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Determination of Individual Magnetic Particle Sources in Sediments from the Wae Tomu River Estuary, Ambon City, Indonesia: Scanning Electron Microscope (SEM) and Energy-Dispersive X-Ray Spectroscope (EDX) Analysis

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ABSTRACT

This paper describes the determination of individual magnetic particle sources found in the sediment of the Wae Tomu river estuary in Ambon City, Indonesia. The sample was magnetic particles extracted from the sediment. As comparative data, magnetic particles were also extracted from the soil in the river upstream. These particles were characterized using a scanning electron microscope (SEM), energy-dispersive X-ray spectroscope (EDX), and X-ray diffraction (XRD). SEM analysis results showed that the magnetic particles found in the sediment have a spherule-shaped and framboid-like surface morphology measuring ≈43-97 μm, while magnetic particles found in the soil were octahedral and angular-shaped with a maximum length of ≈40-60 μm. The majority of the elemental composition of the magnetic particles from the sediment was Fe and O, followed by minor elements of Zn, Cu, S, Al, Si, and Cr. In contrast, the majority of elements from the soil were Fe and O, followed by minor elements of Ti, Al, and Mg. The result of X-ray Diffraction (XRD) analysis showed that the particles of the sediment are chromite and magnesite, while the soil is magnetite. Based on the morphological characteristics and elemental composition, the magnetic particles from the sediment originated from anthropogenic sources, i.e., motor vehicle emissions. Additionally, SEM and EDX can be used to differentiate individual magnetic particles from both anthropogenic and natural sources.

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INTRODUCTION

Recently, anthropogenic activity has become a serious problem for urban areas in the world. High population levels in an area lead to increased anthropogenic activities, resulting in the release of various pollutants, including magnetic particles that pose a significant threat to human health and the environment. Wind and water easily transport these particles from their formation site, leading to their presence on roads (Lu et al. 2011; Zhang et al. 2012), human settlements (Rout et al. 2014), rivers (Zhang et al. 2011), lakes (Akinyemi et al. 2013), and the sea (Horng

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et al. 2009). These particles can originate from various sources, such as fossil fuel combustion in motor vehicle emissions (Kim et al. 2012; Baghdadi et al. 2012), lubricating oil (Sarvi et al. 2011), vehicle brake abrasion (Kim et al. 2007; Lu et al. 2015), and abrasion and corrosion of vehicle engines (Wang et al. 2020). Additionally, other anthropogenic activities such as solid waste (Huliselan et al. 2010), agricultural practices (Xie et al. 2005), and construction activities (Satsangi & Yadav 2014) also produce various particles.

Numerous studies have used the analysis of magnetic properties of materials, such as the c_{LF} vs. c_{FD} scatter plot (Bijaksana & Huliselan 2010), the SIRM/c_{LF} vs. B_{cr} plot (Huliselan et al. 2010), and the Day plot based on the ratio of hysteresis curve parameters H_{cr}/H_c vs. M_{rs}/M_s (Yang et al. 2010; Meena et al. 2011; Zhang et al. 2012). In addition, mineralogical analysis using X-ray diffraction (XRD) is also used as supporting data to distinguish the source of these magnetic particles (Huliselan et al. 2010; Satsangi and Yadav 2014). However, the results of these analyses generally express the particle bulk, while individual particle analysis becomes difficult using these methods. In contrast, individual particle analysis will make recognizing and distinguishing these particles easier.

One appropriate analytical technique for distinguishing these particles is scanning electron microscopy (SEM) in conjunction with energy dispersive X-ray spectroscopy (EDX). SEM's principal advantage lies in its ability to clearly observe the morphological image of particles, whether in groups or individually. This facilitates the process of distinguishing and determining the origin of these particles. EDX analysis more readily determines the elemental composition of individual particles identified by SEM. The combination of SEM and EDX analysis facilitates the determination of the origin of individual magnetic particles, thereby ensuring the precise and accurate identification of their source. The combination of SEM and EDX analysis has been successfully employed to ascertain the morphology and elemental composition of particles (Zhang et al., 2011; Baghdadi et al., 2012; Zhu et al., 2012; Naimi and Ayoubi, 2013; Shi et al., 2014; Kocić et al., 2014; Franzin et al., 2021; Malá et al., 2023; Abdulla & Souri 2024; Dalal Guin & Deswal 2024 and Uğurlu & Kumruoğlu 2024). Researchers conducted analyses on a variety of particles extracted from soil, including sediment (Horng et al. 2009; Zhang et al. 2011), ash, and dust (Rout et al. 2014, Kim et al. 2012, Magiera et al. 2011, and Zhu et al. 2013). Additionally, researchers have conducted studies on fly ash (Rohilla et al., 2018; Rani et al., 2017) and leachate sludge (Huliselan et al., 2010).

