Global J. Environ. Sci. Manage. 7(3): 383-400, Summer 2021

Global Journal of Environmental Science and Management
(GJESM)

Homepage: https://www.gjesm.net/

ORIGINAL RESEARCH PAPER

Natural enrichment of chromium and nickel in the soil surrounds the karst watershed

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ARTICLE INFO

Article History:
Received 18 November 2020
Revised 01 March 2021
Accepted 16 March 2021

Keywords:
Geo-accumulation index
Hazard index
Heavy metals
Pollution load index
Surface soil enrichment
Weathering carbonate rocks

ABSTRACT

BACKGROUND AND OBJECTIVES: As a public concern, monitoring and controlling toxic metals pollution is needed worldwide. Due to the ability of poisonous metals in biomagnification and bioaccumulation, they can cause several adverse impacts on ecological and human health. The study aims to assess chromium and nickel enrichment levels and estimate the soil's ecological risk surrounds the Pangkajene watershed.

METHODS: The total concentrations of chromium and nickel were determined using the Flame Atomic Absorption Spectrophotometer. This study used contamination factor, geo-accumulation index, and pollution load index to evaluate soil enrichment status. The ecological hazard index is used to estimate the potential hazard that may occur due to contamination.

FINDINGS: The mean concentrations of chromium and nickel were 92.9 and 43.18 mg/kg, respectively. Chromium concentration exceeded the soil quality guideline for the protection of environment and human health, while Ni still below the standards. The geo-accumulation index value indicated no human-made-derived contamination in the soil. Weathering of carbonate rocks is the chromium and nickel major enrichment factor in the Pangkep regency. Contamination factor and pollution load index values showed low pollution in the studied soil. However, all study sites exceeded the ecological hazard index value (Ecological hazard index>1), which indicates a considerable ecological risk in the Pangkajene watershed area..

CONCLUSION: These findings may provide baseline information related to chromium and nickel enrichment in the soil for Pangkep regency municipality. The Pangkep regency municipality must highlight the importance of strengthening environmental standards and monitoring mechanism as the priority to maintain a healthy environment.

DOI: 10.22034/gjesm.2021.03.05

NUMBER OF REFERENCES	NUMBER OF FIGURES	NUMBER OF TABLES
70	6	5

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Note: Discussion period for this manuscript open until October 1, 2021 on GJESM website at the "Show Article.

INTRODUCTION

Soil heavy metals pollution becomes a public concern worldwide due to wide distribution, high latency, irreversibility, remediation distress, and contamination process complexity (Su *et al.,* 2014). This issue is also related to food security, where heavy metals can accumulate through the food chain and cause several health problems to humans. More than 10 million soil sites were polluted globally, and more than 50% of those areas were contaminated with heavy metals/metalloids (He *et al*., 2015). This situation was impacting on the economic loss worldwide, which estimated at more than 10 billion US$ per year (He *et al*., 2015) and it can be more become serious in developing countries such as Indonesia due to the increase of financial burden. Heavy metals accumulation in the soil may come from its parent rocks (metal-enriched rocks such as serpentine and black shale) and geological activity such as volcanic activities, earthquakes, landslides, debris flows (He *et al*., 2015; Ma *et al., 2019*). Besides, anthropogenic activities such as wastewater irrigation, open dumping/landfill waste, fossil fuel combustion, incineration, coal ash, smelting, mining, industry, industrial by-products, and agriculture practices such as application of fertilizer and pesticides may enrich the soil with a high concentration of heavy metals. The soil's metal contamination can be transported via dry and wet deposition, stormwater, sewage irrigation, improper disposal of solid waste, and fertilizer application, biosolid, application of manures and pesticides. Moreover, broad land use/land cover shifts after the industrial revolution cause a faster peak of runoffs that ease contaminants' transport. Most toxic metals/metalloids such as Cadmium (Cd), Chromium (Cr), Arsenic (As), Nickel (Ni), Mercury (Hg), and Lead (Pb), are the priority substances to control by ATSDR (USDHHS., 2019). The high concentrations of heavy metals in soil can directly impact the bio-system and indirectly affect animal and human health through the food chain. The human can directly expose to soil heavy metals through inhalation and skin direct contact and indirectly through ingestion of food and groundwater contamination. Most heavy metals can be distributed in the human body through the blood to the tissue (Azeh Engwa *et al*., 2019). Long-term exposure to toxic metals was associated with a harmful effect on human health and become apparent only after several years of exposure (Khan *et al.,* 2008; Jaishankar *et al.,* 2014). The heavy metals poisoning on cellular molecules primarily caused by oxidant and antioxidant imbalance (Tchounwou *et al.,* 2012; Azeh Engwa *et al.,* 2019). Moreover, metals/metalloids such as As, Ni, Cd, Cr(VI) are classified as carcinogenic substances. Cr and Ni were known as the micronutrient for plant growth and animal in small concentrations. It becomes toxic substances at a higher concentration. Since the heavy metal accumulation in soil may pose health risks and adverse effects on the terrestrial ecosystem, the toxicity quantification of contaminated soil can be evaluated using several indices such as geo-accumulation index (I-geo), contamination factor (Cf), pollution load index (PLI), and ecological hazard quotient (EHQ). The characterization of contamination levels and ecological risk is essential to develop specific actions to reduce metal contamination hazards in the terrestrial ecosystem. This present study was conducted in Pangkajene dan kepulauan (Pangkep) regency. This regency is a part of the Maros-Pangkep karst forest area. Like other karst areas, this area is also vulnerable to environmental degradation due to anthropogenic activities such as limestone mining and cement industry and land use shifting (Duli *et al.,* 2019; UGM., 2016). A prior study from Mallongi., (2020) showed Hg contamination and low-level ecological risk in soil surrounds industrial area of Pangkep regency. Nonetheless, a research related to soil heavy metal contamination in the Pangkep Regency is very limited. Therefore, this study aims to assess other heavy metals such as Cr and Ni concentration in the surface soil samples; to characterize soil toxicity and to estimate ecological risk of Cr and Ni surrounding the Pangkajene watershed using several geochemical indices. This study has been carried out in Pangkajene dan Kepulauan (Pangkep) Regency, south of Sulawesi Province, Indonesia in 2020.

MATERIALS AND METHODS

Study area

This study was conducted in Pangkajene dan Kepulauan (Pangkep) regency, South Sulawesi, Indonesia. Pangkep regency located in the south of Barru regency, South Sulawesi Province (110° longitude and 4°.40' – 8°.00' latitude). The total area of Pangkep regency includes 898.29 km^2 of terrestrial area, and 11.464 km^2 of sea. The climate characteristic in the Pangkep regency is tropical monsoon climate with average annual rainfall

and temperature of 2500 – 3000 mm/year and 26.4°C, respectively. This area is dominated by plain and karst hills which have an elevation ranging from 100 – 1000 meters. Maros and Pangkep Regency, located in South Sulawesi Province, Indonesia, have the most prominent and beautiful Karst area worldwide. The karst area, which served to maintain the regional ecosystem's balance, was vulnerable to progressive environmental degradation due to human activities. Besides, it has thin regolith, high porosity, a low carrying capacity of heavy metals, and flexible transport of heavy metals (Zhang *et al.,* 2019). This studied area was enriched by mining materials including limestone, clay/loam, silica sand/quartz, gravel, marble, alluvial gold, chert, feldspar, kaolin, basalt, slate, coal, trachea, propylite, diorite, sandstone, and radioactive material. Marble and limestone mining are the most extensive mining sector in the studied area. In addition, land-use changes and conversion of agricultural land to non-agricultural land have been continually degraded the watershed ecosystem in the Pangkep regency. This condition is potentially enhanced the heavy metals contamination process in soil.

