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SCANNING ELECTRON MICROSCOPY AND MAGNETIC CHARACTERIZATION OF IRON OXIDES IN SUSPENDED SEDIMENTS

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ABSTRACT

River sediment is the product of a sedimentation process in the river environment that originates from the weathering of bedrock or erosion processes, organic materials, particles, or anthropogenic substances. Previous research shows the magnetic mineral content in the sediments of the Citarum River and its tributaries. Suspended sediment was collected from the Citarum River and its tributaries in West Java, Indonesia, for mineralogical and granulometric analysis. The aim of this research is to distinguish between magnetic mineral sources, a scanning electron microscope equipped with energy dispersive X-ray spectroscopy (SEM-EDX), and rock magnetism. The hysteresis parameter verifies that the XRD measurement results show that the main magnetic minerals in suspended sediments are ferrimagnetic minerals such as magnetite (Fe_3O_4). According to the results of the SEM-EDX investigations, the magnetic granules in the Citarum River and its tributaries derive from two distinct sources: pedogenic and anthropogenic. The Citarum River and its tributaries' magnetic granules are generally octahedral or angular in shape, with broken corners, indicating a pedogenic origin.

Keywords: suspended; sediments; Citarum; anthropogenic; lithogenic; tributaries.

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INTRODUCTION

The application of magnetic minerals in environmental studies to interpret pollution in soil, sediment, and vegetation has been carried out since the 1980s [1]. The application of magnetic minerals to study environmental magnetism in river sediments to interpret the presence of pollutants has been widely carried out [2–13], as have studies on soil [14–17], dust [18–20], and leachate [21-22].

The application of magnetic mineral morphology studies, especially iron oxide, has been widely used in environmental magnetic studies [23-24]. Iron oxide is used in this study because iron oxide can experience an increase or depletion due to environmental changes, which can also predict pollutant sources [22]. Previous studies in various environments indicated the presence of different iron oxide morphologies in dust and fly ash due to combustion

processes in various industries and motorized vehicles [25–29]. A different morphology of iron oxide was also found, indicating pollution due to human activities in river sediments [3],[30], marine sediments [14], lake sediment [31], soil [32-33], and leachate [22].

River sediment is the result of a sedimentation process in the river environment that originates from the weathering of bedrock or from erosion processes, organic materials, particles, or anthropogenic compounds (waste resulting from human activities). The problem that arises in river sediment is the presence of materials (substances) that are harmful to the environment (pollutants). The primary source of contaminants in river sediments is human activity, which includes land use activities (both urban and rural) adjacent to rivers, as well as the dumping of industrial, domestic, mining, and agricultural waste [34]. It has previously been proven that magnetic minerals exist in the sediments of the Citarum River and its tributaries [35–37]. Furthermore, if the sediment contains enough magnetic minerals, the magnetic susceptibility is strongly associated with the heavy metal level. However, magnetic minerals in river sediments can be derived from a variety of sources, including local soil and waste. In this study, we use SEM-EDX and magnetic analysis to investigate the origin of magnetic minerals in river sediments and identify the major contributors to the magnetic signature. It is believed that this knowledge would help us better understand how to employ magnetic analysis to identify and quantify pollutants.

METHOD

Figure 1 illustrates the sampling points location of the suspended sediment samples. The suspended sediment samples used in this research came from previous study samples [36]. We selected the samples in this research based on certain magnetic parameter values, including their magnetic susceptibility value. Detailed information on the location, geology, soil type, climate, and sampling methods has been published [36]. Selected samples from the Citarum River and its tributaries were extracted using a magnetic stirrer following procedures elsewhere [37] to determine the morphology, magnetic mineral composition, and determined magnetic mineral domains of the samples.

