



Risk Assessment and Fractionation of Cadmium Contamination in Sediment of Saguling Lake in West Java Indonesia

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Highlights:

- Total Cd content in surface sediment in the rainy season was higher than in the dry season.
- Fractionation of Cd gives information about the source of Cd in the sediment. A high content of resistant fraction indicates a more geological input of Cd.
- The Risk Assessment Code (RAC) values in the dry and the rainy season were 1.70 and 2.64, respectively, which indicates that the surface sediment is in the category of low risk for both the rainy and the dry season.

Abstract. This research focused on the speciation and distribution patterns of cadmium in surface sediment from Saguling Lake, which is located in the Upper Citarum River. Organic compounds and heavy metals from anthropogenic activities in the watershed have contaminated the river. Sample from the upper layer of the sediment from Saguling Lake were taken from 12 locations, representing the dry and the rainy seasons in the period 2015-2018. Sediment cadmium (Cd) classification was conducted through a sequential extraction technique to determine Cd's bioavailability and its risk to the water environment. During the rainy season, the total Cd concentration in the upper layer of the sediment was higher than during the dry season. The average dry and rainy season concentrations were 11.12 ± 2.16 mg/kg and 14.82 ± 1.48 mg/kgm in the sampling locations, distributed differently with the following order of the largest to the smallest concentration: 10B > 1A > 4 > 3 > 2 > 1B > 10A > 7 > 9 > 5 > 6 > 8 for the dry season, and 4 > 1A > 1B > 2 > 7 > 5 > 9 > 3 > 6 > 10A > 8 > 10B for the rainy season. All sampling locations (>60%) showed Cd in the resistant fraction, indicating no significant anthropogenic input of Cd into the surface sediment but more geological input due to high erosion. The values of RAC, ICF, and GFC indicate that the Cd in the surface sediment can be categorized as low risk.

Keywords: *cadmium; risk assessment; Saguling Lake; sediment; speciation,*

1 Introduction

The Upper Citarum River has carried the burden of waste from anthropogenic activities in the watershed for decades. It was once known as one of the most polluted rivers globally. Heavy metals and organic compounds contaminate the river due to poor industrial and domestic wastewater management in the watershed [1-4]. Operated since 1983, the Saguling Dam is located in the headwater of the Upper Citarum River. With the Upper Citarum River Watershed area developing rapidly, the river's pollution burden has increased, negatively affecting the water and sediment quality in Saguling Lake.

Similar to sediment in a natural lake, dam sediment acts as a pollutant sink and source in the aquatic system. In a lake, heavy metals may accumulate in the sediment, resulting in their accumulation in organisms and toxicity. This makes heavy metals a potential risk to human health through the food chain [2,5,6]. Anthropogenic activities that induce heavy metal mobility in water are for example industrial, agricultural and domestic activities, while the natural influx of heavy metals into a lake environment is mainly due to rock weathering and fallout after volcanic eruptions [6-9]. The accumulation of heavy metals in the sediment shows higher toxicity than in the water body, whereas the absorption of suspended solids strongly causes important changes in many aquatic organisms. Sediment contents play an important role due to their components and sink the contaminants at a rate of 99% compared to an aquatic environment. They also provide nutrients for living organisms, are a reservoir of bioavailable trace elements and play an essential role in geochemical cycles. Therefore, lake sediment analysis is important in evaluating contamination strategies [10,11].

Studies have shown that the measurement of total metal concentration in sediments is insufficient to provide information about the exact amount of pollution by heavy metals. The behavior of heavy metals in the environment compartments critically depends on their chemical structure and form, and the binding state (precipitated with primary or secondary minerals complexed by organic ligands). These characteristics influence the bioavailability, mobility, and toxicity of heavy metals to organisms. Their properties depend not only on their total concentration but also on the physicochemical form in which they appear, called 'speciation' [10-12]. Sequential extraction provides information about the primary binding site, as these methods identify and quantify different species, forms, and phases of chemicals present in a material. The sequential extraction technique for geochemical fractionation generates four fractions: F1 = the easily, freely, or leachable and exchangeable (EFLE) fraction; F2 = the acid-reducible

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fraction; F3 = the oxidizable-organic fraction, and F4 = the residual fraction [11-14].