Soil sampling

The field sampling was done in April 2020 which is on rain season. Soil samples were obtained from an area near the Pangkajene river in Pangkajene dan Kepulauan (Pangkep) regency, Indonesia. The selected sampling area includes three sub-districts of Pangkep regency (Bungoro, Minasatene, and Pangkajene), representing watershed areas from upstream to the coast. The study site boundary was less than 5 km from the Pangkajene river. The Pangkajene river is used as irrigation water for farmland and aquaculture. The water of the Pangkajene river was used as the irrigation to the farmland. The study area includes 22 sampling sites with different land use, including; 1) agriculture soil (with 13 sampling sites) and 2) non-agriculture soil (9 sampling sites) (Fig. 1). Seven samples were taken from the upstream area (S01-S05 and S08 -S09), eight samples from the middle stream area (S06 – S07, S10, S14-S18), and eight samples were taken from the downstream region of the Pangkajene river (S11-S13, S19-S22), Pangkep regency, Indonesia. A GPS was used (Garmin 62s) to locate the sampling point during field sampling. Composite soil samples

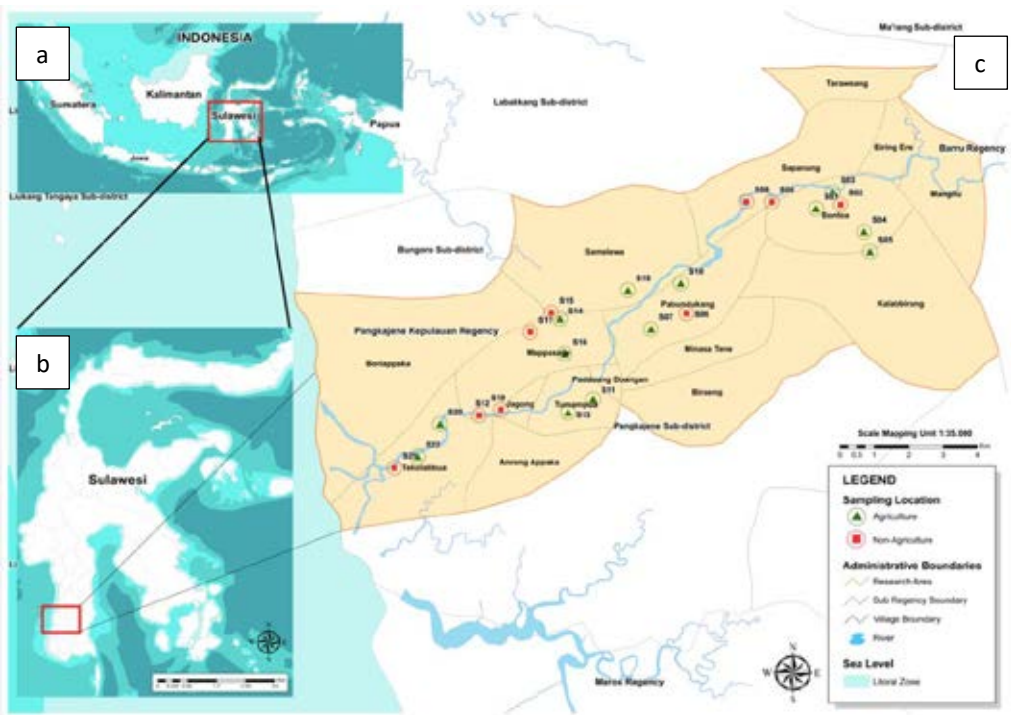


Fig. 1: Geographic location of the study area; (a) Indonesia, (b) Sulawesi Island, (c) Pangkep Regency in Indonesia

Table 1: Analysis method for total Cr and Ni concentration in the studied soil

No	Parameter	Analysis Method	Instrument
1.	Cr	EPA Method 3050b	F-AAS (PerkinElmer PinAAcle 900H)
2.	Ni	SNI 06-6992.6-2004	F-AAS (PerkinElmer PinAAcle 900H)

were collected from the depth 0 – 20 cm of surface soil using a shovel. There are ten individual soils, then they mixed. The interval between an individual sample was 200 meters in an irregular pattern. A 500 gram of soil was labeled and collected into clean zipped polyethene bags after cleaned from rubbish, gravel, grasses, and plant roots. All soil samples were placed at room temperature before brought to the laboratory. Samples were analyzed at the center for plantation-based industry laboratory in Makassar, South Sulawesi Province, Indonesia**.**

Cr and Ni analysis

The soil samples were dried at room temperature. Then, air-dried samples were crushed, sieved through a 2 mm Nylon sieve, homogenized, and placed in the polyethene bottle before acid digestion. The total metal concentration was determined following the analysis method (Table 1). Cr and Ni were analysed using the acid digestion method. To analyse Ni concentration, put 3 g of dried soil samples into Erlenmeyer glass. Added the 25 mL of distilled water and stirred the solution. Then added 5 mL of HNO_3 and three boiling chips into the Erlenmeyer glass. Then, heated the solution at 105^0C until the mixtures reached ±10 mL. Then, cooled at room temperature, added 5 mL of nitric acid and 1 mL of perchlorate acid to the solution, and heated until it was transparent, and filtered the mixtures using filter paper Whatman no 42. The final mixes were measured using a flame atomic absorption spectrophotometer (F-AAS, PerkinElmer PinAAcle 900H). For Cr concentration, acid digestion was done by adding 10 mL HCl to 3 grams of soil sample. Then heat the mixture at 95^0C for 15 minutes and filter the digested sample through Whatman no 42 and collect in a 100 mL volumetric flask. Total concentration of Cr and Ni was analysed using F-AAS with detection limit 0.2 and 0.2 mg/kg, respectively. The quality assurance and quality control of analysis included standard operating procedures and the NIST standard material (NIST 1646a estuarine sediment), all were measured attentively.

Metals Contamination assessment

Geo-accumulation index (I-geo)

The I-geo value was determined using Eq. 1 (Müller, 1986).

$$I_{geo} = log_2\left(\frac{C_x}{1.5C_b}\right) \quad (1)$$

Where, C_x is soil-specific heavy metals in the studied area, C_b is the geochemical/background concentration of specific heavy metals. Due to lack of local background concentration, a background concentration was used from Taylor (1964). The constant of 1.5 accounted to reduce the variation of background concentration which may be influenced by lithologic variation (Laniyan and Adewumi, 2020). The I-geo value interpretation including I-geo £0 means that studied soil is unpolluted with metals; 0< I-geo £1 indicates that the studied soil is uncontaminated up to moderately polluted with metals; 1<I-geo£2 means moderately polluted; 2 < I-geo £ 3 presents that somewhat up to highly contaminated; 3 < I-geo £ 4 indicates highly polluted; 4 < I-geo £ 5 means highly up to extremely polluted; I-geo > 5 demonstrates that the studied soils are significantly/extremely contaminated with metals.

