no elements that have the concentration exceeded the Vietnam standard for surface water except As concentration in samples W13 and W14. These samples were collected from the nearest sampling sites to the gold mine. Interestingly, the level of Zn and Pb from these sites were also higher than other sites. On the other hand, the W8 and W9 samples collected at the nearest sampling sites to the Pb-Zn mine have slightly higher As concentration than those at other sites. However, As concentrations in those samples are lower than the Vietnam standard for surface water.

The abnormally high As concentration of metals in surface water in the sampling site W13 and W14 might result from human activities such as cultivation, livestock production, mining activities, etc. It is said that the mining activities could impact to surface and groundwater due to spill/tailing, erosion, sedimentation, acid mine drainage, lowering of the water table, subsidence, disturbance on the hydrological cycle, and rainfall [13]. And, As is the element which is available in almost every mineral [20]. However, the average concentration of all elements in this study did not exceed the Vietnam standard for surface water and domestic water. This indicated that the quality of surface water and domestic water samples were not at risk for the residences.

3.1.2. Heavy metals concentration in domestic water

In this study area, the households use groundwater from the wells as domestic water. Heavy metal concentrations in groundwater samples were given in Table 3.

Table 3. *Heavy metals concentration in domestic water (μg/L)*

Sample ID	Ni	Cu	Zn	As	Cd	Pb
G2	0.11	0.04	3.45	0.11	0.00	0.10
G3	0.25	5.15	8.34	0.28	0.01	0.46
G6	0.47	2.10	91.8	0.22	0.04	0.70
G7	0.31	0.40	3.77	0.11	0.04	0.17
G12	0.19	0.23	17.45	13.3	0.03	0.33
Min	0.11	0.04	3.45	0.11	0.00	0.1
Max	0.47	5.15	91.8	13.3	0.04	0.7
Average	0.27	1.58	25	2.8	0.02	0.35
Standard*	**70**	**1,000**	**2,000**	**10**	**3**	**10**

"*": Vietnam standard for domestic water QCVN 01-1:2018/BYT (Ministry of Health)

In general, the heavy metals concentrations in domestic water samples do not exceed the Vietnam standard for domestic water except for the As concentration in sample G12. The site of sample G12 was upstream of gold and Pb - Zn mines therefore, it might be the reason for the higher As level in the W12 sample as mentioned above.

3.2. Heavy metals concentration in agricultural soil

Agriculture soil samples were analyzed for heavy metals and the results are shown in Table 4 and Figure 2. In this figure, the sampling sites were arranged according to the distance from the mining sites, the sampling sites near the mining sites the most were in red squares.

Table 4. *Heavy metals level in agricultural soil (mg/kg)*

Sample ID	Elements					
	Ni	Cu	Zn	As	Cd	Pb
D2	30.6	47.3	75.3	26.6	0.33	46.8
D3	33.9	67.8	79.8	15.6	0.20	18.4
D4	36.1	55.4	80.5	20.4	0.75	39.5
D6	37.1	42.6	83.5	26.5	0.30	31.8
D7	41.4	41.2	80.5	17.8	0.37	42.8
D8	38.3	55.9	79.1	19.4	0.46	46.2
D12	44.9	52.5	87.8	26.9	0.19	24.2
D13	33.0	59.0	88.5	27.2	0.94	56.5
Min	30.6	41.2	75.3	15.6	0.19	18.4
Max	44.9	67.8	88.5	27.2	0.94	56.5
Average	36.9	52.7	81.9	22.6	0.44	38.3
Standard*	-	**100**	**200**	**15**	**1**	**70**

"*": Vietnam standard for agriculture soil QCVN 03-MT:2015/BTNMT (Ministry of Natural Resources and Environment)