Several indicators have been developed over the past decades to assess heavy metal sources and their bioavailability in surface sediments. Two of these indicators are the global contamination factor (GCF) and the risk assessment code (RAC) [12]. The RAC assesses the ecological risks associated with metals by calculating the percentage of metals present in both the exchangeable and the carbonate fractions [15]; higher mobility of heavy metals poses a higher environmental risk and threatens aquatic biota [16]. A study on Rawa Kalong Lake, Indonesia as an urban lake has shown that Cd content in the water column plays an essential role in the bioaccumulation capability onto microalgae biomass [17]. Total phosphorus is another important physicochemical parameter. The study also found that the average concentration of Cd in the water column was above the threshold limit for safe aquatic life.

Indonesia does not regulate sediment quality; there is no heavy metal threshold limit for sediments. On the other hand, data and studies have revealed that the heavy metals content in river water is higher than the standards for aquatic ecosystems [1,2,17,18]. A previous study using the geochemical background concentration (Cbg) and the GCF method in the water catchment area of the Saguling Dam revealed that it had been polluted by Cd, Cr, Cu, and Pb, with Cd in the very high category [19]. The purpose of the present study was to evaluate the Cd concentration in surface sediment from Saguling Lake by identifying the sources of Cd using the global contamination factor (GCF) and to assess the bioavailability of Cd using the risk assessment code. This paper provides important information about Cd pollution in Saguling Dam surface sediment for sediment quality management in Indonesia.

2 Materials and Methods

2.1 Study Area

The area of Saguling Lake is 4,710 Ha, with a water storage capacity of 730.5 million m³. The primary purpose of the dam is to generate hydroelectricity for Java and Bali islands, and irrigation and raw water for Jakarta City. Saguling Lake is located in a hilly area with many tributaries, rendering the shape of Saguling Lake very irregular or dendritic, with many extended bays. The water in Saguling Lake comes from the Upper Citarum Watershed, a plateau encircled by mountains that form a basin located between 7°19' and 6° 24' south latitude and 106°51' and 107°51' east longitude [20]. Some industries, mainly textile and some metal processing and electroplating industries, are also located within the Upper Citarum Basin.

2.2 Sample Collection and Processing

Samples from the surface sediment were taken from 12 sampling points at Saguling Lake. The locations of the sampling points and their GPS coordinates are presented in Figure 1 and Table 1 respectively.

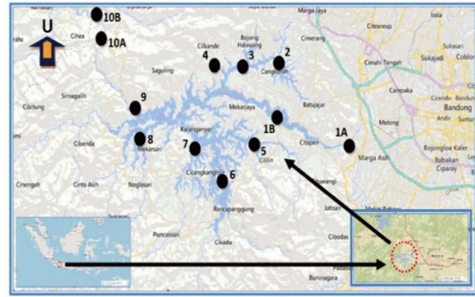


Figure 1 Sampling point locations at Saguling Lake.

Table 1 Sampling locations.

Station	Location	Coordinate	
		South	East
1A	Citarum River Nanjung Section	06°56'29.8"	107°32'10.7"
1B	Citarum River section Trash Boom Batujajar	06°54'58.9"	107°28'35.0"
2	Cijaur Village Cipeundeuy	06°53'13.5"	107°28'32.3"
3	Cimerang	06°53'13.4"	107°27'09.0"
4	Cihaur Estuari Maroko Village	06°54'13.0"	107°27'54.5"
5	Cipatik Estuary	06°56'07.6"	107°27'25.5"
6	Ciminyak Estuary-fishing floating located	06°47'14.6"	107°26'03.8"
7	Cijere Estuary	06°56'14.9"	107°24'50.8"
8	Cijambu Estuary	06°56'00.4"	107°22'22.4"
9	Near Intake Structure	06°54'54.4"	107°22'26.3"
10A	Tailrace	06°51'49.8"	107°20'57.0"
10B	S. Citarum after Tailrace at Bantar Caringin	06°51'10.8"	107°20'58.0"

A portable global positioning system (GPS) was used to determine the sampling locations. The surface sediment sample represented the dry and wet seasons, following the classification of the Meteorology, Climatology, and Geophysics Agency of West Java Province. The rainy season samples were taken in November 2015 and April 2017, while the dry season samples were taken in August 2016 and September 2017. The surface (upper layer) sediment samples were taken from the sediment's top 10 cm at each sampling point. Four to five samples were randomly collected and mixed in one sample. The samples

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collected by using a Grab sampler were placed into polyethylene bags and refrigerated at 4 °C, then dried at 50 °C and subsequently grinded by using pestle and mortar. The samples with all particles that passed a 200-mesh nylon sieve continued to the geochemical fractionation process.