Contamination factor (C_f)

To assess heavy metals contamination in the soil effectively, several pollution indices were used. Two methods were used, including C_f, and PLI to determine the soil heavy metal pollution. Contamination factor (C_f) is widely used to determine toxic substances pollution and soil/sediment quality (Kowalska *et al.*, 2018). The pollution index can be calculated using Eq. 2 (Hakanson, 1980):

$$C_f = \frac{C_x}{C_b} \quad (2)$$

C_f is the contamination factor for measured heavy metals. The pollution load index (PLI) is used

Table 2: The heavy metals concentration in soil at the Pangkajene watershed area (mg/kg)

Land use	Samples	HMs	Mean±SD	Min	Max	Background value*	Permissible value
Agriculture land	13	Ni	35.38±17.1	10	60	75	35
		Cr	89.9 ± 16.8	68.9	117.6	100	100
Non-agriculture land	9	Ni	54.4 ± 20.6	20	70	75	35
		Cr	97.18 ± 21.6	60.6	126.6	100	100
Overall	22	Ni	43.18 ± 20.56	10	70	75	35
		Cr	92.9 ±18.82	60.6	126.6	100	100

*Background value was obtained from continental crust background value in the study by Taylor (1964)
**Permissible value of heavy metals in soil was obtained from WHO permissible value for heavy metals (WHO, 1996)

to determine the severity level of multiple heavy metals' pollution in soils using Eq. 3 (Gati *et al.,* 2016; Kowalska *et al.,* 2018).

$$PLI = \sqrt[n]{CF1 \times CF2 \times CF3 \times ..CFn} \quad (3)$$

Where, n is the number of trace elements.

Ecological hazard quotient (EHQ)

The potential ecological risk was determined using a quantitative screening Ecological Hazard Quotient (EHQ). This index can assess the likelihood that the adverse environmental effects may exist due to toxic substance exposure. This index was used to evaluate adverse biological effects because of heavy metals contamination in the soil (Feng *et al.,* 2011; Mallongi *et al.,* 2014). The specific potential for toxicity in study area soils was estimated by calculating the hazard quotient using Eq. 4.

$$EHQ = \frac{EEC}{screening\ benchmark} \quad (4)$$

Where, EEC is the Estimated or maximum soil heavy metals concentration at sampling sites. The screening-level benchmark is the soil concentration below which toxicity is not likely to occur (Beyer and Sample, 2017). The screening value was obtained from the toxicity value for terrestrial plant by Efroymson *et al.,* (1997). The ecological hazard index is used to determine potential detrimental effects caused by multiple toxic substances (OHIO EPA, 2008). This index is calculated by summing of EHQ.

Data analysis

Cr and Ni concentrations in the soil were collected after analysis in the laboratory. Then, the statistical analysis was conducted using SPSS 24.0 version package. Descriptive analysis (mean, standard deviation, minimum and maximum value) was presented in this study. The IDW interpolation was used to map distribution of Cr and Ni concentration at whole study area.

RESULTS AND DISCUSSION

Distribution of Cr and Ni concentration in the studied area

Table 2 presented the concentration of heavy metals in the studied soil. The mean concentrations showed that Cr was higher than Ni. The Cr concentration was below the soil permissible value. Although Cr was below the standard, it can be accumulated in the soil over time. Ni concentration exceeded the allowable value. In Table 3 demonstrated that Cr concentration in present study exceeded the threshold limit value by CCME (2007). While Ni concentration was below threshold limit value by CCME (2007). Cr concentration in Pangkep regency was higher than Cr world average concentration in soil. While Ni concentration is still below the world average concentration. Compared to other study in karst area, soil in Pangkep watershed has lower concentration of Cr than soil in Huixian karst watershed, China (Huang *et al.,* 2020). Whereas, the concentration of Ni is higher in the Pangkep regency than in the Huixian karst watershed, China.

Table 3: Comparison of mean concentration in studied soils to other soil standards and studies (mg/kg)

HMs	Mean[a]	Earth crust[b]	Average shale[c]	Average surface rocks[d]	Threshold limit value[e]	World average soil[f]	Huixian, China[g]	Beke-Cave watershed, Hungary[h]
Cr	92.9	100	90	71	64	70	118.18	-
Ni	43.18	75	68	49	50	50	43.04	37.47

[a] present study, [b] Taylor (1964), [c]Turekian and Wedepohl (1961), [d,f]Martin and Whitfield (1983), [e]CCME (2007) [g]Huang *et al.,* (2020), [h]Kaszala and Barany-Kevei (2015)

Based on the Table 3, soil in present study has higher concentration of Ni than soil in the karst watershed Hungary. The soil texture can influence the total concentration of metal in soil due to the ability to absorb and mobilization of metals (Kaszala and Barany-kevei, 2015). The highest concentration of Ni is available in clay soil (Kaszala and Barany-kevei, 2015). Both Cr and Ni concentrations were higher in the non-agricultural land than in the agricultural area. Efe (2014) mentioned that soil content depends on its parent rock material and climate. Based on litho-tectonic, the Pangkep regency was classified in the west mandala structure dominating sedimentary rocks such as alluvial deposits, limestone Tonasa formation, Malawa formation, and volcanic rock (Camba volcanoes) (Sompotan, 2012). Another study by Ramli *et al.,* (2009) mentioned that Pangkep regency was developed by alluvium/ sediment material, limestone, breccias, lava, tuffs, conglomerates, ultra-basalt, basalt, trachyte, and mixed rocks. Based on Fig. 2 the highest concentration of Cr is located in S08 from Tonasa formation which has formed by limestone. Sampling site 8 is located near limestone mining and cement industry, the high concentration may be influenced by deposition of dust which is emitted from industry. While the highest concentration of Ni is located in S12, S15 and S19 formed by alluvium sediment and limestone from Tonasa formation. Carbonate rocks and alluvium deposits were dominated the soil parent material in the Pangkep regency. Heavy metal enrichment in the karst area due to the high background concentration of heavy metals in carbonate rocks and secondary enrichment was undergone when the weathering process (Tang *et al.,* 2020). Carbonate rocks may influence the high concentration of Cr and Ni in Pangkep. It has several characteristics, including poor drainage and leaching; thus, it can keep trace elements (Yolcubal and Akyol, 2007; Hasan *et al.,* 2020). Limestone is the metal stabilization agent to inhibit metal mobility by increasing pH. In contrast, when the soil has high alkaline properties, it can be enhanced metal leaching at high concentration (Yun and Yu, 2015). High rainfall and acid rainfall temporarily reduce the acid buffering capacity in karst soil. The Maros-Pangkep karst area is one of the most beautiful karst areas formed in the late Eocene to middle Miocene (40 million to 15 million years ago). The limestone forming in the Miocene era has a high concentration of $CaCO_3$ (92%) (Efe, 2014). The $CaCO_3$ content in the soil can affect heavy metals concentration where it can inhibit the absorption of heavy metals into plants (He *et al.,* 2020). A previous study by Huang *et al.,* (2016) confirmed an effect of carbonate and phosphate on the metals immobilization in the polluted soil. It is caused by the reduction of free-metal activity or the exchangeable fraction of metals in the soil. CaO content in soil karst is much higher than in non-karst soil (Li *et al.,* 2021). It can be caused by the weathering carbonate rocks and pedogenesis. Li *et al.,* (2021) showed the insoluble residues of carbonate rocks which has iron, manganese, aluminium and other trace element was remained as soil parent material, while major elements (potassium, calcium, sodium, and magnesium) were rinsed and leached by soil solution when carbonate rock dissolution in karst ecosystem.