This study examines the morphology and elemental composition of magnetic particles extracted from estuarine sediments and soils from the upper reaches of the Wae Tomu River in Ambon City. The results obtained were used to distinguish and determine the origin of the magnetic particles. Additionally, this scientific information aids in comprehending the morphology and elemental composition characteristics of both anthropogenic and natural magnetic particles.

MATERIALS AND METHODS

The Wae Tomu River is one of the rivers located in Ambon City, Maluku Province, Indonesia. The estuary of this river is connected to the sea in the inner Ambon Bay and close to the highway and is »100 m from the Ambon City public transport terminal. Motorized vehicle activities on the highway and offices and residential areas line the river basin, reaching upstream of the river. The location for sediment sampling at the river estuary and soil samples were also taken from the upstream area of the Wae Tomu River (Fig.1).

This research was started with the collection of 500 g of sediment samples each at 4 spots (4 samples) of the Wae Tomu River, and the samples were inserted into the sample plastic bag. Furthermore, the samples obtained underwent a separation process between magnetic particles and non-magnetic particles. This separation was started with placing 50 g of sediment into a

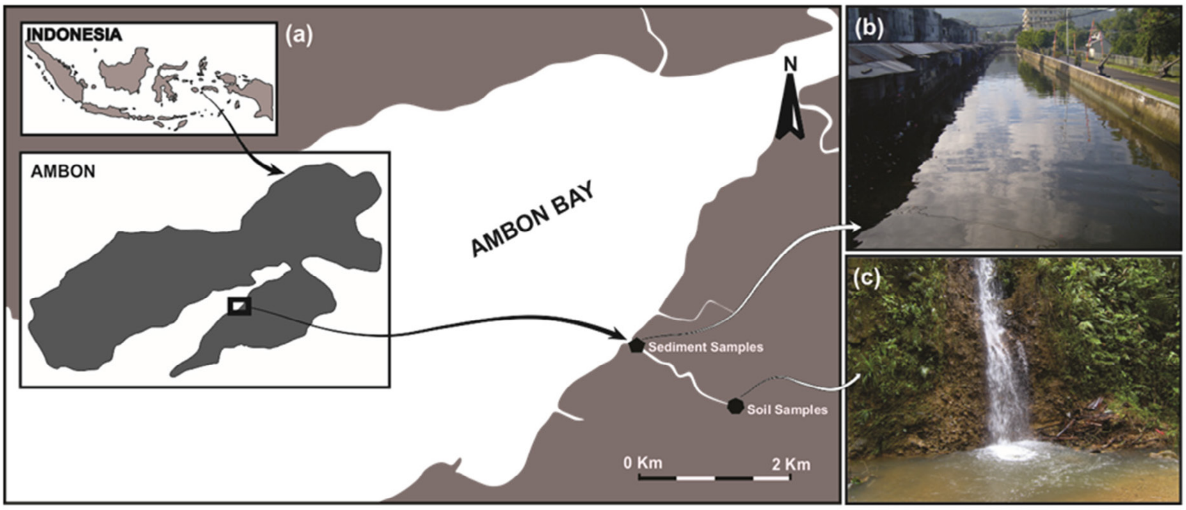

Fig. 1. Map of sampling location for sediment and soil samples: (a). map of Ambon Bay, (b). estuary area of the Wae Tomu River, and (c). upstream area of the Wae Tomu River

1000 mL glass measuring cup. To facilitate the extraction process, the sediment was mixed with 200 mL of methanol.

The extraction process was performed using a permanent magnetic bar after the magnet was wrapped in a thin, transparent plastic. The plastic wrapping of the magnets was intended to make it easier to separate magnetic particles, so they did not directly stick to the magnets. The magnetic particles attached to the plastic were removed and put into a sonicator bath containing 100 mL of methanol to perform a sonication process for 5 min. This process aimed to separate impurities from the surface of the particles. After the sonication process, the second stage extraction process was performed, and this process was repeated three times to obtain a pure magnetic particle (Fig.2). The same steps and processes were also carried out for soil samples, each taken at 4 spots (4 samples) in the upstream area of the Wae Tomu River.