For geochemical fractionation of Cd in the samples, the modified sequential extraction technique (SET) was used [12-14,21]. Fraction 1 (F1) was extracted from the whole sample by shaking 10 g of sample with 50 mL of 1.0 M ammonium acetate ($\text{NH}_4\text{CH}_3\text{COO}$) at pH 7.0 for three hours at room temperature. Fraction 1 (F1) was the supernatant; the residue continued to the next procedure of extracting Fraction 2 (the acid-reducible fraction). Extracting F2 was done by shaking the residue with 50 mL of 0.25 M $\text{NH}_2\text{OH}\cdot\text{HCl}$ -hydroxyl ammonium chloride for three hours, acidified to a pH of 2 with HCl at room temperature. The supernatant was taken as F2. The third fraction was the oxidizable-organic part (F3).

The F2 procedure's residue continued to the oxidizing process using 30% of H_2O_2 in a water bath at a temperature of (90-95) °C. The oxidizing solutions were cooled down and then shaken for 3 hours with 1.0 M ammonium acetate ($\text{NH}_4\text{CH}_3\text{COO}$) to remove the metal from organic complexes. After that, the solution continued to acidification until a pH of 2 by adding HCl at room temperature. Then the supernatant was taken for F3 determination. The last fraction (F4), which is the most resistant fraction of Cd, was separated by digesting the residue from the F3 process in 10 mL concentrated HNO_3 (ratio 4:1).

The residue from all fractionation processes was then washed with 20 mL of double-distilled water (DDW) and filtered through a Whatman no. 1 filter before weighing of each fraction. The supernatant of each fraction was ready for Cd analysis using inductively coupled plasma optical emission spectrometry (ICP-OES). This procedure must be carried out immediately after analyzing Fraction 1 to minimize the loss of volatile elements. In this study, the blank procedure was implemented to ensure no metal contamination was present in the sample and reagent used.

2.3 Calculation

The risk assessment code (RAC) was used to find the potential risk attributable to the presence of individual metals. The assessment was classified based on the percentage of F1 in the total metal and categorized as shown in Table 2 [15,16].

Table 2 Criteria for risk assessment code (RAC) [15,16].

F1 (%)	Class	Risk
< 1	1	No risk
1-10	2	Low risk
11-30	3	Moderate risk
31-50	4	High risk
>50	5	Very high risk

The metal contamination factor indicates the degree of risk from heavy metals to the environment related to their retention time [8,15]. The individual contamination factors (ICF) of each sampling station were calculated using the fractionation data, as shown in Eq. (1) for ICF_{metal} , by dividing the sum of exchangeable, acid-reducible, and oxidizable-organic fractions (F1, F2 and F3) with the residual fraction (F4) for each site. The global contamination factor (GCF) at each sampling point was determined based on the sum of the ICF values at each location, as shown in Eq. (2) [15]. The values of ICF and GCF at each sampling point were categorized as contamination factors, as shown in Table 3.

$$ICF_{metal} = \frac{C_{non\ resistant}}{C_{resistant}} = \frac{C(F_1+F_2+F_3)}{C F_4} \quad (1)$$

$$GCF = \sum_{i=1}^n ICF_{metal} \quad (2)$$

Table 3 Degree of contamination criteria [15].

