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Preliminary Assessment of Morphology and Elemental Composition of Fine Particulates in Selected Urban Areas of Java Island

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ABSTRACT

Rapid urbanization and high population density in three major cities in Indonesia, Bandung, Jakarta, and Tangerang have led to significant air quality issues. Fine inhalable particles ($PM_{2.5}$) with distinct morphologies and elemental compositions pose considerable health risks. This study evaluates the morphology and chemical composition of $PM_{2.5}$ in these urban areas on Java Island. $PM_{2.5}$ samples were collected for 24-hour periods using a Teflon filter with the Super Speciation-Air Sampling System (SuperSASS) following the EPA sampling schedule, from May to September 2022. The Teflon sample with the highest $PM_{2.5}$ concentration, representative of both weekdays and weekends, was selected for morphological analysis using Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) and elemental characterization using Energy-Dispersive X-ray Fluorescence (ED-XRF) Epsilon. SEM images revealed distinct morphological characteristics at each site. In Bandung, particles were irregularly shaped, agglomerated, and flaky, with sizes ranging from 1.1-1.6 µm on weekdays and 0.9-1.3 µm on weekends. Jakarta has particles with semi-crystalline, tabular, elongated, and puff-like morphology, with sizes predominantly from 0.5-0.8 µm on weekdays and 0.9-1.3 µm weekends. In Tangerang, particles were irregularly faceted and agglomerated, with sizes between 0.5-1.3 µm on weekdays and 0.9-1.4 µm on weekends. Teflon-derived minerals (C,F) were present across all sites. EDX spectra revealed Ca-rich particles in Bandung, while S-rich particles were observed in Jakarta and Tangerang. XRF analysis further proved the major and minor elements, reflecting local pollution sources. The combined use of SEM-EDX and XRF offers a comprehensive profile of $PM_{2.5}$, highlighting specific pollution sources in each city.

1. INTRODUCTION

Air pollution is a global issue and is recognized as the second-largest contributor to the global disease burden (IHME, 2019). As of 2023, Indonesia ranked as the 14th most polluted country in the World (IQair, 2023a). Among various air pollutants, fine inhalable particulates ($PM_{2.5}$) with aerodynamic dimensions of 2.5 micrometers or smaller, with a size 30 times smaller than human hair diameter (EPA, 2023) poses significant health risks due to their ability to penetrate deeply into the lungs and irritate the alveolar wall (Yang et al., 2020). In Indonesia, rapid urbanization and industrialization have exacerbated $PM_{2.5}$ levels, particularly in densely populated areas like Java Island, where population densities range from 500-1,000 people per square kilometer (Statistics Indonesia, 2023). Data indicate that $PM_{2.5}$ concentrations in major cities on Java Island, such as Bandung, Jakarta, and Tangerang, exceed World Health Organization (WHO) guidelines by 7 to 10 times (IQair, 2023b). This increase can be attributed to various factors, including industrial activities, traffic congestion, agricultural burning, and rapid urbanization (Hopke et al., 2008; Lestari et al., 2020; Santoso et al., 2020; Santoso et al., 2008a). As of 2023, Bandung, the fourth-biggest city, has a

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population of 2,714,215 (World Population Review, 2024b) and 2.2 million vehicles (Syahrial, 2023), Jakarta is categorized as a megacity with a population of 11,436,004 (World Population Review, 2024a) and approximately 24 million vehicles as of 2024 (Adji, 2024), Tangerang, the third-biggest city, has an estimated population of 2,570,980 (World Population Review, 2024c) with around 934,720 vehicles as of 2022 (Statistics Indonesia, 2022). Therefore, monitoring of $PM_{2.5}$ levels in these three cities-Bandung, Jakarta, and Tangerang is a significant concern in understanding the sources and composition for effective air quality management.

Extensive research on $PM_{2.5}$ has been conducted globally (Hopke et al., 2020; Manisalidis et al., 2020). In Indonesia, research on air pollution began in the early 2000s, focusing on various aspects such as the elemental analysis of industrial emissions in major cities (Santoso et al., 2019), health impacts of air pollution (Ramdhan et al., 2021), the effects of biomass burning and forest fires (Permadi and Kim Oanh, 2013; Santoso et al., 2022), and integration of satellite data with real-time monitoring (Sinaga et al., 2020; Siregar et al., 2022). Despite the growing research on $PM_{2.5}$, studies focusing on the surface morphology of $PM_{2.5}$ in Indonesia are still limited, particularly in Urban Areas on Java Island. Existing research primarily relies on data from other regions, which may not fully capture the unique characteristics of Indonesian urban areas. The morphology of $PM_{2.5}$, including its size, shape, and surface characteristics, plays a crucial role in understanding its sources, transport, and potential health impacts. The shape of particulate matter (PM) significantly influences its deposition efficiency and interactions with biological fluids in the lungs (Mokbel, 2024; Pallarés et al., 2020; Stearns et al., 2001). The irregular and sharp-edged particles, in particular, can cause injury to lung tissues upon inhalation (Fatima et al., 2022). Previous research has documented various common shapes of $PM_{2.5}$, such as spherical particles associated with anthropogenic and combustion sources (González et al., 2016; Khobragade and Ahirwar, 2023), aggregated and irregular particles linked to carbonaceous material and road dust (Khobragade and Vikram Ahirwar, 2023), the lamellar appearance of silicates (Khobragade and Ahirwar, 2023), rectangular particle (EPA, 2002), and prismatic regular shapes associated with Si-rich particles from natural sources (González et al., 2016; Paoletti et al., 2003). Despite the importance of this information, research on the surface morphology of $PM_{2.5}$ in Indonesia remains scarce. Therefore, investigating the morphology and elemental composition of fine particulates in selected urban areas is essential for gaining comprehensive understanding of $PM_{2.5}$ in the region. Currently, urbanization in Indonesia is accelerating, with 56% of the population residing in urban areas (Statistics Indonesia, 2023). Compared to other Asian countries, Indonesia's urbanization rate is growing rapidly at 4.1% per year (The World Bank, 2016). This urbanization trend is likely contributing to rising $PM_{2.5}$ levels. The cities of Jakarta, Bandung, and Tangerang were selected for this study to represent urban areas on Java Island.