Naturally, Cr and Ni concentrations in limestone are 10 and 20 ppm, respectively (Adriano, 1986). This study showed the enrichment of Cr and Ni in the soil because the concentration of Cr and Ni in the studied soil was higher than the natural concentration in limestone. Based on the geo-accumulation value, all the sites have I-geo < 0 (Fig. 3). It indicates that Pangkep regency's soil was uncontaminated with Cr and Ni, which derived human-made sources. This result is in line with the study by Miko *et al.,* (2003), which mentioned that soil-derived carbonate rocks

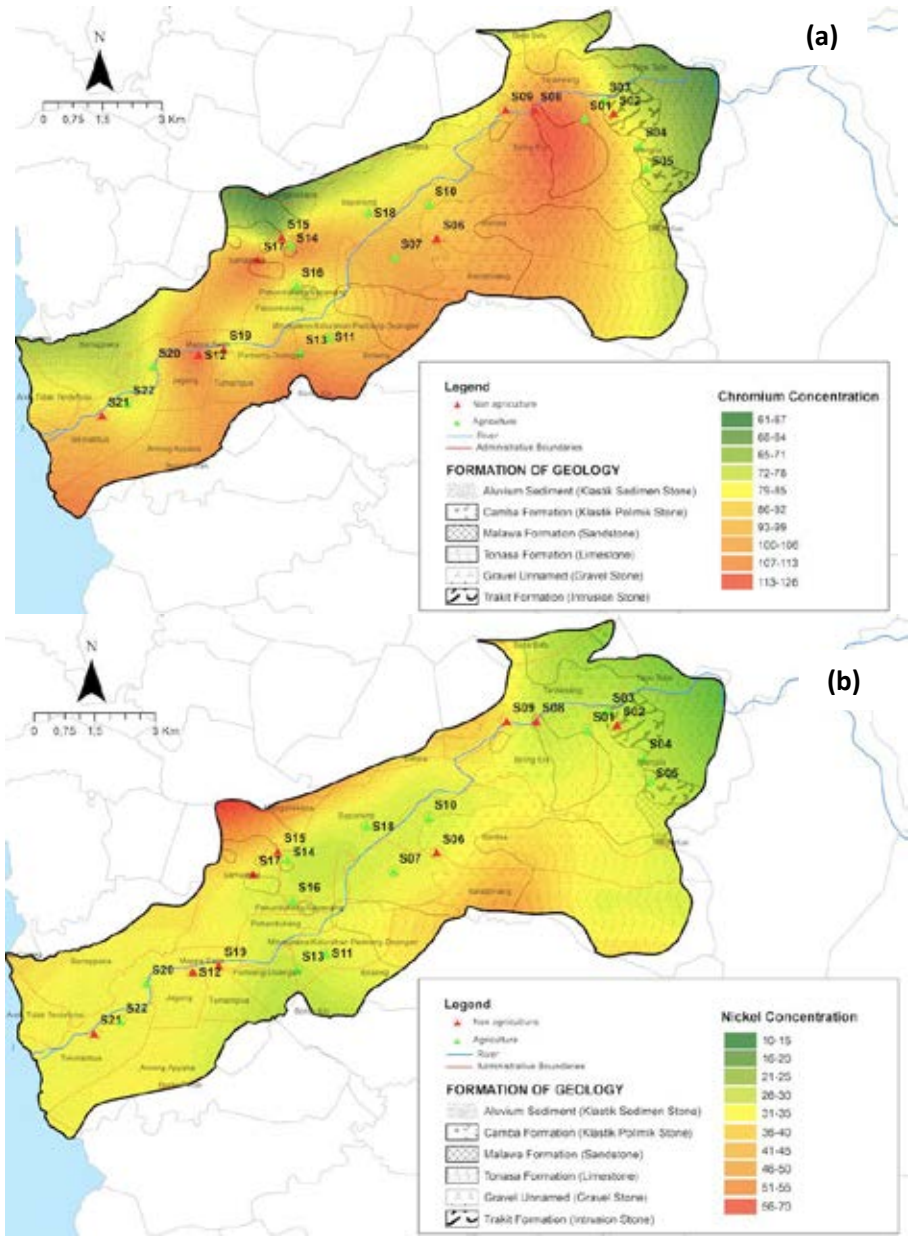
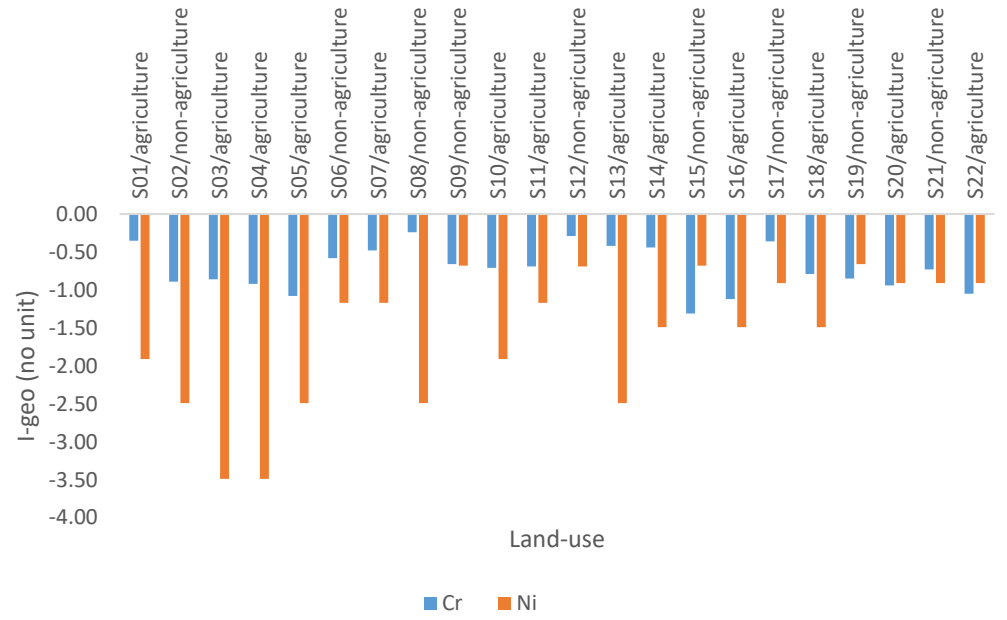
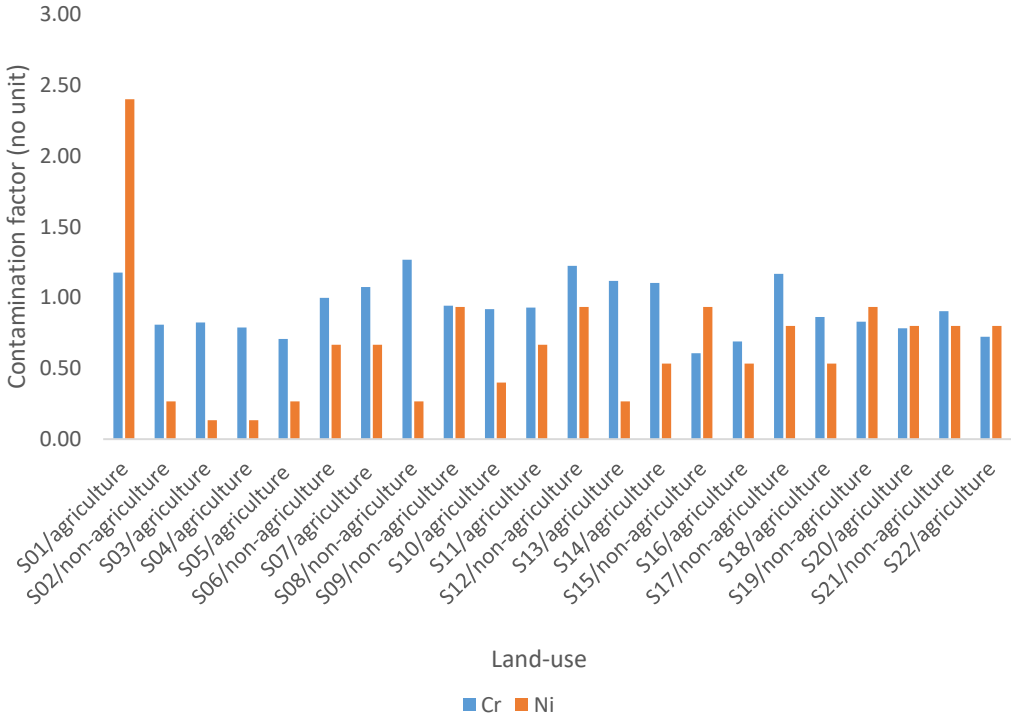