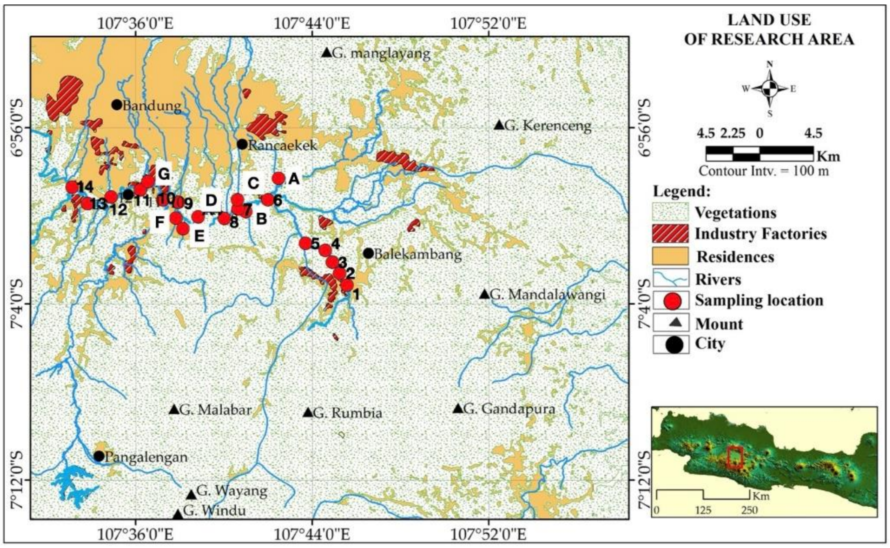

Figure 1. A map of the land use of studied area shows the sampling sites along Citarum River from Balekambang (1) to Nanjung (14) as well as the tributaries (A to G) (modified from [38]).

XRD SmartLab (Rigaku, Japan) equipment identified magnetic materials in extracted samples. Magnetic hysteresis parameters (Bc, Bcr, Ms, and Mrs) were determined using a vibrating sample magnetometer (VSM 1.2 H/CT/HT by Oxford Instrument, Oxfordshire, UK) at a maximum applied field of 1 T. A small amount of bulk sample was used for the analysis. At room temperature, magnetization was measured in relation to applied field for each sample. The ratios of Mrs/Ms against Bcr/Bc indicate the sample's primary magnetic domain state. The extracted magnetic minerals were attached to a sample holder made of metal using carbon tape, then analyzed using SEM (JSM 6360LA) JEOL, Tokyo, Japan. This instrument is equipped with an EDX system for semi-quantitative compositional analysis.

RESULTS AND DISCUSSION

Figure 2 shows the diffractogram results of selected extraction samples from the Citarum River sediments and its tributaries, which show that the magnetic mineral content of the Citarum River sediment samples and its tributaries is dominated by magnetite minerals. This result was obtained after the XRD data of the sample in the form of different diffraction peaks, which are the hkl values of each mineral in the sample, was evaluated based on the mineral data base. However, it was also seen that the tributary samples contained the mineral albite ($NaAlSi_3O_8$), which was not present in the Citarum River sediments.

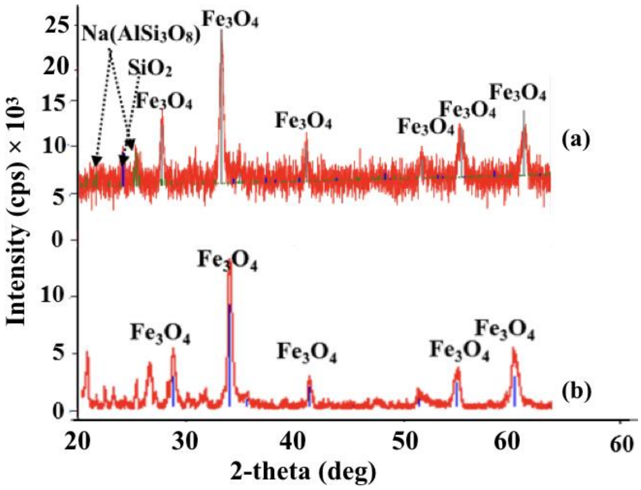

Figure 2. Diffractogram shows the mineral magnetite (Fe_3O_4) in suspended sediment samples from a tributary (Cikasungka River) (a) and Citarum River (location 15) (b).