The magnetic particles obtained were then prepared for the process of morphological and mineralogical analysis using a scanning electron microscope (SEM) and an energy dispersive X-ray spectroscope (EDX). Firstly, the extracted magnetic particles from the sediment and soil were attached to the specimen holder. After cleaning the specimen holder with a hand blower, we applied gold-palladium coating to the specimen. The particles were inserted into the specimen chamber. Then observation and image shooting were carried out using the SEM (JEOL JSM-6360LA model) equipped with the EDX system (JEOL JED-2300), which was operated at a voltage of 10 kV. To strengthen the results of the elemental composition analysis of magnetic particles obtained from EDX, XRD analysis was carried out on unextracted and extracted sediment and soil samples. This analysis utilized a PANalytical X'Pert PRO PW3040/x0 X-ray Diffraction. The Petrology and Mineralogy Laboratory, Center for Geological Survey, Bandung, Indonesia, performed this analysis.

RESULTS AND DISCUSSION

Morphology and elemental composition of particles

Fig. 3 displays the results of the morphological analysis of the magnetic particles extracted from the sediment using SEM. The magnetic particles in the sediment have three different shapes and sizes on the SEM images: a spherule with a smooth surface (Fig. 3a), a framboid with a rough surface (Fig. 3b), and a spherule with fine particles scattered on the surface (Fig.

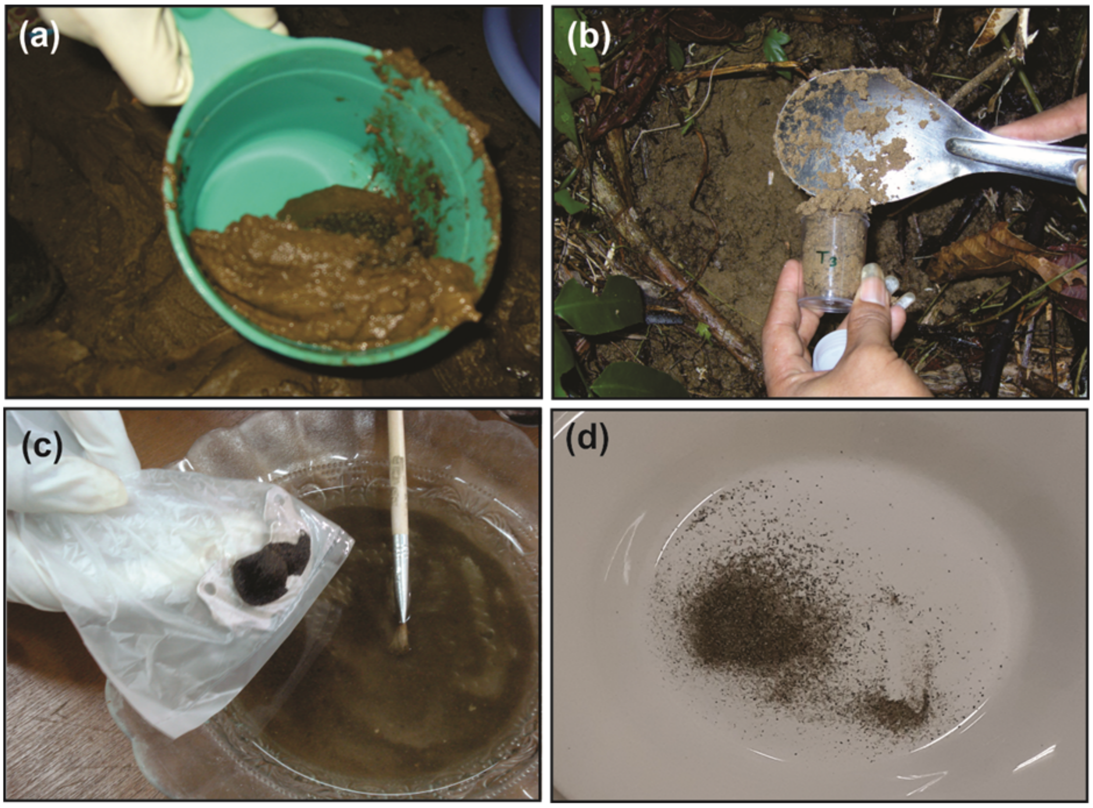

Fig. 2. Sampling and extraction process of sediment and soil samples: (a). sediment sample, (b). soil sample, (c). extraction process, and (d). magnetic particles