Fig. 2: The distribution of Cr (a) and Ni (b) in the studied soil (mg/kg)

have a higher concentration of Mn, Co, Al, As, Cd, Cr, Ni, Fe, La, Th, V, Cu, and Sr. The high and acid rainfall in the karst area can generate the karst soil loss and progressively enhance the rock weathering process (Kamon *et al.*, 1996; Lyu *et al.*, 2018; Wang *et al.*, 2019; Zhao and Hou, 2019). Moreover, acid rain can cause depleting the surface soil layer, degrade soil nutrients content and soil carrying capacity, and transfer soil metals to other places. Based on NCEANET (2020)'s data, acid precipitation in Maros regency has a pH ranged from 4.3 to 5.6. The main problem of acid rain is possibly caused by air pollution. Similar to the Maros regency, there are mining and industrial activities in the Pangkep regency area, including cement and



(a)



(b)

Fig. 3: Geo-accumulation index (a) and contamination factor (b) of Cr and Ni in the studied area

Table 4: Summary of metals contamination index value in the studied area

Contamination index	HMs	Mean ± SD	Range	Contamination status	Contamination factor classification: $C_f < 1$; $1 \leq C_f < 3$; $3 \leq C_f < 6$; $C_f \geq 6$, representing low, moderate, considerable, and very high pollution (Hakanson, 1980). PLI ≤1, PLI > 1 demonstrating no metals contamination and there is metal contamination, respectively.
Contamination factor	Ni	0.7 ± 0.5	0.1 – 2.4	Low	
	Cr	0.9 ± 0.2	0.6 – 1.3	Low	
Pollution load index		0.7 ± 0.3	0.3 – 1.7	No pollution	

marble. The cement industry contributed 5% of total global anthropogenic CO_2 emission (Mahasenan *et al.*, 2003) and enormous quantities of acid rain precursor such as SO_2, NO_x, CO, and PM to ambient air (Lian *et al.*, 2019). On the lowlands of Pangkep regency, there are alluvial deposits, swamp, and the coast, which were Holocene aged, containing gravel, sand, loam, limestone, coral, and mud (Sompotan, 2012). Based on Fig. 2, Cr's highest concentration is located in non-agriculture land (sampling sites 8). At the same time, high Ni concentration is situated in non-agricultural land (sampling sites 9, 12, and 15). Both high concentration of Cr and Ni were located in non-agricultural land. Based on prior study by Sayadi and Rezaei (2014) high concentration of Ni and Cr may originate from residential-road soil and dairy farm land. High enrichment factor is also occur in the residential and industrial area (Sayadi and Sayyed, 2011). Another study showed that highly concentrated of toxic metals in soil located in areas where people live (Laniyan and Adewumi, 2020). The potential source of Cr are mining, pharmaceutical, metal, textile and leather industries. While, nickel is originated from batteries, power plants or incinerator, combustion of fossil fuels, rubber and plastic industry, electroplating, petroleum by-products and rocks natural weathering. Rauf *et al.*, (2020) and Mallongi *et al.*, (2020) mentioned that the area near cement industry has high concentration of Cr and Hg. The coal combustion is one of the source of metal contamination in soil. In addition to lithogenic sources, human-induced contamination such as domestic, mining, and industry sewages are potentially increased the Cr and Ni concentrations in the soil. Sampling sites 8 and 9 are the residential soil near the cement industry in the Bungoro sub-district. Thus, domestic and industrial sewage is the potential source of pollutants in these sites. Pb, Cr, Ni, and Mn are generally found in areas located near industrial sites, and they potentially pose residents at risk of detrimental health effects. Trivalent chromium is essential for human nutrient, while hexavalent chromium has carcinogenic effects (Jaishankar *et al.*, 2014; Costa and Klein, 2006). Human exposure to chromium is related to skin cancer and lung cancer. A study from McDermott *et al.*, 2014 demonstrated that a median Cr and Ni concentration of 19.13 mg/kg and 4.58 mg/kg in soil is related to low birth weight cases in pregnant women located near industrial sites in the South Carolina USA. It implicates exposure to contaminated soil metals may cause adverse impact to pregnant women living in the near of industrial contaminated sites. Contamination of Cr also effects on gastrointestinal disease (Costa and Klein, 2006). Human may expose to Cr and Ni through soil contact or through eating contaminated food. Furthermore, leaching of metals to drinking water is also possible source of Cr and Ni. Nickel is also classified as human carcinogenic substance by International Agency for Research on Cancer (Das *et al.*, 2019). Chronic exposure to nickel via soil, water or direct contact may induce allergic dermatitis (Das *et al.*, 2019).