Category	Degree	Speciation indices	
		ICF	GCF
I	Low	<1	<6
II	Moderate	1-3	6-12
III	Considerable	3-6	12-24
IV	High	>6	>24

3 Results and Discussion

3.1 Distribution of Cadmium in the Sediments

This study found that the total Cd concentration in the surface sediment from Saguling Lake, both in the rainy and the dry season, exceeded the standard based on ANZECC [22]. Total Cd concentration in the rainy season ranged from 11.43 to 16.68 mg/kg, with an average value of 14.82 ± 1.48 mg/kg. A lowest concentration of 11.43 mg/kg was found at sampling point 10B (Citarum River after the tailrace in Bantar Caringin). A highest concentration of 16.68 mg/kg was found at point 4 (Cihaur Estuary Maroko Village). On average, the Cd concentration in the dry season was lower than in the rainy season. The mean Cd concentration in the dry season was 11.12 ± 2.16 mg/kg. Figure 2 presents the Cd

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concentration in the surface sediment in the rainy and dry seasons. The ranking of Cd concentrations (highest to lowest) in the surface sediment was $10B > 1A > 4 > 3 > 2 > 1B > 10A > 7 > 9 > 5 > 6 > 8$ for the dry season, and $4 > 1A > 1B > 2 > 7 > 5 > 9 > 3 > 6 > 10A > 8 > 10B$ for the rainy season. The highest Cd concentration in the dry season was the same as the lowest concentration in the rainy season.

Saguling Lake receives water from the Upper Citarum River. Therefore, the water and surface sediment quality is affected by the watershed condition. Research in the upper Citarum River has shown that land-use changes in some sub watersheds have led to a decrease in the quantity and quality of water resources [19,23,24]. Increased flood frequency and more erosion in the upper watershed result in sedimentation in the river downstream and in Saguling Lake. This research calculated the Upper Citarum River pollution loads based on West Java Province Environmental Protection Agency water quality monitoring data [23] for the rainy and dry seasons. The rainy season load, represented by data from September to October 2018, was 272 kg/day and 12.65 kg/day in the dry season based on data from April to May 2018. The Cd content in the river water is related to several anthropogenic activities, such as industries, deposition of organic and fine grain sediments, leachate from defused Ni-Cd batteries, and Cd based electroplating processes. There are 556 industries in the Upper Citarum Watershed, dominated by textile industries (442), 40 electroplating industries, 25 food and beverage industries, 16 rubber and plastic industries, 14 chemical industries, industries, 11 tanning industries, 8 pulp and paper industries, and metals industries [23].

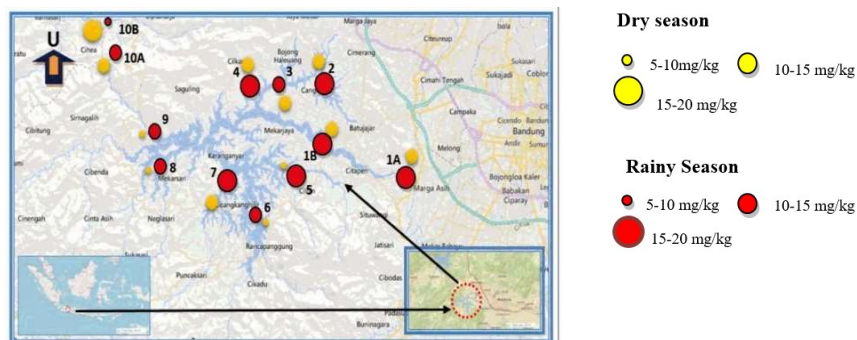


Figure 2 Concentrations of Cd in sediment from Saguling Lake in the rainy and dry seasons.

Compared to data from other countries, the Cd concentration in the surface sediment from Saguling Lake was found to be higher. In Songkhla Lake, Southern Thailand, it ranges from 0.1 to 2.4 mg/kg [25], in Laguna Lake, the Philippines, it

ranges from 0.23-0.33 mg/kg [26], in Manchar lake, Pakistan, it ranges from 4.9-9.7 mg/kg [27], in Taihu Lake, China, it ranges from 0-0.94 mg/kg [28]. Meanwhile, the Cd concentration was (0.1-0.39) mg/kg in sediment from the Bonan Dolok River, Samosir-North Sumatra, Indonesia [18].

3.2 Metal Speciation

Metal speciation in sediments determines its bioavailability to biota. Metals in Fractions 1, 2, and 3 (F1, F2, and F3) are labile metals, whose movements are strongly influenced by environmental conditions. Fraction 1 is the metal with the highest mobility, the most available form, the most exchangeable, and weakly adsorbed into the sediment matrix. Thus, the most easily released and dissolved water column is the most open and potentially absorbed, which can cause toxicity to aquatic organisms [15,29,30].