3c). Researchers have also found particles with this morphology in leachate sludge (Huliselan et al. 2010), sediment (Horng et al. 2009), dust (Zhu et al. 2013), fly ash (Rohilla et al. 2018; Sun et al. 2020; Rani and Jain 2017), and urban soil (Wang et al. 2017; Baghdadi et al. 2012; Zong et al. 2017). In terms of grain size, the magnetic particles of the sediment were »97 mm (Fig. 3a), »86 mm (Fig. 3b), and »43 mm (Fig. 3c) in diameter. Several studies showed that particles of this size are also found in aerosols (Chen et al. 2019) and dust (Zhu et al. 2012; Liati et al. 2012; Kelepertzis et al. 2019). Magnetic particles from the sediment have a different morphology than the particles from the soil. The soil contains magnetic particles that are perfectly octahedral (Fig. 3d) and angular, featuring sharp and cracked edges (Fig. 3e-f). These particles have a maximum inter-angular length of 60 mm (Fig. 3d), 40 mm (Fig. 3e), and 50 mm (Fig. 3f). Particles of this morphology and size are also found in the soil from landfills (Huliselan et al. 2010) and urban areas (Baghdadi et al. 2012).

The results of elemental composition analysis using EDX validated the suspected sources of magnetic particles, as illustrated in Fig.4. The EDX analysis results presented in Fig. 4a-c illustrate the composition and mass percentage of the magnetic particles identified in the sediment. Fig. 4d-f displays the composition and mass percentage of the magnetic particles present in the soil. Fe and O elements dominate the elemental composition of the magnetic particles extracted from the sediment and soil. The total mass percentage of these particles ranged from 74.13 to 98.75% in the sediment and from 85.51 to 90.34% in the soil. This indicated that these particles were iron oxide (Zhu et al., 2013).

Furthermore, the composition and mass percentage of each particle element in the sediment differed from those observed in the soil. The sediment displays the mass percentage of the largest to the smallest elements in each particle, as follows: (i) The particles in Fig. 3a show