Based on the results of XRD analysis, the magnetic mineral contained in sediment samples from the Citarum River and its tributaries was magnetite. This is in accordance with its geological condition, where The Citarum River and its tributaries were located in the Cibeureum Formation, Kosambi Formation and Cikapundung Formation. The Cibeureum Formation consists of volcanic breccia composed of scoria fragments, andesite igneous rock, basalt and pumice (the result of undecomposed volcanoes, part of Mount Tangkuban Perahu), and tuff with a low level of consolidation, as well as several basalt lava inserts, with Late Pleistocene-Holocene age. The Kosambi Formation consists of mudstone, siltstone, and sandstone that has not yet compacted with the Holocene age. The Cikapundung Formation consists of conglomerate and compact breccia, tuff and andesite lava inserts of early Pleistocene age, and volcanic (quaternary) rock deposits (which are volcanic rock deposits) [39]. The magnetic mineral magnetite is most abundant in igneous rocks [40]. The sediment of the Citarum River and its tributaries comes from the weathering of these igneous rocks. As is known, the Citarum River sediment also comes from its tributaries, so the magnetite mineral indicated in the Citarum River sediment samples can also come from its tributaries [36].

Figure 3 shows the results of VSM measurements for the extracted suspended sediment samples in the form of hysteresis curves. This curve can be thought of as having two parts: an upward curve that depicts an increase in the magnetic field (H), and a downward curve that depicts a decrease in the magnetic field. The dotted line is the result of the initial VSM measurement, indicating a mixture of paramagnetic and ferrimagnetic materials. This is the same as that obtained by Dillona and Franke [41], who obtained a hysteresis graph like the results of this research with saturation magnetization, which is usually reached in low to moderate fields (<500 mT), which is indicative of the presence of a soft ferrimagnetic mineral phase.