The Cd speciation in Figure 3 shows that Fraction F4 was dominant in all samples in the rainy and dry seasons. On average, F2, F3, and F4 concentrations were higher in the rainy season than in the dry season, whereas the F1 concentration was higher in the dry season compared to the rainy season. The highest concentration of fraction F1 was found at monitoring point 1A in the rainy and the dry season compared to the other sampling points with similar concentrations of 1.105 mg/kg and 1.135 mg/kg. The highest and lowest concentration of fraction F2 was found in the rainy season at sampling point 6 at 1.95 mg/kg, and at sampling point 10A at 0.58 mg/kg. Sampling station (SS) 5 had the highest concentration of F3 in the rainy season and the lowest concentration of F3 was found at SS 3 in the dry season. SS station 1B showed the highest concentration of F4 in the rainy season with the lowest concentration at sampling point 8 in the dry season.

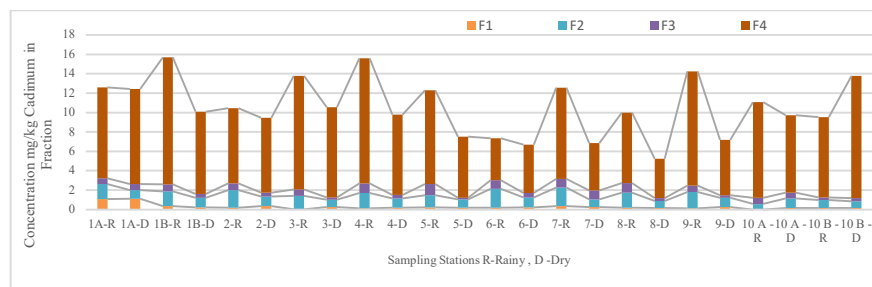


Figure 3 Cd speciation in the rainy and dry seasons.

Overall, the composition of Cd speciation was similar in the rainy and the dry season: F4 > F2 > F3 > F1. Table 4 shows the Cd speciation compared to other countries.

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Table 4 Results of Cd speciation in several countries.

Location	Cd Speciation (mg/kg)	Reference
Jiangsu China River	F1: 7.02 ± 0.17 , F2: 3.99 ± 0.13 F3 0.28 ± 0.01 , and F4 0.14 ± 0.01	Bo <i>et al.</i> , 2015
Ghalechay Iran River	F1: 0.7, F2: 0.0, F3: 1.7, and F4: 15.6	Forghani <i>et al.</i> , 2009
Taihu Lake China	F1: 0.7, F2: 0.1, F3: 1.0, F4: 15.6	Yin <i>et al.</i> , 2011
Skandar Montenegro Lake	F1: 5.50 ± 1.96 - 25.79 ± 3.77 , F2: 4.41 ± 0.47 - 25.81 ± 8.20 , F3: 3.45 ± 0.80 - 14.64 ± 5.66 , and F4: 0.03 ± 0.02 - 5.05 ± 0.02	Kastatrovik <i>et al.</i> , 2016
Saguling Lake Rainy season	F1: 0.26 ± 0.28 F2: 1.47 ± 0.41 , F3: 0.77 ± 0.22 , and F4 ± 2.53 : 9.60	This study
Dry season	F1: 0.30 ± 0.26 , F2: 0.88 ± 0.11 , F3: 0.43 ± 0.20 and F4: 7.50 ± 2.44	

This study showed that the most bioavailable fraction to biota was in a small portion of all Cd content in the surface sediment. The high concentration of F4 was similar to that in Ghalechay River, Iran, and in Taihu Lake, China.

3.3 Risk Assessment Code (RAC)

The Risk Assessment Code values at different sampling points in the rainy and dry seasons are shown in Figure 4. The dry season had higher RAC values than the rainy season. Both the rainy and the dry season are in the low-risk category.