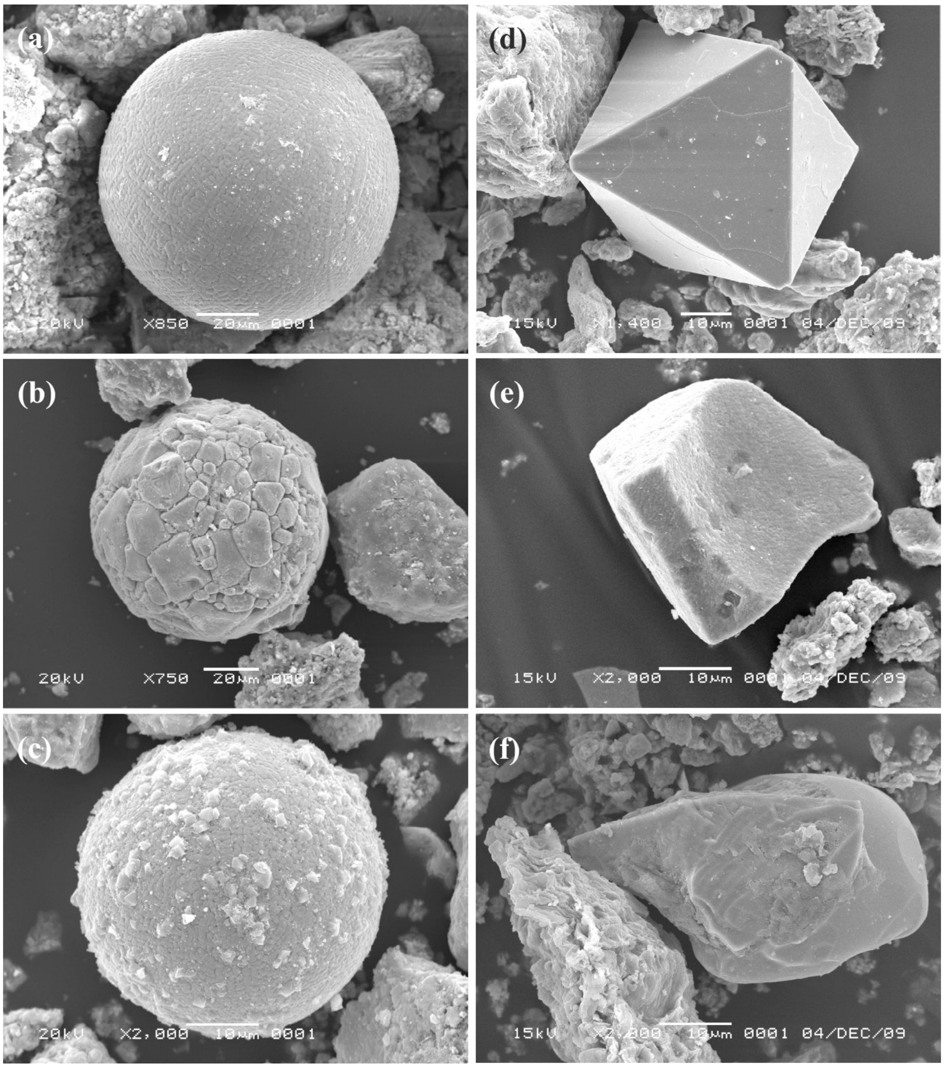

Fig. 3. SEM images of magnetic particles: (a-c) magnetic particles from the sediment, and (d-f) magnetic particles from the soil.

that there is more Fe than O, Al, Cu, Si, and Cr, based on the EDX results in Fig. 4a. (ii) The EDX results in Fig. 4b show that the particles in Fig. 3b have more Fe than O, Zn, Cu, Al, and Si. (iii) The particles in Fig. 3c of element Fe exceed O, S, Al, and Si, respectively, based on the EDX results in Fig. 4c. Another study's results showed that spherule iron oxide particles, which include elements Cr, Cu, Mn, and Pb, were found in Fig. 3a of the element Fe>O>Al>Cu>Si>Cr. Studies have also revealed that anthropogenic sources provide the Zn (Trippetta et al., 2016). In contrast, the magnetic particles from the soil (Fig. 3d-f) exhibit a similar elemental composition and mass percentage of elements, with Fe, O, Ti, Al, and Mg occurring in the same order of magnitude (Fig. 4d-f). Furthermore, the composition of these elements is comparable with the results of previous studies investigating the origin of particles in dust (Trippetta et al., 2016) and aerosols (Li et al., 2011).

Figure 5 presents XRD analyses of powdered samples of sediment and soil, both without extraction and with extracted magnetic particles, in addition to EDX. The magnetic particles extracted from the sediment exhibit distinct characteristics compared to those extracted from the soil. The sediment primarily yields chromite and magnesite particles, while the soil primarily contains magnetite. Also, the sediment and soil samples that were not extracted show clear differences. The sediment particles that were extracted have a lot of xanthoconite, quartz, zirconia