The research involves the collection of $PM_{2.5}$ samples using Super Speciation Air Sampling System (SuperSASS), determination of $PM_{2.5}$ concentration through the gravimetric method, analysis of $PM_{2.5}$ surface morphology using Scanning electron microscopy-energy dispersive X-ray (SEM-EDX), and characterization of elemental composition using Energy Dispersive XRF(ED-XRF) Epsilon 5. Accordingly, this study aims to bridge the existing gap by conducting a preliminary assessment of $PM_{2.5}$ morphology and elemental composition to obtain a more comprehensive profile of $PM_{2.5}$ in selected urban areas of Java Island.

2. METHODOLOGY

2.1 Collection of fine particulate ($PM_{2.5}$) sample using SuperSASS

The $PM_{2.5}$ was collected from May to September 2022 in three selected cities in Indonesia (Bandung, Jakarta, and Tangerang) following the Environmental Protection Agency (EPA) sampling schedule in 2022. The sampling sites of the selected three urban areas on Java Island are shown in Figure 1. Bandung site (1), the largest city on the southern tip of Java Island is 630 meters above sea level, Bandung sampling site is located at Kawasan Kerja Bersama (KKB)-BRIN JL. Tamansari number 71 (-6.8885 407208374305, 107.60795837023203). The Bandung site has a distinctive geographical landscape, characterized by a mountainous landscape with basin topography which affects air circulation and may trap pollutants within the basin (Kurniawati et al., 2024). As depicted in Figure 1, KKB Tamansari is located in a densely populated region. Within a 1-kilometer radius, there is a public sports area (SABUGA) to the north, Bandung Zoo (Kebun Binatang Bandung) to the south, the Bandung Institute of Technology (ITB) to the east, and department stores and hotels to the west.

The Jakarta site (2) is located at the Laboratorium Lingkungan Hidup Daerah (LLHD), Jl. Casablanca Kav. 1 Kuningan, Jakarta Selatan (-6.2243 38159194404, 106.83546398262821). The Jakarta site at LLHD is positioned on the main road of south Jakarta and surrounded by the offices area, hotels, and train station. Within a 1-kilometer radius, there are hotels and a plaza mall to the north and west, public residences to the east, and an LRT station to the south.

Tangerang Site (3) is located at the Science and Technopark/Kawasan Sains dan Teknologi (KST) B.J. Habibie, Serpong, Jl. Raya Puspitek Serpong, Kecamatan Setu, Kota Tangerang Selatan (-6.353488465685582, 106.67229646758928). The sampling site in Tangerang is situated close to the main road of Serpong. About 1 km to the north,are public school, several offices, and a bus station. To the east, there are public sports areas and residential housing, while the west and south are occupied by offices and residential areas.

Figure 1. Sampling site of $PM_{2.5}$ of three urban Areas (Bandung, Jakarta, and Tangerang) on Java Island in Indonesia

$PM_{2.5}$ was collected over 24-hour using the Super Speciation Air Sampling System (SuperSASS) instrument, developed and field-tested by the United States Environmental Protection Agency (US EPA). The samples were collected on a 47 mm PTFE (Polytetrafluoroethylene) $(C_2F_4)_n$/Teflon filter with pore size of 0.2 um, maintained at a constant flow rate of 6.7 L/min. The SuperSASS consists of two canister sets: Set 1, containing canisters 1, 2, 3, and 4, and Set 2, containing canisters 5, 6, 7, and 8, as illustrated in Figure 2. The canister sets were used alternately on a weekly basis, following the EPA schedule. The PTFE filter was placed inside the 1st, 2nd, 3rd, and 4th canisters, designed for elemental and inorganic analysis of $PM_{2.5}$ samples (Met One Instruments, 2001). Each canister in the SuperSASS is equipped with a sharp cut cyclone (SCC), which efficiently separates solid and liquid coarse particles without grease or oil. The sampling data , including duration, filter volume, flow rate, temperature, and pressure were extracted via an RS-232 port and transferred to a PC using SASSCommAQ software (Met One

Instruments, 2001). The recorded pressure during the sampling period remained relatively stable at each site, with pressure ranging from 693 to 694 mmHg in Bandung, 755 to 756 mmHg in Jakarta, and 752 to 753 mmHg in Tangerang.

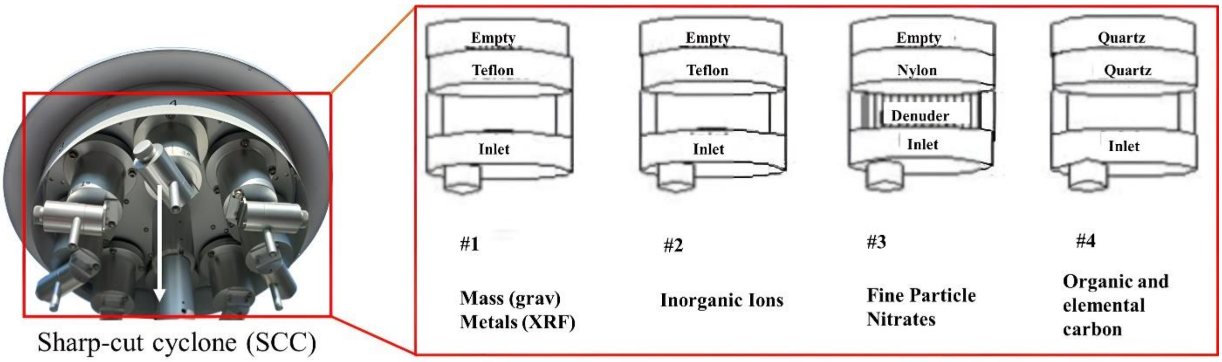

Figure 2. Canister sets of Super Speciation Air Sampling System (SuperSASS)