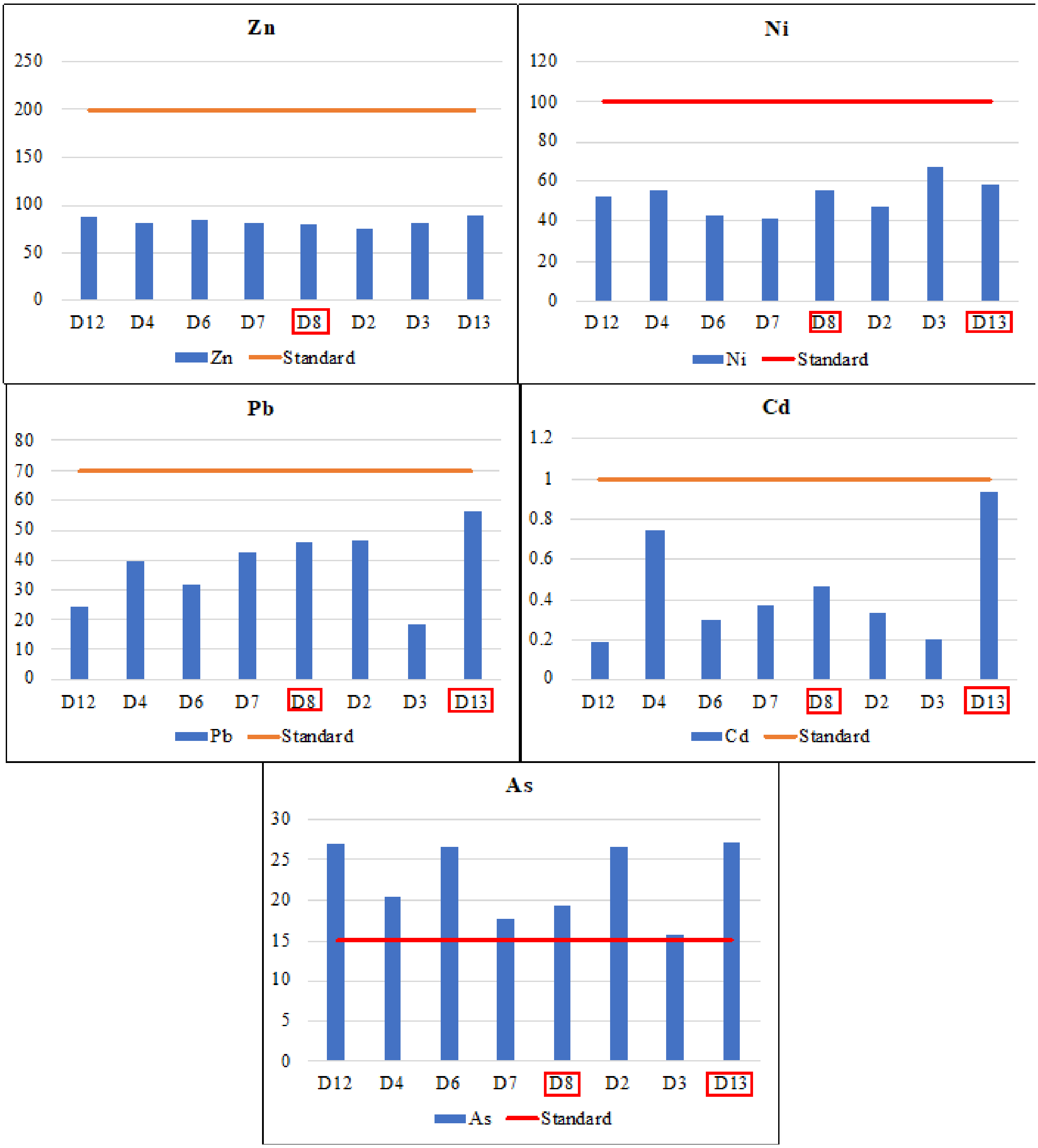

Figure 2. *Comparison between heavy metal level in agricultural soil and Vietnam Standard for Agricultural Soil (mg/kg)*

As shown in Table 4, the level of Ni, Cu, As, Cd, and Pb do not exceed the Vietnam standard for agricultural soil. However, all As level in soil samples exceed the standard. The source of heavy metals contamination in agricultural soil could be natural occurrence, anthropogenic (using pesticides, mining activities,…) sources, or both of them [18]. The high accumulation of heavy metals in agricultural soil is a potential risk for food safety because rice and other food crops are usually cultivated in the topsoil and anthropogenic heavy metals are deposited there [8]. Ko et al., 2020 reported that the average levels of As and Cd in paddy soil near the Nui Phao mining site were found to be 45.9 and 6.6 mg/kg, respectively [14]. Additionally, Chu et al., 2009 conducted a study in the vicinity of Tin mine in Dai Tu district, Thai Nguyen province and the results showed that the average levels of As, Cd, Cu, Ni, Pb, and Zn in paddy soil were 52.5; 1.18; 179; 6.10; 50.7; and 84.3 mg/kg, respectively [4]. Both of the studies had a much higher concentration compared to the Vietnam standard for agricultural soil. In this study, the heavy metal level in agricultural soil was lower compared to other studies, and only As level exceed the Vietnam standard but not too much. This indicated that agricultural soil sample quality was not at risk for cultivation and further purposes. The heavy metal concentrations were not in any trend according to the distance from the mining sites, this could be the consequence of different behavior in using pesticide or fertilizer for the cultivation. Cd, Ni, Pb, Zn, and As were reported to be one of the pesticide or fertilizer components [7, 15] and when applying them for cultivation, these elements could be released to the soil and uptake by plant.