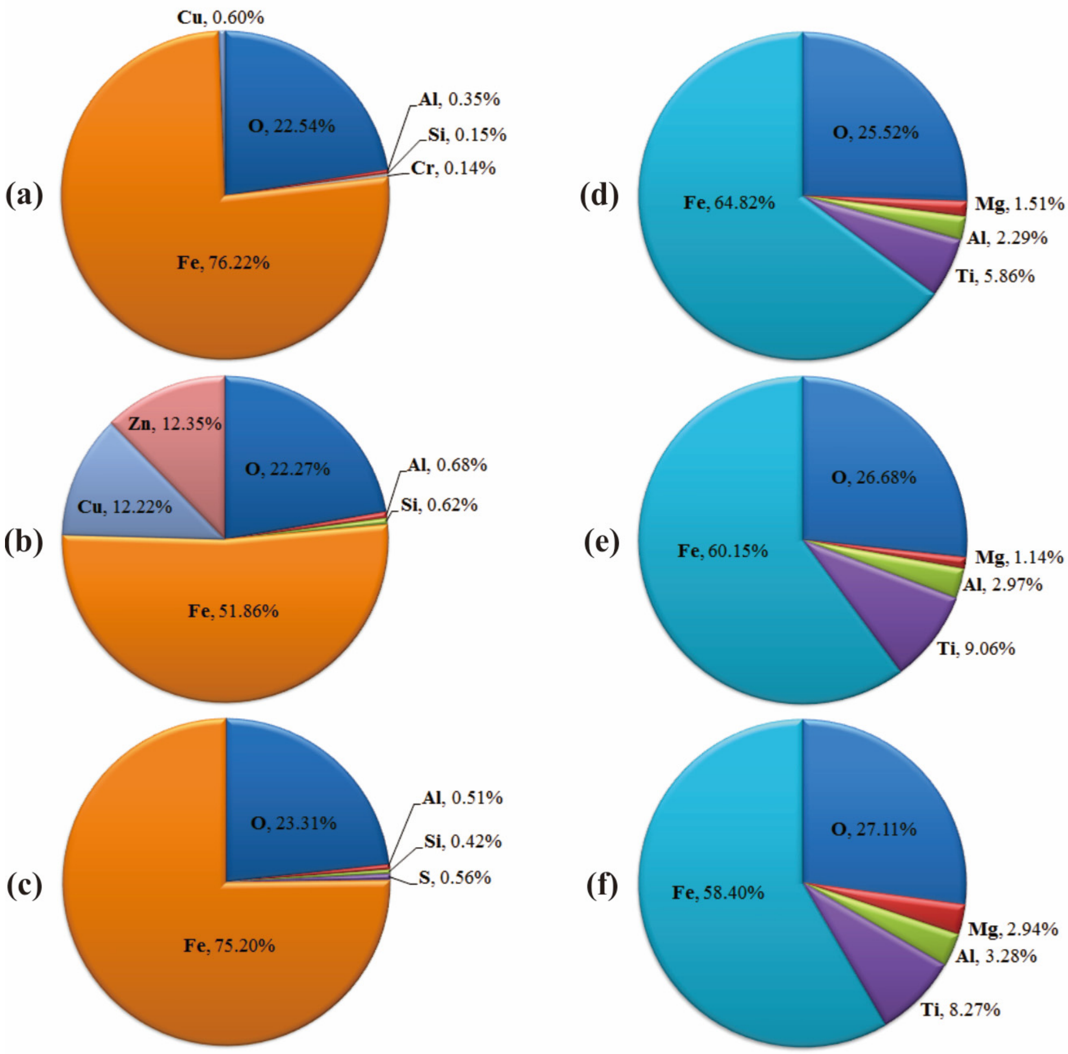

Fig. 4. EDX analysis results of magnetic particles from the sediment and the soil show: (a-c) the elemental composition of the sediment particles, and (d-f) the elemental composition of the soil particles.

(Ce-doped), and kyanite. On the other hand, kaolinite and quartz dominate the soil samples. The results showed that anthropogenic processes derived the magnetic particles present in the sediment, while natural processes produced the ones observed in the soil. According to Adekiya et al. (2024), the minerals such as kaolinite and quartz are from the natural process of soil.

Determination of magnetic particles source using SEM and EDX analysis

The Wae Tomu River's sediment contains magnetic particles that may have originated from natural or anthropogenic sources, or both. Natural sources can originate from the parent rocks' weathering and/or erosion. In contrast, anthropogenic sources can come from various human activities in the upstream area, along river flows, or near river estuaries. The morphological analysis using SEM reveals that the magnetic particles from the sediment share a morphology (spherule and framboid-like surface) with anthropogenic activity at high temperatures (Francová et al. 2017). These activities originate from the combustion process of motorized vehicles (Abu Khatita et al. 2016; Kelepertzis et al. 2019). Based on the grain size, the spherule particles and framboid-like surface of the sediment have a size (»43–97 mm) proportional to the particle size resulting from various anthropogenic activities. Numerous studies demonstrate the presence