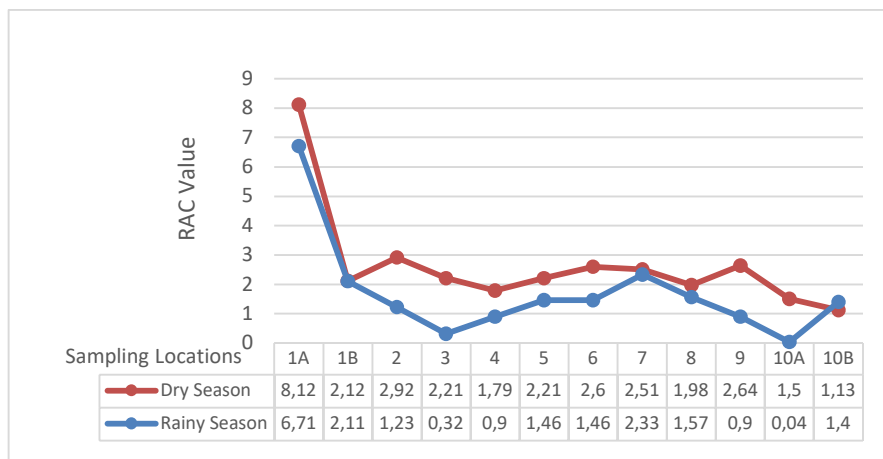


Figure 4 RAC values for Saguling Lake in the rainy and dry seasons.

Sampling point 1A had the highest RAC values in the dry and rainy seasons; this sampling point is the primary inlet of Saguling Lake from the Upper Citarum River. The result indicates that Cd is dominantly coming from the Upper Citarum

River watershed. That result is related to Yin [31] finding in Lake Taihu, China, that the Hg, Cu, Cr, Cd, and Pb might originate from domestic sewage and industrial wastewater. The ecological risk indices showed that the Sediment of Lake Taihu is moderated polluted. Sampling points 1B, 7, and 10B had the same RAC value in the rainy and dry seasons, while the other sampling points showed different values. High RAC values have been found in the China Xiawangang River with a value of 37.77, which is categorized as high risk. The RAC values found in Saguling Lake were similar to those in Chauho Lake, ranging from 4-5 and classified as low risk [33].

3.4 Value of Individual Contamination Factor (ICF) and Global Contamination Factor (GCF)

The ICF and GCF values of the surface sediment from the sampling points at Saguling Lake are presented in Table 5. The ICF for the rainy season ranged from 0.12 to 0.70, with an average value of 0.29. Sampling point 6 had the highest ICF values and sampling point 10A had the lowest values. The GCF value for the rainy season was 3.46. For the dry season, the ICF values ranged from 0.09 to 0.40, with an average value of 0.24. The highest ICF values were found at sampling point 7 and the lowest were found at sampling point 10B. The GCF value for the dry season was 2.82. The result showed that both in the rainy and the dry season, the ICF and GCF values were similar, while the GCF values were higher in the rainy season. All sampling points had a low contamination degree (contamination degree I), and the GCF value also showed contamination degree I.

Table 5 ICF and GCF values for Saguling Lake in the rainy and dry seasons.

Sampling points		1A	1B	2	3	4	5	6	7	8	9	10A	10B	Average	GCF values:
Rainy	ICF	0.34	0.2	0.35	0.18	0.21	0.27	0.7	0.34	0.38	0.21	0.12	0.15	0.29	3.46
Dry	ICF	0.27	0.19	0.23	0.14	0.19	0.19	0.35	0.4	0.3	0.26	0.22	0.09	0.24	2.82

Figure 5 presents the proportion of non-resistant to resistant fractions at each sampling point. Most of the Cd in the sediment was in resistant form, which means it is difficult to release to the water column. This fraction of Cd is firmly bound to crystalline minerals. On the other hand, heavy metals derived from human activities were found in the non-resistant fractions (F1, F2, and F3).

The non-resistant fraction of Cd at each sampling point ranged from (8.73-21.61) % in the rainy season and from (7.66-20.02) % in the dry season. A lower portion was found in the rainy season at sampling points 10A and 10B, which are located at the lake's outlet. In the rainy season, these two outlets may have strong currents and remove the surface sediment, including the Cd content. The highest proportion of non-resistant fraction was found at sampling point 6 for both the

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rainy and the dry season. The non-resistant and resistant Cd fractions may depend on the sampling point's location. Saguling Lake has several inlets from tributaries. The main one is at sampling point 1A with a strong water current from upstream of the Upper Citarum River. The water condition at sampling points 2, 3, 4, 5, 6, 7, 8 and 9 is still and relatively stagnant.