2.2 Gravimetric analysis of $PM_{2.5}$

The concentration of $PM_{2.5}$ was determined by gravimetric analysis, which involved weighing the initial and final mass of the Teflon filter using the microbalance (model MX5 Mettler Toledo) at room humidity of around 45% and air temperature at 20±2°C. The $PM_{2.5}$ concentration was calculated based on Equation 1 (Research Triangle Institute, 2008), where the difference mass of the filter after sampling (Mf) and before sampling (Mi) was divided by filter volume (Va). During the sampling period, the Teflon filter containing the highest $PM_{2.5}$ levels on both weekdays and weekends was chosen for morphology and elemental analysis.

$$PM_{2.5}\left(\frac{\mu g}{m^3}\right) = \frac{Mf\,(mg) - Mi(mg)}{V_a\,(m^3)} \times 1{,}000 \quad (1)$$

2.3 Morphology analysis using SEM-EDX

The morphology, including the shape and size of $PM_{2.5}$ was characterized using Scanning electron microscopy (SEM) coupled with energy dispersive X-ray analysis (EDX) (JEOL JSM 6510LA) in the Research Center for Nuclear Beam Analysis Technology, National Research and Innovation Agency (BRIN). The SEM analysis was conducted in three cycles throughout 2022: July 28-29, September 23, and October 29. For the SEM-EDX analysis, the non-conductive Teflon filter with the highest $PM_{2.5}$ concentration from both weekends and weekdays was cut into 5 mm^2 size, and coated with a thin layer of gold (Au) to render it electrically conductive. This coating was applied using a vacuum plasma coating device. The coated Teflon sample was measured at an accelerating voltage of 10 kV, with a working distance (WD) of 10 mm, and a magnification ranging from 1,000-10,000x. Particle sizes observed in SEM images were estimated using ImageJ software.

2.3 Elemental analysis using ED-XRF

2.3.1 EDXRF Epsilon 5 set up

The elemental characterization of the Teflon filter with the highest $PM_{2.5}$ levels on both weekdays and weekends was carried out using a non-destructive three-dimensional EDXRF Epsilon 5 spectrometer (PANalytical) in the Research Center for Nuclear Beam Analysis Technology, National Research and Innovation Agency. The elemental analysis was conducted using ED-XRF Epsilon 5 with the following specifications: 100 kV, Gd anode element, polarizing optical path, and secondary target (i.e., Al_2O_3, Fe, CaF_2, Ge, Zr, CeO_2, Mo, Ag, and Al), providing low detection limit of ng/cm^2. The instrument's calibration was set up using standard micromatter®, and a blank standard for multielement. The Teflon filters were loaded into the StainlessSteel sample holders. The sample holder containing 8 filters was positioned in the fixed tray inside the instrument. The system has a fully integrated X-Y sample changer, and the suction device integrated with the handling arm will automatically move each sample to the sample holding (airlock) position (PANalytical, 2005). Each sample was irradiated by a secondary X-ray resulting from the polarizing target, for 1 h. The spectrum of the excitation energy of the sample is recorded by the PAN-32 Ge X-ray detector, placed along the third axis, with an energy resolution of <140 eV (PANalytical, 2009, PANalytical, 2005).

2.3.2 Elemental concentration quantification

To determine the elemental concentration, a calibration and sensitivity curve was made by measuring the thin-film micrometer standards from Micromatter Technologies Inc, with a concentration range of 17-64 µg/cm^2. The standard was measured and the Epsilon software automatically plotted the calibration curve, with the elemental concentration on the X axis and the corresponding intensities on the Y-axis. The detailed calibration steps have been described by (Royani et al., 2016). To assess elemental sensitivity, the atomic number (Z) of the element was plotted on the X-axis against its sensitivity on the Y axis (Royani et al., 2016).

Prior to quantifying the elemental concentration, method validation was conducted using Standard Reference Material (SRM) NIST 2783, Air Particulate on Filter Media, which contains known elemental concentrations. This validation was necessary to ensure that the method's recovery and standard deviation were within acceptable limits. The Teflon filter samples were then analyzed using the same experimental procedure. The elemental concentration (Ci) in Teflon filter was calculated using Equation (2) and (3) (Arana et al., 2014).

$$N_i = aCi + b \qquad (2)$$

$$Ci\ (ng/cm^2) = \frac{(Ni \times Fi)}{(Time \times Current \times Sen_i)} \qquad (3)$$

Where; a is the slope of calibration curve, b is the intercept, Ni is the net peak area of particular element (i) characteristic X-ray (counts), Fi is the absorption factor of element i (≈1), Sen_i is sensitivity of element I in the sample (counts/ng/cm^2/s/mA), Time is measurement time (sec), Current is the current of X-Ray tube (mA), and Ci is the concentration (ng/cm^2). The concentration obtained from EDXRF Epsilon was then converted to ng/m^3 using Equation (4).

$$Ci\ (ng/m^3) = \frac{Ci\ (\frac{ng}{cm^2})}{V\ (m^3)} \times \text{area of dust on filter}\ (cm^2) \qquad (4)$$

Where; Ci is the elemental concentration of $PM_{2.5}$ (ng/m^3), V is the sampling volume using SuperSASS (±9.7 m^3), and the area of dust on the filter (circular area) is 12.56 cm^2.