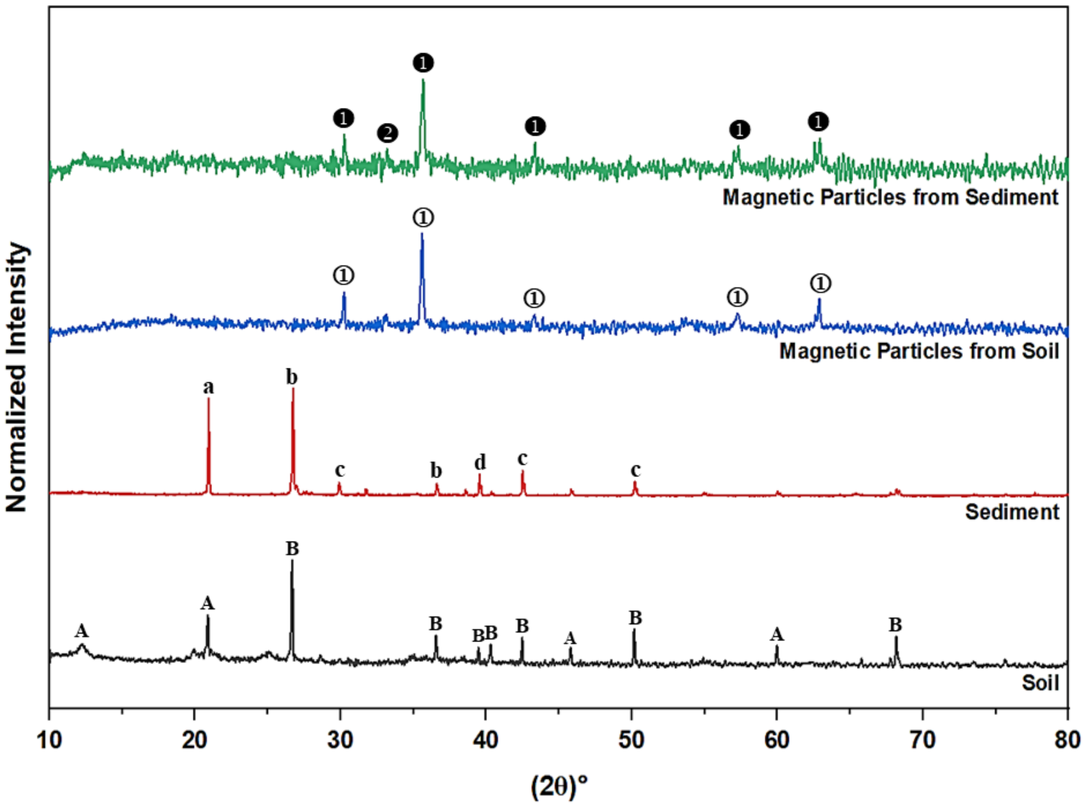

Fig. 5. X-ray diffractograms of sediment and soil that are extracted magnetic particles and not extracted ((❶) chromite syn (Fe +2Cr_2O_4); (❷) magnesite ($Mg(CO_3)$; (①) magnetite (Fe_3O_4); (a) xanthoconite (Ag_3AsS_3); (b) quartz (SiO_2); (c) zirconia (Ce-doped) syn ($(Zr_{0.88}Ce_{0.12})O_2$); (d) kyanite (Al_2SiO_5); (A) kaolinite ($Al_2Si_2O_5(OH)_4$); (B) quartz (SiO_2)).

of spherule magnetic particles of this size in dust (Zhu et al. 2012; Kelepertzis et al. 2019), sediment (Horng et al. 2009), soil (Baghdadi et al. 2012), and leachate (Huliselan et al. 2010). The combustion temperature from which these particles originate determines whether the surface of the spherule particles is smooth or not (Xie et al. 2005).

Moreover, by analyzing the elemental composition of the particles using EDX, we can prove that these particles originate from anthropogenic sources, given the diversity of anthropogenic activities in the area along the river flow and around river estuaries. The EDX analysis results reveal that Fe and O dominate the elemental composition of the sediment's particles, with minor elements Zn, Cu, S, Al, Si, and Cr following closely behind. The discovery of the elements Zn, Cu, S, and Cr in the samples proved that the magnetic particles from the sediment were purely anthropogenic. Fe, Cu, Al, Si, and Cr elements suggest the origin of these particles from the abrasion and corrosion of motor vehicle engine parts (Wang et al. 2020; Liati et al. 2012), while the presence of S and Zn elements likely stems from fuel oil and lubricating oil disposal (Sarvi et al. 2011). Another prediction is that the spherule-shaped and framboid-like surface particles originate from anthropogenic activity at high temperatures. Thus, it is possible that these particles come from the combustion process of motorized vehicles or ship engines burning within the bay of Ambon. The morphology and elemental composition of the magnetic particles from the sediment are different from those of forest fires (Ukraintsev et al. 2020) and cooking activities using gas and electric stoves (Buonanno et al. 2009). The magnetic particles in the sediment are almost identical to those in the garbage dump leachate, but their surfaces are imperfect or cracked (Huliselan et al. 2010). Connecting the estuary of the Wae Tomu River to the Ambon Bay, a hub of ship activities, could potentially lead to the origin of these particles from ship engine emissions. Although the morphology of the particles originating from ship engine emissions is in the form of a spherule, the elemental composition is different from that of the particles from the sediment (Kim et al. 2015).