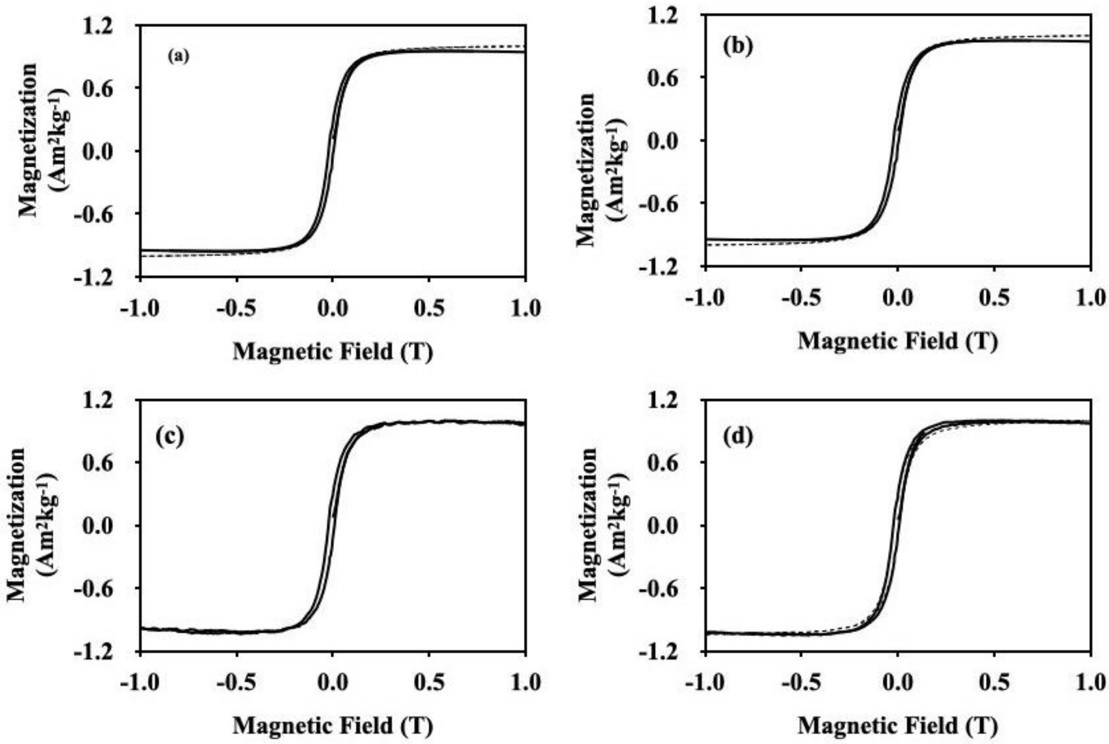

Figure 3. Magnetic hysteresis curves before the correction for the paramagnetic contribution (represented by the dotted line) and after the correction (represented by the dotted line) for selected samples.

To investigate further, SEM-EDX analysis was carried out to determine the details of the morphology and texture of magnetic mineral grains contained in sediment samples from the Citarum River and its tributaries that had been extracted using a magnetic stirrer. This is important because the specific morphology of anthropogenic magnetic particles can be distinguished from that of pedogenic particles [42]. Fig. 4 (a-d) show the magnetic grains from Citarum River sediments, while Fig. 4 (e-h) shows the magnetic grains of the tributary sediments. Based on the morphological details, magnetic grains from Citarum River sediments are dominated by octahedral and angular shapes. Meanwhile, magnetic grains from tributary sediments show an octahedral and angular shape with damage at the corners and a perfectly round shape. In terms of grain size, magnetic mineral grains from Citarum River sediments have a size of 2.5–20.1 mm, as well as from tributaries 6.4–59.9 μm). The size of the magnetic minerals has been measured using the ImageJ application. EDX analysis of the magnetic grains from both sites shows that the main magnetic grains are iron oxide. EDX analysis on the selected grains in Fig. 4 (a-d) shows that the main elements are

Fe with Ti, Al, Co, Cd, Zn, Pb, Hg, Ni, Cu, and Mn as minor elements. A similar analysis of magnetic grains originating from the tributaries of Fig. 4 (e-g), indicates that the main element is Fe with Ti, Al, Co, Cd, Zn, Pb, Hg, Ni, Cu, and Mn as minor elements (Table 1).

Table 1. EDX analysis on the selected grains from the suspended sediments of the Citarum River and its tributaries.

Element	Citarum River					Tributaries			
	Grain M1	Grain M2	Grain M3	Grain M4	Grain M5	Grain M6	Grain M7	Grain M8	Grain M9
O	29.64	27.35	30.85	31.92	35.54	4.01	40.94	43.80	54.29
Al	3.06	2.90	2.55	3.52	1.13	1.69	0.44	2.50	10.91
Ti	10.20	11.55	9.75	8.28	1.20	1.47	0.26	0.31	0.89
Mn	-	-	0.16	-	-	0.77	0.05	0.07	0.24
Fe	47.38	52.81	49.74	47.56	53.60	91.56	57.79	52.68	33.25
Co	4.54	4.52	4.25	4.66	4.33	-	0.27	0.23	0.06
Ni	0.78	0.28	0.18	-	0.93	0.10	0.21	0.14	0.10
Cu	0.10	0.04	-	-	0.56	-	0.04	-	-
Zn	1.10	0.51	0.84	0.47	-	-	-	0.01	0.01
Cd	0.58	0.04	0.27	0.25	0.14	0.05	-	0.22	0.06
Hg	1.06	-	0.76	1.84	1.48	0.03	-	0.04	0.19
Pb	1.56	-	0.65	1.01	1.09	0.32	-	-	-
Total	100.00	100.00	100.00	99.51	100.00	100.00	100.00	100.00	100.00

Based on the results from the SEM and EDX analyses, it is shown that the magnetic grains from the Citarum River and its tributaries come from two sources, namely pedogenic and anthropogenic. Based on their morphology, the magnetic grains from the Citarum River are predominantly octahedral or angular in shape and broken at the corners, indicating a pedogenic origin. Octahedral and angular shapes are typical of titanomagnetite fragments [42]. Based on the size of the magnetic mineral grains from the Citarum River, which show a relatively small grain size, it also confirms that the magnetic grains from the Citarum River are dominated by pedogenic sources. Additionally, the presence of magnetic mineral grains in the shape of spherules suggests their anthropogenic origin, likely from river tributaries. This is attributed to the fact that the sediment from the Citarum River also originates from its tributaries.