3. RESULTS AND DISCUSSION

3.1 Gravimetric analysis of $PM_{2.5}$

The $PM_{2.5}$ concentration at each sampling site is depicted in Figure 3. During the sampling period, fluctuation in $PM_{2.5}$ levels was observed. Bandung recorded the most data points compared to Jakarta and Tangerang, as the SuperSASS instrument was initially installed in Bandung, resulting in an extended sampling period. Following the completion of sampling in Bandung, the SuperSASS was to Jakarta and Tangerang. As illustrated in Figure 3(b), Jakarta has only two data points on weekends, due to some samplings not adhering to the EPA sampling schedule, thus excluded in this work.

The $PM_{2.5}$ concentrations determined through gravimetric analysis are presented in Figure 3. In Bandung, the $PM_{2.5}$ concentrations on weekends were found to be relatively higher than on weekdays. The average $PM_{2.5}$ concentration in Bandung was 41 µg/m^3 on weekdays and 42 µg/m^3 on weekends. The slight difference between weekday and weekend concentrations is likely attributable to increased transportation activity and a higher number of visitors during the holiday season, particularly around public areas such as the Bandung Zoo. The highest $PM_{2.5}$ concentrations recorded were 86.5 µg/m^3 on weekdays and 63.2 µg/m^3 on weekends, both of which exceed the Indonesian daily standard of 55 µg/m^3 (The Ministry of State Secretariat of Republic Indonesia, 2021).

In Jakarta and Tangerang, as shown in Figures 3(b) and 3(c), $PM_{2.5}$ levels were higher during weekdays compared to weekends. In Jakarta, the average $PM_{2.5}$ concentration was 36.5 µg/m^3 on weekdays and 36.3 µg/m^3 on weekends. The highest concentrations were 51.6 µg/m^3 on weekdays and 37.4 µg/m^3 on weekends, both below the daily standard of 55 µg/m^3 (The Ministry of State Secretariat of Republic Indonesia, 2021). The sampling site in Jakarta (LLHD-Jakarta) is situated along a main road and is surrounded by office buildings, contributing to elevated traffic and $PM_{2.5}$ levels during weekdays.

In Tangerang, the average concentration of $PM_{2.5}$ was 48.3 µg/m^3 on weekdays and 38.5 µg/m^3 on weekends. The highest concentration recorded was 69.0 µg/m^3 on weekdays, exceeding the daily standard of Indonesian regulation, while the highest weekend concentration was 52.3 µg/m^3, falling below the standard. The SuperSASS instrument in Tangerang is located within the Science and Technopark, approximately 500 meters from the main road, which likely accounts for the higher $PM_{2.5}$ levels during weekdays due to local office and transport activities.

A t-test was conducted to compare the $PM_{2.5}$ concentration between weekdays and weekends at each site (Bandung, Jakarta, Tangerang). The p-values obtained were 0.74 for Bandung, 0.94 for Jakarta, and 0.13 for Tangerang, indicating no statistically significant difference between weekday and weekend concentration at any of the sites (p>0.05).

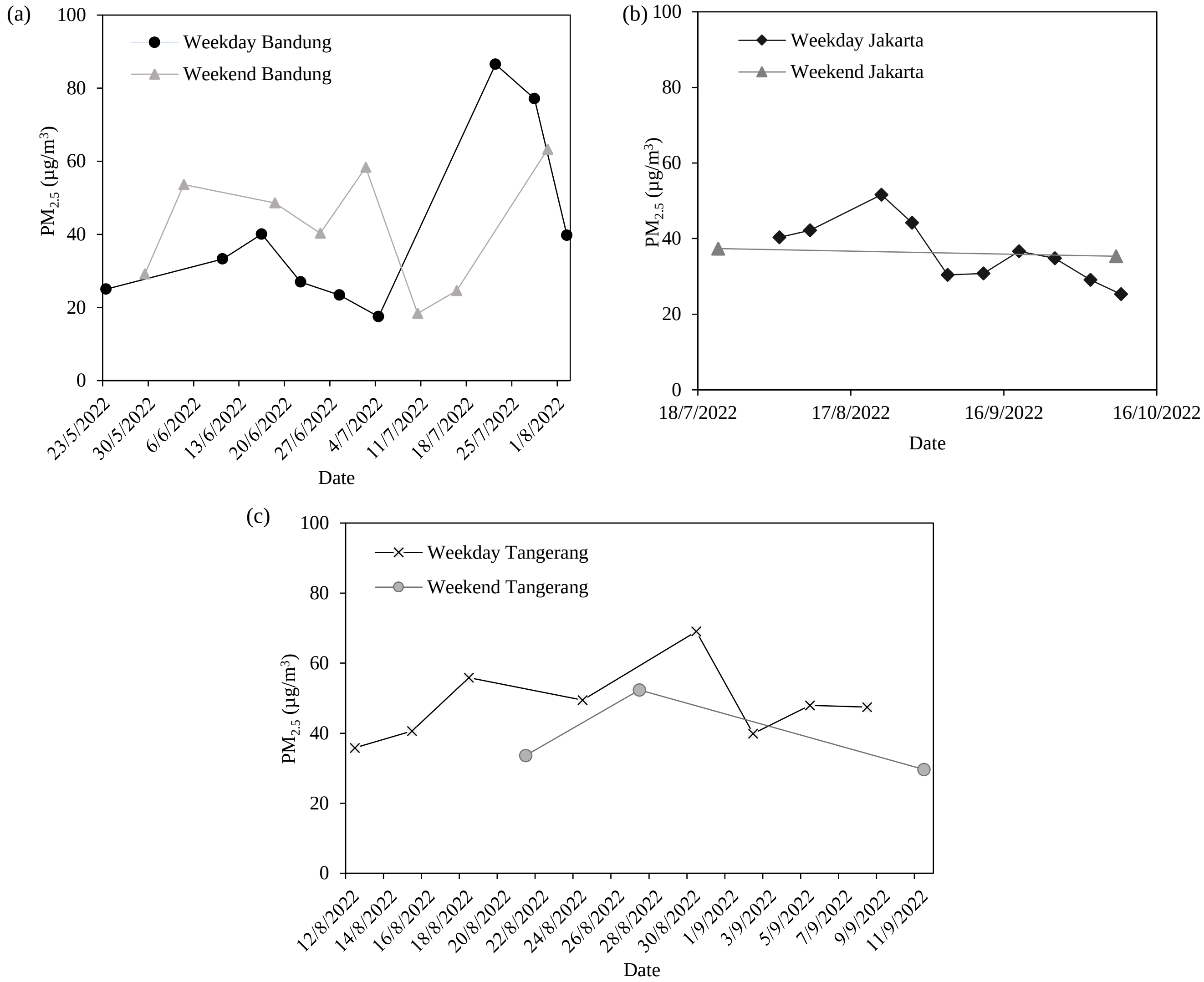

Figure 3. $PM_{2.5}$ of Bandung (a), Jakarta (b), and Tangerang (c) on weekdays and weekends during the sampling period for each site.