On the other hand, if the magnetic particles found in the sediment come from the soil in the upstream area, then the morphology and elemental composition will be the same. However, SEM analysis shows that the shape of magnetic particles in the soil is different from that of particles in the sediment, which have a spherule and framboid-like surface. The shape of the particles in the sediment is octahedral and angular. Magnetic particles with octahedral and angular morphology are derived from natural processes (Huliselan et al. 2010; Baghdadi et al. 2012; Baatar et al. 2017). These natural processes occur through weathering and/or erosion of the parent rocks, which depend on the mineral composition, structure, and texture of the rocks, climate, and anthropogenic pressures (Marszałek et al. 2014). In addition, during weathering, amorphous iron oxides are released and crystallize over time, and the rate of the process depends on the pedo-environmental conditions (Jaworska et al. 2016).

Furthermore, the particles that originate from natural processes exhibit an irregular and angular shape, lacking porosity (Connolly et al. 2020). The analysis results of the soil's magnetic particle composition, primarily consisting of Fe and O with minor elements of Al, Ti, and Mg, reinforce this assumption, suggesting that these particles originated from natural processes like weathering and/or erosion from the parent rocks. The presence of Fe-Ti elements indicates that these particles are thought to be titanomagnetite (Perkins 1996). This assumption was proven by XRD analysis of magnetic particles extracted from the soil to be magnetite (F_3O_4). The composition of these elements is also comparable with the elemental composition of the particles from soil dust (Trippetta et al. 2016). The differences in morphology and composition of magnetic particles from sediment and soil proved that the two groups of particles originated from different sources. The magnetic particles found in the sediment came from anthropogenic sources, i.e., motor vehicle emissions, while the particles found in the soil came from natural sources. This is likely due to the fact that Ambon City is not an industrial city, and the majority of anthropogenic activities near river estuaries are related to transportation activities from public transport terminals and roads. On the other hand, the presence of magnetic particles associated with heavy metals such as Fe, Zn, Cu, S, Al, Si, and Cr will act as potential contaminants and adversely affect humans and the environment if they enter the food chain. To reduce this condition, it is expected that vehicle combustion exhausts must have particle filters.

CONCLUSIONS

Detailed morphological and elemental composition analyses were performed on magnetic particles extracted from the sediment and soil of the Wae Tomu River. SEM analysis results show that the magnetic particles found in the sediment have spherule-like and framboid-like surface morphology with particle sizes of »43–97 mm. Particles with this morphology generally derive from anthropogenic sources at high temperatures. On the other hand, magnetic particles found in the soil have different morphology because they are octahedral and angular without pores on the surface with a maximum length of »40–60 mm, suggesting that these particles have a natural origin. The SEM results were further confirmed by EDX analysis, which shows that the elemental composition of the magnetic particles found in the sediment was different from the soil. This means that the magnetic particles found in the sediment are not from the soil. The discovery of Fe, O, Cu, Al, Si, and Cr elements on magnetic particles from the sediment proved that these particles originated from anthropogenic sources, especially abrasion and corrosion of motor vehicle engine parts. Moreover, the discovery of S and Zn elements suggests that fuel oil and lubricating oil are the source of these particles. Magnetic particles found in the soil have elements of Fe, O, Ti, Al, and Mg, proving that these particles originated from natural sources, such as weathering and/or erosion from the parent rocks. The results of EDX analysis are also confirmed by XRD analysis, which shows that the magnetic particles contained in sediments tend to come from anthropogenic sources while in soils, from natural sources. In particular, morphological characteristics and elemental composition can be used to determine the source of the anthropogenic magnetic particles. On the other hand, if these anthropogenic particles multiply and accumulate over time, it can lead to adverse effects on both the environment and human health.

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CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

LIFE SCIENCE REPORTING

This article does not contain experiments on humans or animals.

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