Magnetic mineral grains from tributaries show that at each site taken, it contains magnetic minerals that are octahedral and broken at the corners, which indicates that these magnetic minerals are of pedogenic origin but have undergone weathering [42]. Besides that, there are also magnetic minerals that are perfectly spherical in shape, which indicates that these magnetic minerals originate from anthropogenic processes. Numerous studies have shown that magnetic spherules are common in fly ash, roadside dust, and sediments, which originate from burning fossil fuels from vehicles or furnaces at high temperatures [29],[36],[3]7,[42].

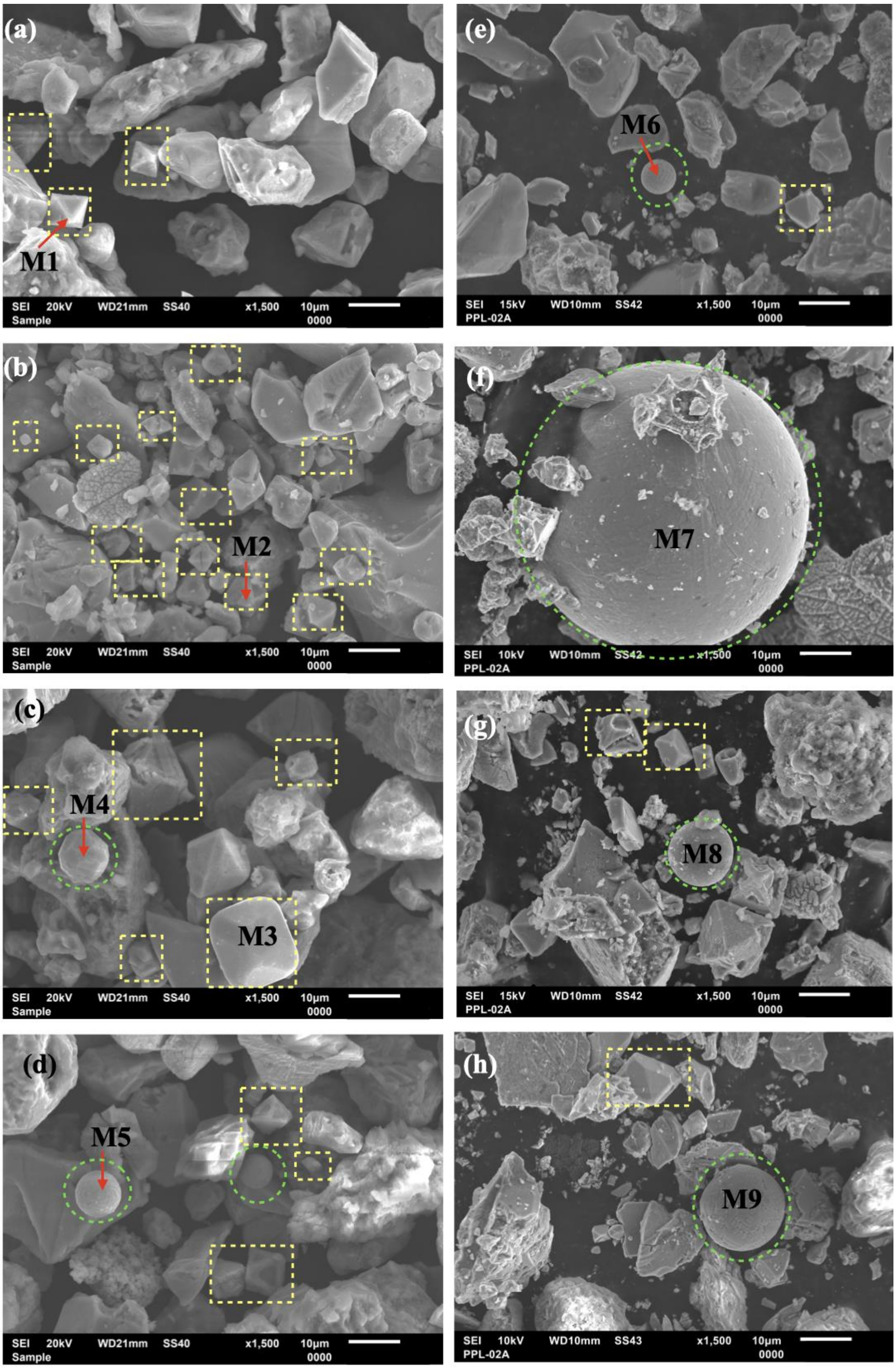

Figure 4. Scanning electron microscopy (SEM) images of the magnetic extracts in sediment samples: (M1–M5) magnetic particles from Citarum River and (M6–M9) magnetic particles from the tributaries Based on EDX analyses, the particles shown on the picture are iron oxides, probably magnetite and titanomagnetite. (M1–M5), the grains are predominantly octahedral (yellow rectangle) and spherules (green circle). The grains M6–M9 are spherules.

The results above are in accordance with what was explained by Huliselan and Bijaksana [42] regarding the shape of magnetic mineral grains in the soil around the study area, which are octahedral or angular in shape and damaged at the corners due to weathering. Based on this, it can be concluded that the magnetic minerals contained in the suspended sediments of the Citarum River and its tributaries are of pedogenic and anthropogenic origin. This is also supported by the EDX analysis of magnetic mineral grains in sediment samples. This semi-quantitative composition analysis shows that the elemental composition of the magnetic mineral grains in the Citarum River sediments is comparable to that in the tributary sediments. An indication that the magnetic mineral grains from Citarum River sediments and its tributaries are of anthropogenic origin is supported by the EDX results, which show the presence of Fe with Ti, Al, Co, Cd, Zn, Pb, Hg, Ni, Cu, and Mn as minor elements (Table 1). However, there are differences in the content of the element Mn in the magnetic mineral grains; not all magnetic mineral grains originating from the Citarum River contain Mn, but all magnetic grains originating from tributaries do.