3.2 Morphology analysis of $PM_{2.5}$

The morphology of $PM_{2.5}$ was assessed using a blank Teflon filter, which was cut into small parts for SEM analysis. To ensure adequate conductivity during SEM analysis, a thin layer of gold (Au) was sputter-coated onto the Teflon filter. A high magnification of 10,000x was utilized to observe the filter matrix profile in detail. The resulting morphology of the Teflon $(C_2F_4)_n$ filter is depicted in Figure 4. The surface profile of the blank Teflon filter closely resembles that reported in previous studies (Casuccio et al., 2004). It consists of fibrous material made of carbonaceous particles (C) and fluorine (F), interwoven to form a web-like structure. As shown in the Energy Dispersive X-ray (EDX) profile, the Teflon material is primarily composed of carbon (C) and fluorine (F). The Teflon filter is classified as the "depth" filter, effectively capturing fine particles throughout its depth rather than merely on the surface (EPA, 2002), which leads to a lower probability of particle overlap. During the collection of $PM_{2.5}$, fine particles were trapped below the surface or within the filter's web-like structure. As a result, the 24-hour sampling period is unlikely to significantly affect SEM analysis. However, particle agglomeration remains a possibility (Fatima et al., 2022; Liu et al., 2018; Wu et al., 2015), despite the Teflon medium's higher sampling capacity (EPA, 2002).

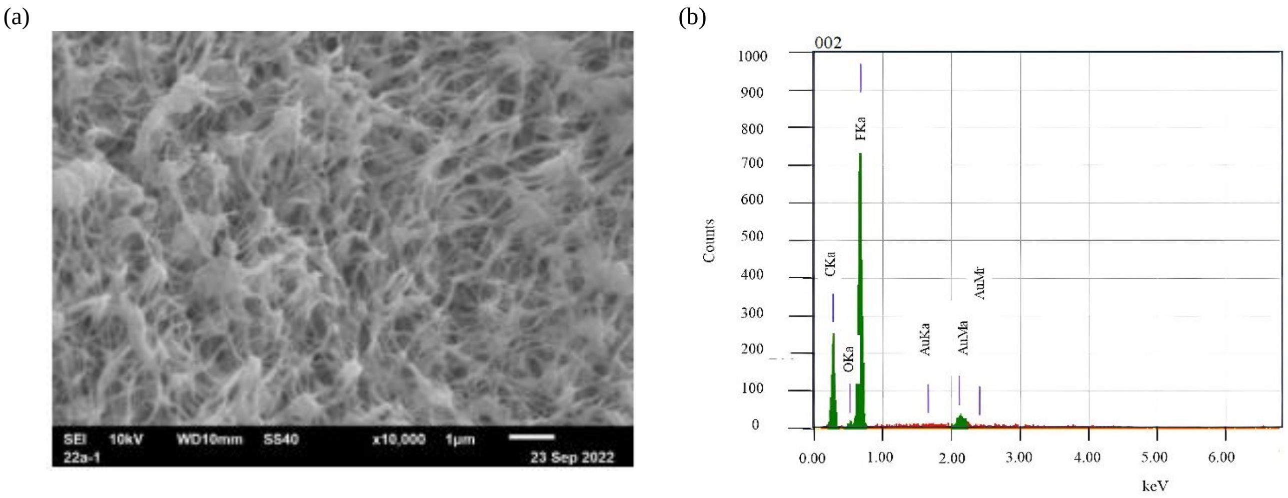

Figure 4. Morphology of Blank Teflon filter with 10,000x magnification (a) EDX profile of Teflon filter (b)

3.2.1 Morphology of $PM_{2.5}$ in Bandung

The Teflon filters exhibiting the highest $PM_{2.5}$ concentrations were selected for detailed morphological and energy-dispersive X-ray (EDX) analysis. The analysis was conducted across multiple magnifications (1200x, 2000x, 3000x, 4000x, 4500x, 5000x), with representative images presented in Figure 5. Additional images are available in the supplementary materials.

The morphological analysis revealed that $PM_{2.5}$ particles on both weekdays and weekends displayed irregular shapes. The particle size distribution, detailed in Figure 6, was assessed by measuring 78 particles on weekdays and 80 particles on weekends at various magnifications. On weekdays, 47 particles had diameters less than 2.5 µm, whereas 70 particles exhibited diameters less than 2.5 µm on weekends. The particle size distribution was comparable between the two periods, with weekdays primarily showing particles in the range of 1.1-1.6 µm, and weekends showing particles in the range of 0.9-1.3 µm. However, particle agglomeration still observed on both weekdays and weekend, having diameter greater than 2.5 µm.