Cr and Ni enrichment status

Based on the I-geo value (I-geo<0) in Fig. 3a, soil in our studied area was naturally enriched by Cr and Ni. Cr and Ni's concentration in the studied soil was lower than the soil's background value. It indicates that lithogenic activities in the studied area caused increased Cr and Ni concentrations. Based on Table 4, the studied soils were low contaminated with Cr and Ni (mean value of Cf<1). The result of Cf showed that the soils in Pangkep regency ranged from low to moderately contaminated with Cr and Ni. PLI mean value showed that Pangkep's soils are uncontaminated with Cr and Ni. However, ranged of pollution load index of soil indicated no pollution to moderate pollution. Based on Fig. 3b, contamination factor (Cf) at the studied area was relatively low at agriculture land for nickel contamination, except in sampling site 1 (S01) which had the highest value Cf for Ni. Geologically, sampling site 1 located in the

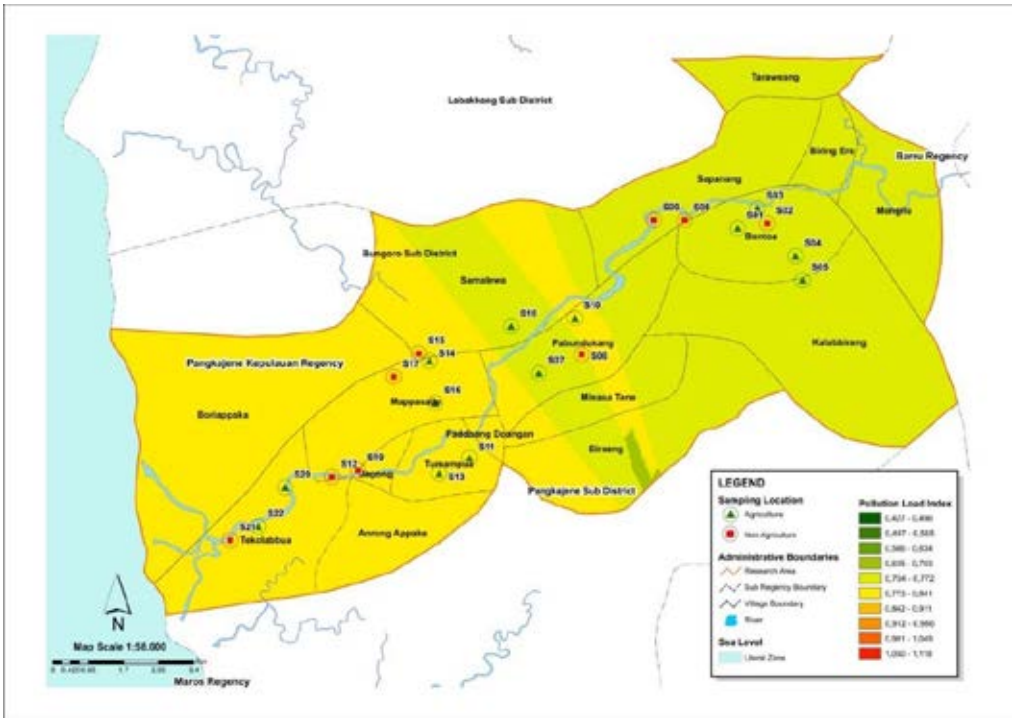


Fig. 4: Pollution load index in the studied soil

Bungoro sub-district was formed by the combination of trachyte formation, Tonasa formation, and gravel stone. Enrichment of metals and minerals little can be influenced by ultramafic and basalt rocks in Pangkep regency (Fatinaware *et al.*, (2019); Suryani and Ritung, 2018; Tonggiroh, 2013; Wilson, 1996). Weathering ultramafic and basalt rocks can cause the high accumulation of Cr and Ni in the soil (Suryani and Ritung, 2018; Sayadi and Sayyed, 2011). Compared to Barru regency in study by Suryani and Ritung, 2018, the total concentration of Cr and Ni in Pangkep regency is far below the total concentration of Cr and Ni in Barru regency. The sampling sites 1 – 9 were used for the mining activities such as limestone, clay, trachyte, feldspar, sandstone, and marble. In addition, there is a cement industry that may influence the contamination of nickel and chromium. According to other study, the soil near cement industry may contaminate with high level of metals such as Pb, Cr, Cd, Ni, Hg, Zn, Cu, Co from aerial deposition (Laniyan and Adewumi, 2020; Mallongi *et al., 2020;* Ogunkunle and Fatoba, 2014; Rauf *et al.,* 2020). The distribution of metals in top soil can be influenced by the wind velocity and particle size (Ogunkunle and Fatoba, 2014). Cr and Ni mean concentration in present study was higher than concentration of Cr and Ni in study conducted by Laniyan and Adewumi, (2020). Furthermore, the high value of Cf in agriculture can be influenced by intensive tillage, and application of fertilizers and pesticides. Higher PLI values are distributed in the middle and downstream area (Fig. 4). An alluvial deposit forms the soil in the middle and downstream. Alluvial and alluvial-colluvial areas are commonly considered to have high Cr content, especially in the calcareous environment (Li *et al.,* 2009).

The middle and downstream areas of the Pangkep regency are dominated by domestic/municipal, agriculture and aquaculture activities. The studied area landscapes are dominated by karst hills in the upstream, gently sloping in the middle stream and alluvial plain in the downstream area. Bungoro and Pangkajene sub-district were classified as the critical land in Pangkep regency, and it is vulnerable to undergo worsening environmental degradation. Soil erosion can transfer metals to further sources. When

Table 5: Degree of ecological risk in soil surrounds the watershed area of Pangkajene regency, Indonesia

HMs	EHQ value	Description	Number of samples	Percentage of samples
Cr	< 0.1	No hazard	-	-
	0.1 - 1.0	Low hazard	-	-
	1.1 – 10	Moderate hazard	-	-
	>10	High hazard	22	100
Ni	< 0.1	No hazard	-	-
	0.1 - 1.0	Low hazard	8	36.4
	1.1 – 10	Moderate hazard	14	63.6
	>10	High hazard	-	-
Total			22	100

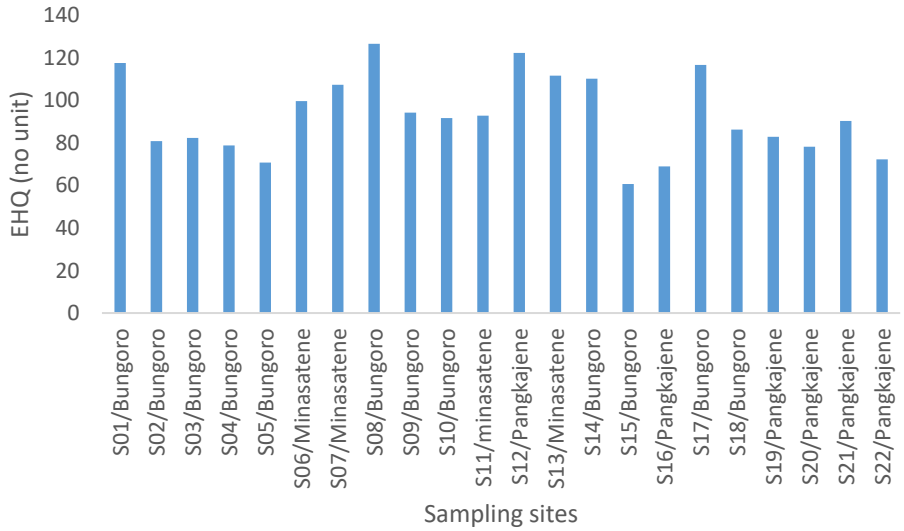
The classification of hazard based on the prior study by (Lemly, 1996)
The screening benchmark for Cr and Ni was 1 and 30 mg/kg (Efroymson *et al*., 1997)

the soil has been disturbed by human activities, including mining, industry, and agriculture, fresh deposits of heavy metals will be transported from higher elevations via the natural process of erosion. It may be remobilized together with excavated soil/ sediments (Jarsjö *et al.,* 2017). Besides, soil erosion is related to the physical disturbance from mining activities and infrastructure (roads) and water use. As Mortatti and Probst, (2010) mentioned, transport of particulate heavy metals could be instantaneously discharged from the source and influence the watershed drainage area. It is expressed when the downstream area has a relatively high concentration of Zn, Cr, Ni, and Cu (Mortatti and Probst, 2010). Agricultural land can transport heavy metals to non-agricultural land by eroded soil. Mohammadi *et al.,* (2019) mentioned soil erosion in farmland could transport heavy metals to the rivers.