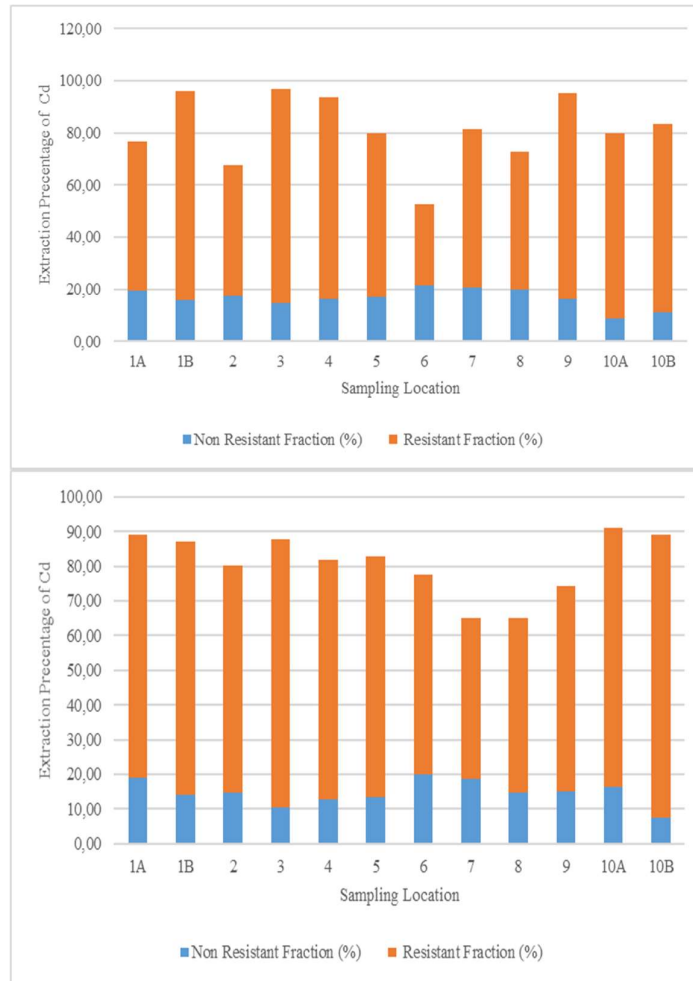


Figure 5 Fraction (%) of resistant and non-resistant Cd for the 12 sampling stations at the Saguling Dam (left: rainy season, right: dry season).

4 Conclusion

This study identified Cd content in the surface sediment from Saguling Lake and its fractionation. Total Cd content in the surface sediment in the rainy season was higher than in the dry season. The average dry and rainy season concentrations were 11.12 ± 2.16 mg/kg and 14.82 ± 1.48 mg/kg, respectively. The overall Cd concentration in the surface sediment exceeded the heavy metal standard based on ANZECC. Total Cd concentration was distributed differently in the sampling locations, with the order of the largest to the following smallest concentrations: $10B > 1A > 4 > 3 > 2 > 1B > 10A > 7 > 9 > 5 > 6 > 8$ for the dry season, and $4 > 1A > 1B > 2 > 7 > 5 > 9 > 3 > 6 > 10A > 8 > 10B$ for the rainy season.

The Risk Assessment Code (RAC) values in the dry and the rainy season were 1.70 and 2.64, respectively, indicating that the surface sediment was in the low-risk category for both the rainy and dry seasons. The average ICF values in the dry and the rainy season were 0.29 and 0.24, respectively, with GCF values of 3.46 in the rainy season and 2.82 in the dry season.

The ICF and GCF values were in the low-contamination category, which means that the surface sediment in Saguling Lake is dominated by the resistant fraction (63.97-64.74%). The high content of the resistant fraction indicates a higher geological input of Cd. The values of contamination risk indicators such as RAC, ICF, and GCF showed that the Cd content in the surface sediment can be categorized as low risk.

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