The EDX spectra revealed that both periods were predominantly composed of carbon (C), fluorine (F), and calcium (Ca), as depicted in Figure 5. The high levels of C and F are attributed to the Teflon filter material and carbonaceous components within $PM_{2.5}$. Calcium, likely originating from dust, was present at approximately 21.7% (Figure 5(a)) and 20.3% (Figure 5(b)) by mass on weekdays. However, Ca mass% was notably higher on weekends, with values of 33.1% (Figure 5(c)) and 12.9% (Figure 5(d)). Weekday EDX analysis identified amorphous, agglomerated Ca-rich particles, consistent with prior research (González et al., 2016) and detected soot aggregates indicative of gasoline emissions and biomass combustion (Khobragade and Vikram Ahirwar, 2023). On weekends, Ca-rich particles exhibited an irregular flaky morphology, potentially sourced from windblown dust and road dust (Yue et al., 2013).

The EDX spectra indicated that Ca-rich particles contained significant amounts of carbon and oxygen, suggesting the presence of $CaCO_3$ and limestone/$CaCO_3$ deposits in proximity to the sampling site. Figure 5(a) shows the detection of carbonate minerals along with trace levels of sodium (Na), magnesium (Mg), sulfur (S), and silicon (Si). Elevated sulfur content, indicative of secondary sulfate formation from biomass burning and vehicular emissions, was also observed. Additionally, the presence of secondary Ca-rich particles containing S and O points to the formation of $CaSO_4$ through reactions between $CaCO_3$ and SO_2 during fuel combustion. On weekends, the EDX spectra predominantly featured carbonate minerals (C, O, Ca) with minimal Mg. However, due to the limitation of EDX, other elements contained in $PM_{2.5}$ of Bandung were not observed. Thus, the remaining elements are shown in Figure 11(a).

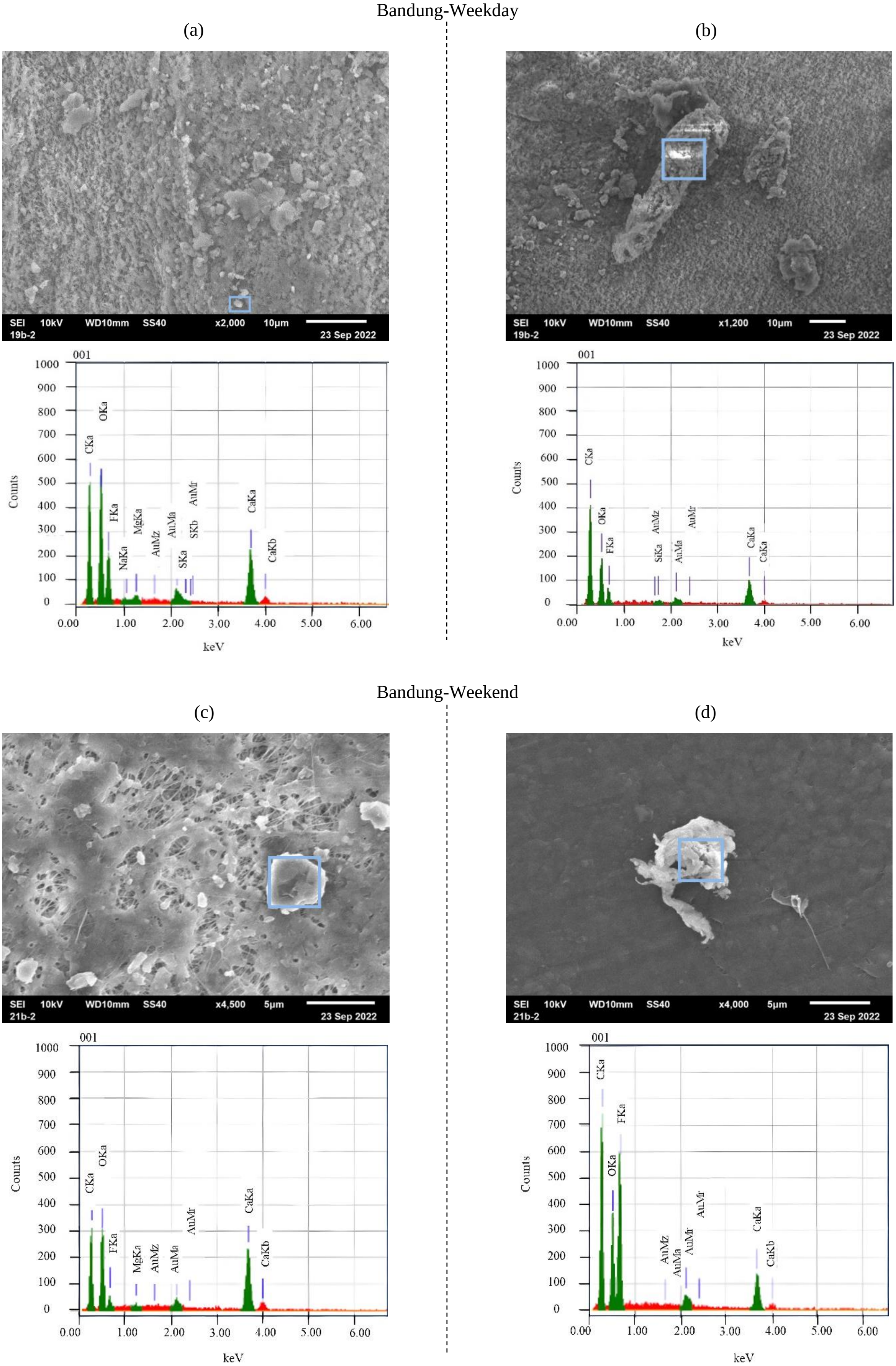

Figure 5. Morphology and EDX spectrum of $PM_{2.5}$ in Bandung on weekdays at 2000x (a) and 1200x (b) magnification, on weekends (b) at 4500x (c) and 4000x (d) magnification.