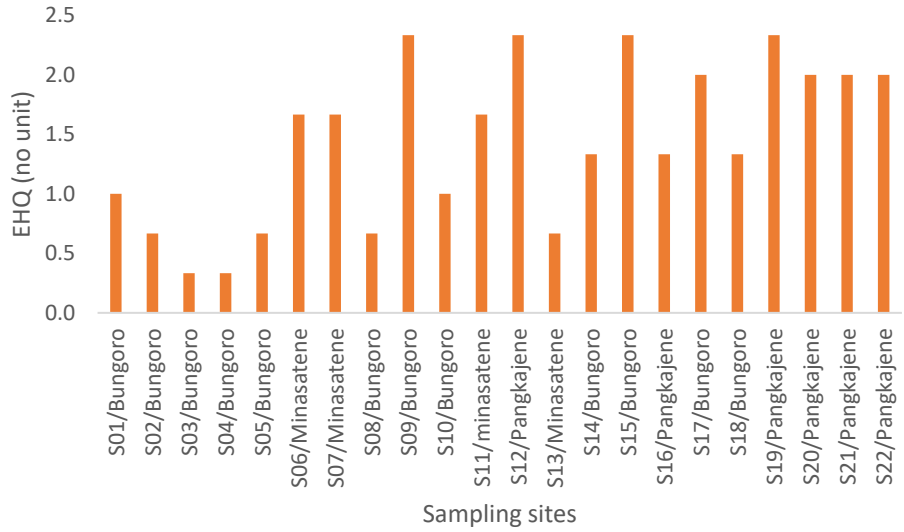
The agricultural sectors may increase heavy metals content in the soil by applying fertilizers, manures and pesticides, and wastewater irrigation. Phosphate fertilizers are the source of heavy metals (Mendes *et al*., 2006). Since Pangkep regency still used urea, SP36, and ZA as fertilizers in the farmland, it could be attributed to an accumulation of heavy metals. The soil heavy metal concentration will be influenced by many factors such as pH, soil type, land use pattern, relief of topographic and lithology aspects. Thus, it might affect the various level of content-heavy metals in this study area. The present study only analysed the total concentration of Cr and Ni in soils. Further research should be undertaken to investigate the several factors that may influence metals binding in the soil of the Pangkep regency.

The potential ecological risk

The US EPA used EHQ calculation to estimate if the risk of adverse effects to the environment is likely or not happened due to the pollutant (USEPA, 2016). Based on Table: 5, all studied soils have a high ecological risk from Cr enrichment. However, Ni enrichment has a low and moderate risk to the ecological system. The ecological risk has a critical value of 1. It means if the value of EHQ > 1, the soil contamination has a potential hazard to the environment, or there is a hazard due to pollutants at the site. Fig. 5 showed sampling site 8 in Bungoro sub-district has the highest EHQ value for Cr, while the highest value of EHQ for Ni is located in Bungoro and Pangkajene sub-district (Sampling site 9, 12, 15, and 19). These results showed that Cr's ecological risk tends to be happened in the upstream area, whereas Ni's ecological risk is likely to occur in the middle and downstream area. Soil is often contaminated with metals which are from far place. Soil erosion can attribute to metal transportation (Amorosi, 2012). Fig. 6 shows that the upstream and the middle stream areas have a high risk from Cr and Ni enrichment. Ecological hazard index (EHI) for studied soils is higher than 1, and it means there is a



(a)



(b)

Fig. 5: EHQ value related to the enrichment of Cr (a) and Ni (b) in the studied area

potential adverse impact on ecology due to Cr and Ni contamination. Cr can have beneficial and adverse effects on humans, plants, and animals. Chromium is not an essential substance for plant (Ertani *et al.,* 2017). Cr can accumulate metals on roots. The toxicity of Cr is depended on its speciation. The toxic effects of Cr and Ni on the terrestrial plant are related to the generation of reactive oxygen species (ROS), which impair the plant's metabolism and physiological process (Hassan *et al.,* 2019; Sharma *et al.,* 2020; Ertani *et al.,* 2017). Cr toxicity symptoms on plants including a decrease of germination,

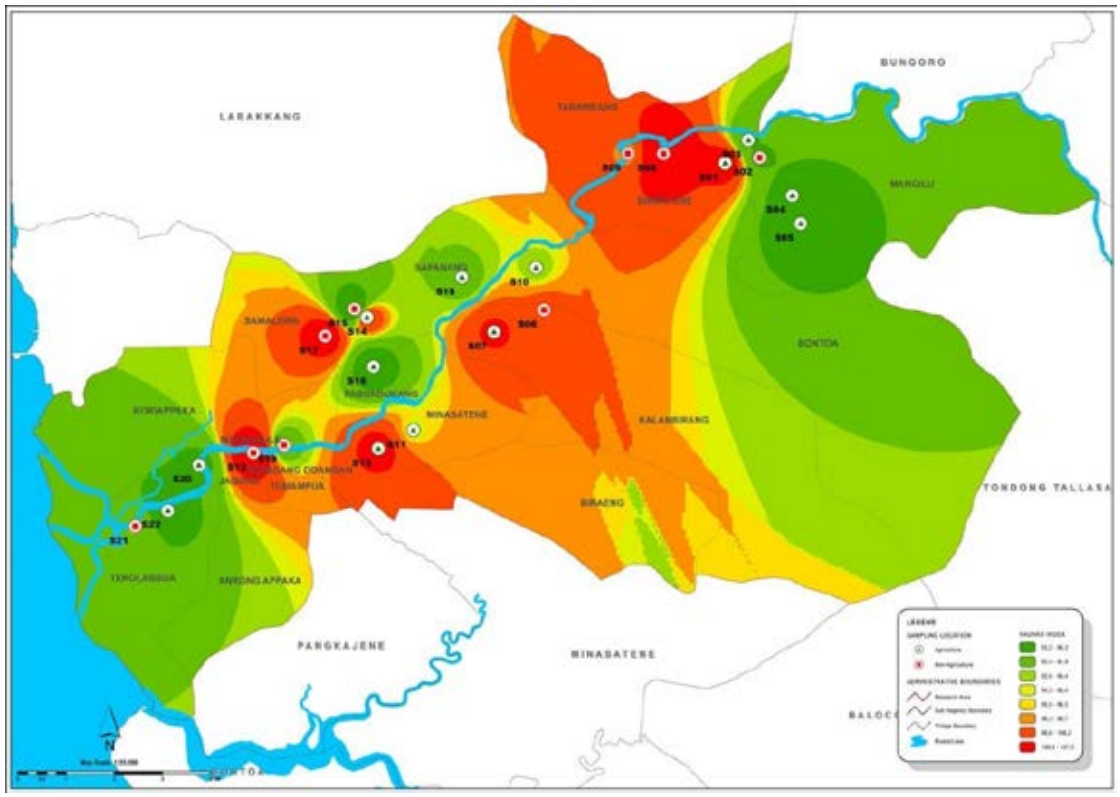


Fig. 6: EHI of Cr and Ni in the studied soil

impairment of enzymatic process, reduction of the growth process, genotoxicity, impairment of photosynthesis, and oxidative shortcoming (Ertani *et al.,* 2017). The higher EHI value is dominated in the upstream area and middle stream area of the watershed, where the mining and industry activities contribute to the accumulation of heavy metals. Besides, in the midstream area, there are municipal/ domestic and agriculture activities. Several home-based enterprises surround the middle stream area of Pangkep watersheds, such as tofu factory, automotive services, machine shops, dry cleaning, metal plating, metal mechanic and metal finishing, and furniture which may possibly discharge their pollutants into soil and water river. The upstream area (S01- S03, S08, and S09) in Bungoro subdistrict is dominated by industry and mining activities.