The magnetite grains that do not contain Mn in the Citarum River samples come from locations 1 and 2, which are located in the upstream area. Meanwhile, downstream sediment samples (location 15) from the Citarum River revealed the presence of Mn-containing magnetic grains. This shows that sediment originating from tributaries will influence the sediment of the Citarum River. This also indicates that the magnetic mineral grains originating from tributaries are more anthropogenic in origin because these tributaries originate from industrial areas, dense settlements, heavy traffic, and areas exposed to air polluted by industrial exhaust. This is in accordance with what was stated by previous researchers: that the presence of Mn was caused by human activities (anthropogenic) in industrial areas [43], densely populated areas [44], fossil fuel and traffic emissions [45]. Therefore, investigating river sediments using SEM EDX is useful for estimating the sources of anthropogenic processes, especially for the Citarum River and its tributaries. The application of this investigation in other areas requires comprehensive research to confirm these results.

CONCLUSION

Detailed morphological and mineralogical analyses were carried out on suspended sediments from the Citarum River and its tributaries in Bandung, Indonesia. Based on hysteresis and XRD analysis, magnetic grains in suspended sediments are dominated by ferrimagnetic minerals such as magnetite (Fe_3O_4). SEM observations identified that magnetic grains in suspended sediments from the Citarum River were mostly octahedral and angular in shape, indicating their lithogenic origin, whereas magnetic grains from its tributaries were imperfectly spherical in shape, indicating an origin from anthropogenic processes. These results indicate that river tributary sediments contain magnetic minerals resulting from human activities, which have the potential to be a polluting factor. This study warns us of the potential for contamination of the sediment of the Citarum River tributaries, which will also affect the sediment of the Citarum River and the surrounding environment.

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