3.3. Heavy metals concentration in grain rice samples

After grain rice samples were collected, they were separated into husk and rice and the analysis results of rice were shown in Table 5 and Figure 3. In Figure 3, the sampling sites were arranged according to the distance from the mining sites, the sampling sites which were near the mining sites the most were in red squares.

Table 5. *Heavy metals level in rice samples (mg/kg)*

Sample ID	*Ni*	*Cu*	*Zn*	*As*	*Cd*	*Pb*
R2	0.13	4.80	15.9	0.20	0.01	0.09
R3	0.13	4.04	15.1	0.23	0.04	0.06
R4	0.23	4.76	13.8	0.35	0.06	0.10
R6	0.27	21.9	15.3	0.40	0.09	0.11
R7	0.48	11.2	15.6	0.32	0.34	0.08
R8	0.23	9.84	18.6	0.18	0.02	0.08
R12	0.50	171	13.9	0.34	0.07	0.12
R13	0.53	6.94	11.2	0.32	0.02	0.06
Min	0.13	4.04	11.2	0.18	0.01	0.06
Max	0.53	171	18.6	0.40	0.34	0.12
Average	0.31	29.4	14.9	0.29	0.08	0.09
Standard*	-	-	-	**1.00**	**0.40**	**0.20**
CODEX	-	-	-	**0.20**	**0.40**	-

"*": Vietnam standard for milled rice TCVN 11888:2017

"CODEX": General Standard for Contaminants and Toxins in Food and Feed (Codex Stan 193 - 1995), 2015.