The cement and marble industry were established for a long time in the Bungoro sub-district. A prior study showed high Hg contamination by industrial and mining activities in the Bungoro subdistrict (Mallongi *et al.*, 2020). Another study in the karst area of Maros regency showed the contamination of Hg and Cr in the area near the cement industry of Maros regency (Rauf *et al.,* 2020). Contamination of soil may be influenced by wind direction and intensive tillage of the agriculture field (Rauf *et al.*, 2020). Study by Rauf *et al*. (2020) also mentioned that Cr tends to accumulate near the cement plant. Thus, limestone mining is one of the contributors to Cr contamination in this area. Since the Cr and Ni tend to be accumulated in the soils and have potentially exposed biota and humans, maintaining and improving the environmental quality is needed in the Pangkep regency. There are several possible actions to reduce Cr and Ni concentration at studied soil including 1) increasing the organic matter content in the soil, 2) applying agricultural lime ($CaCO_3$), and 3) planting the crops that not consumed such as agroforestry (Suryani and Ritung, 2018). The conducting comprehensive research was used on the amount of heavy metals concentration in agriculture products and heavy metals exposure

on human health through the food chain. Since soil erosion has commonly happened in the watershed area, the importance of maintaining and controlling the watershed area relating to the transportation of heavy metals via the erosion process needs to be highlighted by the regional government.

CONCLUSION

The results indicated the soils in the Pangkep regency watershed were generally contain a low level of Cr and Ni. The order of mean concentrations is Cr>Ni. Cr and Ni's highest value is located in the upper and middle stream of the Pangkajene River. This location is dominated by industry, mining, and agriculture activity. I-geo and PLI values indicated no pollution of Cr and Ni in the studied area. The potential sources of Cr and Ni are mainly derived from natural sources. This study area is enriched with carbonate rocks. So, it may influence the immobilization of heavy metals in soil. Soil erosion and flood in the karst area may transport heavy metal from the upstream region to the lowland. These study results also demonstrated that Cr concentration was below WHO permissible value, while Ni concentration exceeded the WHO standard. However, Cr and Ni can accumulate in the soil over time if there is no remediation action at the site. Heavy metal controlling and monitoring activity needs to be done in the studied area due to metals bioaccumulation properties and their adverse impact that may happen in long-term exposure to biota and humans. Increasing the organic matter content in the soil, applying agricultural lime ($CaCO_3$), and developing agroforestry activities are the remediation plan for the studied area. Since lack of standards for heavy metals for soil and information on background concentration in the study area, we recommend to local authorities to develop the standards and environmental monitoring mechanism to reduce the risk of heavy metal accumulation. However, the EHI value >1 means that the metals enrichment has a potential adverse impact on ecological health from Cr and Ni contamination. The importance of landform shifting to transport heavy metals and identifying soil erosion process in the watershed area needs to be concerned in Pangkep regency to control heavy metals contamination better. For further research, there are several suggestions: 1) physical and chemical properties of soil relating to heavy metals contamination, 2) comprehensive study on the amount of heavy metals content in agriculture products, and 3) the effects of heavy metals exposure on human health.

AUTHOR CONTRIBUTIONS

R.D.P. Astuti performed research concepts and designs, assembled data, wrote the article, data analysis, and interpretation. A. Mallongi performed research concept and design and critical revision of the article. A.U. Rauf assisted in collecting data and writing the paper.

ACKNOWLEDGEMENT

This study was supported by the Directorate General of Higher Education Ministry of Education and Culture Republic of Indonesia (DIKTI) [PMDSU program [SK No.6/E/KPT/2019]. Authors are also thankful to Pangkep municipality due to kind cooperation during this research.

CONFLICT OF INTEREST

The authors declare no potential conflict of interest regarding the publication of this work. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and, or falsification, double publication and, or submission, and redundancy have been completely witnessed by the authors.

ABBREVIATIONS

Al	Aluminum
As	Arsenic
ATSDR	Agency for toxic substances and disease registry
$CaCO_3$	Calcium carbonate
C_b	The geochemical/background concentration of specific heavy metals
CCME	Canadian Council of Minister of The Environment
Cd	Cadmium
C_f	Contamination factor
Co	Cobalt
CO	Carbon monoxide
CO_2	Carbon dioxide
Cr	Chromium
Cr (VI)	Hexavalent chromium

Cu	Copper
C_x	soil-specific heavy metals in the studied area
EEC	Estimated environmental concentration
EHI	Ecological hazard index
EHQ	Ecological hazard quotient
EPA	Environmental Protection Agency
Eq.	Equation
F-AAS	Flame atomic absorption spectrophotometer
Fe	Iron
g	Gram
GPS	Global Positioning System
HCl	Hydrochloric acid
Hg	Mercury
HMs	Heavy Metals
HNO_3	Nitric acid
IDW	Inverse distance weighted
I-geo	Geo-accumulation index
La	Lanthanum
max	Maximum
mg/kg	Milligram per kilogram
mg/L	Milligram per litre
min	Minimum
mL	Millilitre
mm	Millimetre
Mn	Manganese
n	The number of trace elements.
Ni	Nickel
NIST	National Institute of Standards and Technology
NOx	Nitrogen oxide
Pangkep regency	Pangkajene dan Kepulauan regency
Pb	Lead
pH	Power of hydrogen or basicity/acidity indicator
PM	Particulate matter
PLI	Pollution load index
ppm	Part per million
ROS	Reactive Oxygen Species
SD	Standard deviation
SNI	Standar Nasional Indonesia or Indonesia's National Standard
SO_2	Sulfur dioxide
SP36	Superphosphate 36
Sr	Strontium
Th	Thorium
US$	United States dollar
V	Vanadium
WHO	World Health Organization
ZA	Diazonium sulfate (Zwavelzure ammoniac)
Zn	Zinc
%	Percent
^oC	Celsius degree

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HOW TO CITE THIS ARTICLE

Astuti, R.D.P.; Mallongi, A.; Rauf, A.U., (2021). Natural enrichment of chromium and nickel in the soil surrounds the karst watershed. Global J. Environ. Sci. Manage., 7(3): 383-400.

DOI: 10.22034/gjesm.2021.03.05

url: https://www.gjesm.net/article_243011.html