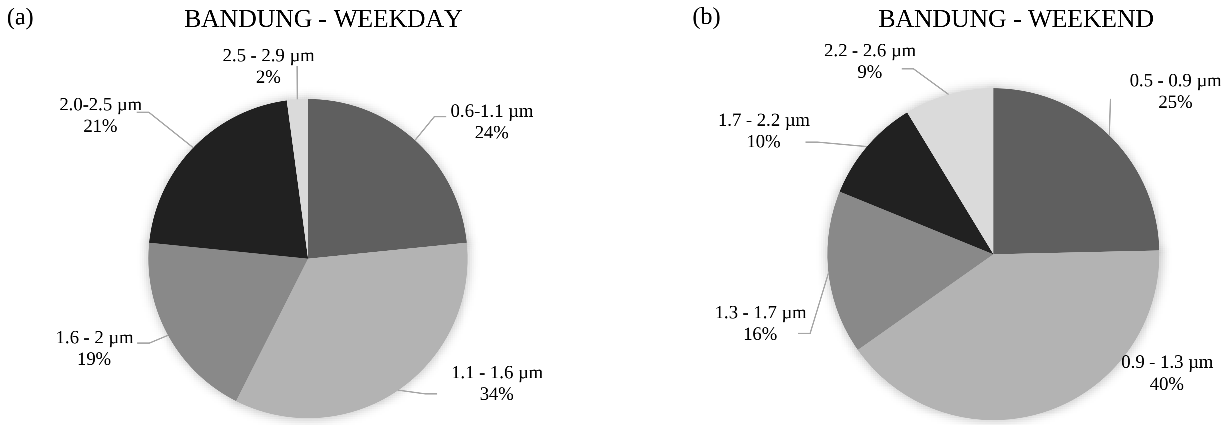

Figure 6. Size distribution of $PM_{2.5}$ collected on weekdays with total selected particle (n=78) (a) and weekends (n=80) (b) in the Bandung site

3.2.2 Morphology of $PM_{2.5}$ in Jakarta

Figure 7 presents the surface characterization of $PM_{2.5}$ in Jakarta. On weekdays, $PM_{2.5}$ particles were predominantly semi-crystalline tabular and elongated forms, while on weekends, they exhibited irregular, puff-like morphologies. In Jakarta sites, the particle size distribution for both weekdays and weekends is shown in Figure 8. The analysis involved measuring 127 particles on weekdays and 115 particles on weekends across various magnifications (2000x, 3000x, 4000x, 5000x, 8000x, 10000x). Results showed that on weekdays, 117 particles had diameters less than 2.5 µm, whereas 109 particles on weekends fell below this size threshold. Weekday $PM_{2.5}$ primarily comprised particles in the size range of 0.5-0.8 µm, whereas weekend particles were mostly in the 0.9-1.3 µm range. However, particle agglomeration was still observed on both weekdays and weekends, with diameters exceeding 2.5 µm.

The EDX spectra presented in Figures 7(a) and 7(b) for weekdays reveal that the tabular particles are mainly composed of carbonaceous material (C) and aluminosilicate minerals (Al, O, Si), along with trace amounts of sodium (Na), potassium (K), sulfur (S), and nitrogen (N), indicating contributions from urban/soil dust (Li et al., 2016), and vehicular emissions. Specifically, Figure 7(b) shows a sulfur-rich elongated particle, suggesting the presence of sulfate (SO_4) (Li et al., 2016). Additionally, both weekdays and weekends showed Teflon-related elements (C and F) and carbon species from $PM_{2.5}$. On weekdays, the EDX spectrum revealed sulfur mass percentages of 6.6% (Figure 7(a)) and 0.3% (Figure 7(b)).

During weekends, as shown in Figures 7(c) and 7(d), the EDX spectra also identified aluminosilicates (Al, O, Si), salt (Na, Cl), magnesium (Mg), sulfur (S), and potassium (K). Figure 7(c) illustrates aggregates containing Na, S, and O, indicative of sodium sulfate (Na_2SO_4) (Li et al., 2016). Notably, the EDX spectrum for a specific SEM image area on weekends showed elevated aluminum (Al) and silicon (Si) levels, with mass percentages of 8.2% for Al and 9.4% for Si (Figure 7(d)).

A more comprehensive elemental profile of Jakarta's $PM_{2.5}$, obtained through EDXRF analysis, is depicted in Figure 11(b). The presence of sulfur (S) and potassium (K) in $PM_{2.5}$ indicates contributions from both biomass burning (K) and gasoline-powered vehicles (S) (Santoso et al., 2014). Compared to Bandung and Tangerang, Jakarta's $PM_{2.5}$ uniquely features NaCl minerals in tabular and irregular forms, likely originating from sea salt due to its proximity to local rivers and its location approximately 11 km north of Ancol Beach (Li et al., 2016).

3.2.3 Morphology of $PM_{2.5}$ in Tangerang

The morphology of $PM_{2.5}$ particles collected in Tangerang is illustrated in Figure 9. Analysis revealed that during weekdays, the $PM_{2.5}$ particle in Tangerang tends to have facetted morphology with relatively small particle size. The particle size distribution for Tangerang is shown in Figure 10, based on measurements of 142 particles on weekdays and 91 particles on weekends. On weekdays, all 120 particles had diameters below 2.5 µm, while on weekends, 65 out of 91 particles were under this size threshold. Weekday $PM_{2.5}$ was predominantly composed of particles in the size range of 0.5-1.3 µm, while weekend particles were mostly in the 0.9-1.4 µm range. However, particle agglomeration still occured on both weekdays and weekends, with diameters exceeding 2.5 µm.

On weekends, the particles were observed to have irregular, agglomerated shapes. Both weekday and weekend samples contained carbonaceous particles (C), including carbonates (Ca, O, C), and sulfur (S)-rich particles.