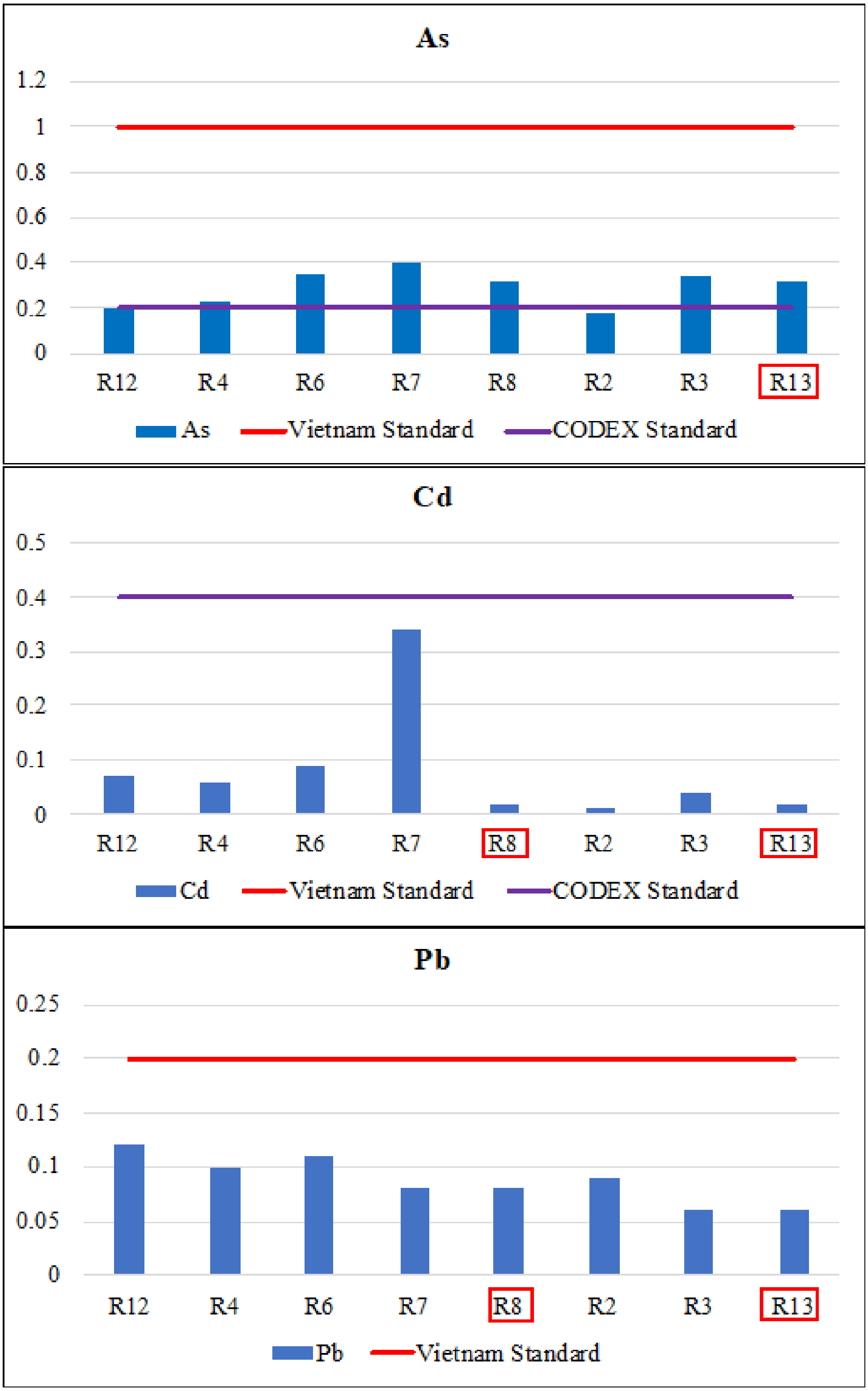

***Figure 3.** Comparison between heavy metal level in rice and Vietnam and CODEX standard for polished rice (mg/kg)*

For the average values, the heavy metal levels in rice follow a decreasing level in order Cu > Zn > Ni > As > Pb > Cd. The levels of As, Cd, and Pb did not exceed the Vietnam standard for milled rice. There was no sample that has the As, Cd, and Pb levels higher than the Vietnam standard for milled rice. This may be due to the low levels of these elements in soil and water. Numbers of research had found a positive correlation between heavy metals level in soil, irrigation water, and grain rice [3, 9, 14].

When comparing the As levels with the CODEX standard for polished rice, there were seven out of eight samples that exceeded the standard. Nevertheless, one thing we have to take into account that the As levels limitation in the Codex standard is for iAs while the As level in this study was tAs. According to General Standard for Contaminants and Toxins in Food and Feed [5], if the tAs level is below the maximum limitation for iAs, no further testing is required and the sample is determined to be compliant with the maximum limitation. If the tAs level is above the maximum limitation for iAs, follow-up testing shall be conducted to determine if the iAs level is above the maximum limit. This difference in regulation due to the different chemical forms of As, considerable differences in As toxicity exist; inorganic forms such as iAs, As (III), and As (V) are toxic to plants, animals, and humans [21]. Although the tAs level did not exceed the Vietnam standard for milled rice, it was higher than the CODEX standard for polished rice. For more assessment and conclusion, this study needs to analyze the iAs in rice. The findings from previous studies have indicated that approximately 50% of As found in grains is iAs [19, 22]. According to that percentage of iAs in rice, the As level in this study might not pose a threat to humans.

4. CONCLUSION

The quality of soil, water, and rice in the vicinity of mining sites in Bac Kan province were reported through this study. Only the level of As in some water and agriculture soil samples are concerned but the levels were not much higher compared to the Vietnam standard. Although the As level in rice samples exceeded the threshold value of the CODEX standard, the inorganic As in rice samples are needed to analyze for further conclusion. This study has indicated the current of water, agricultural soil, and rice in the vicinity of mining sites in Bac Kan province. For further risk assessment, more types, numbers of samples, and studies are required.

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Tóm tắt

Do sự phát triển nhanh chóng trong nhiều ngành công nghiệp như luyện kim, khai khoáng và hóa chất ở Việt Nam, nhiều vấn đề liên quan đến ô nhiễm môi trường đã được đặt ra. Nghiên cứu này được thực hiện nhằm bước đầu điều tra hàm lượng kim loại nặng trong nước, đất nông nghiệp và gạo tại xã Thượng Quan, huyện Ngân Sơn, tỉnh Bắc Kạn, Việt Nam. Kết quả phân tích sáu nguyên tố (As, Cd, Cu, Pb, Ni và Zn) cho thấy hiện trạng chất lượng đất nông nghiệp, nước và gạo vùng phụ cận khu vực nghiên cứu. Hàm lượng trung bình của sáu nguyên tố nghiên cứu trong các mẫu nước mặt và nước sinh hoạt không vượt Quy chuẩn Việt Nam đối với nước mặt và nước sinh hoạt. Trong khi đó, hàm lượng As trong các mẫu đất nông nghiệp nằm trong khoảng 15,6 – 27,2 (mg/kg), cao hơn mức giới hạn của Quy chuẩn Việt Nam cho đất Nông nghiệp. Mặc khác, hàm lượng As tổng số trong các mẫu gạo trắng nằm trong khoảng từ 0,18 - 0,40 (mg/kg), thấp hơn mức giới hạn của Tiêu chuẩn Việt Nam đối với gạo trắng. Qua nghiên cứu này, As là nguyên tố duy nhất cần phải nghiên cứu thêm vì hàm lượng tương đối cao trong các mẫu đất nông nghiệp. Có đến bảy trong tám mẫu gạo có hàm lượng As tổng số cao hơn tiêu chuẩn CODEX cho gạo trắng, As vô cơ cần được tiếp tục phân tích cho những kết luận tiếp theo.

Từ khóa: *ICP-MS, kim loại nặng, nước, đất nông nghiệp, gạo.*