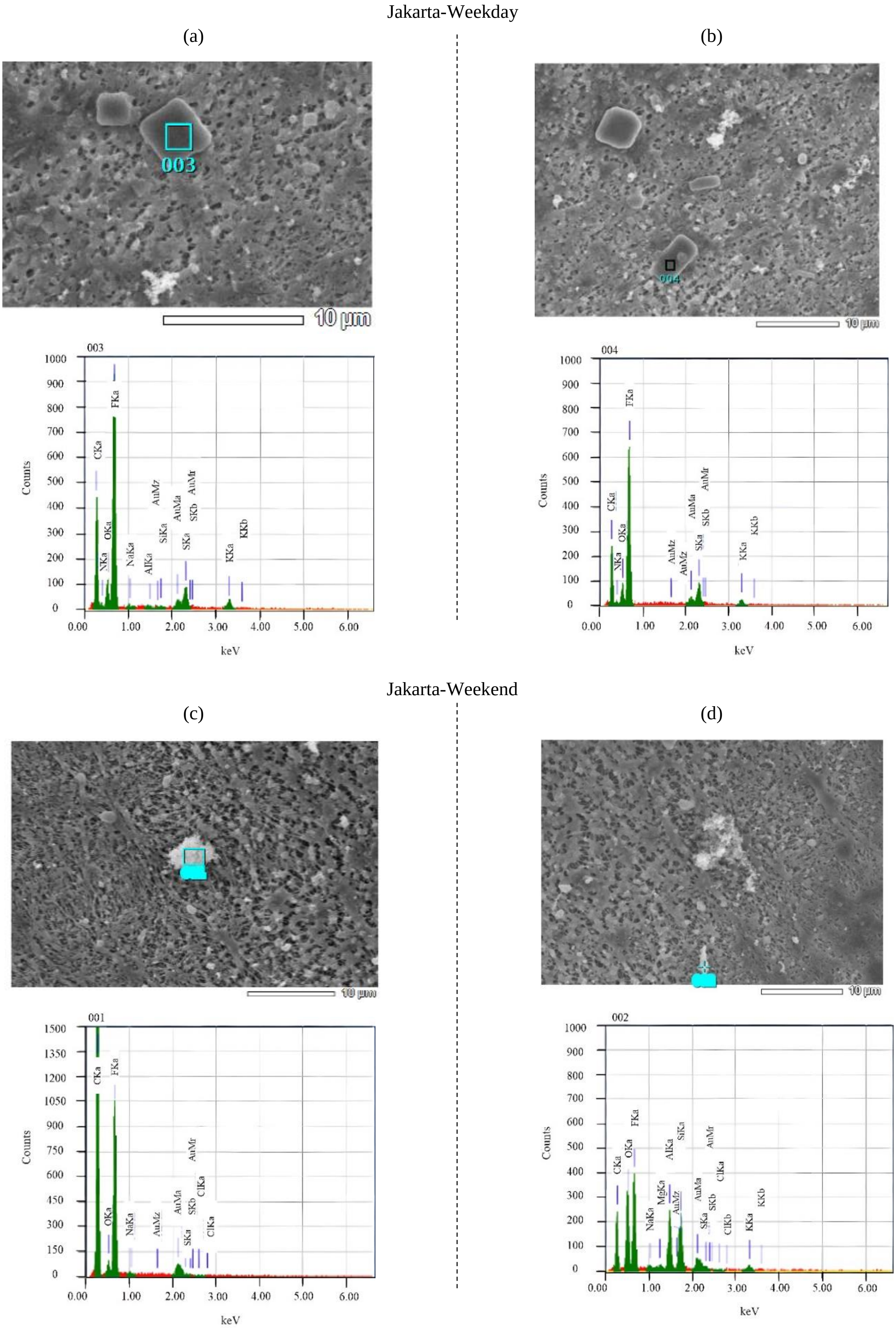

Figure 7. Morphology and EDX spectrum of $PM_{2.5}$ in Jakarta on weekdays at 3000x on area 3 (a) and area 4 (b) and weekends at 3000x magnification on area 1 (c) and area 2 (d).

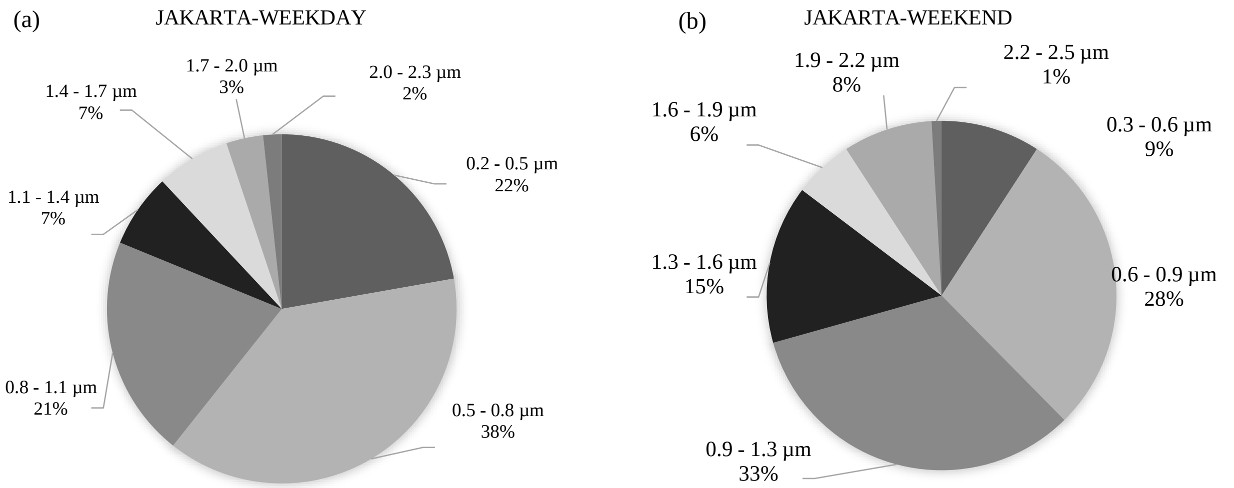

Figure 8. Size distribution of $PM_{2.5}$ collected on weekdays with total selected particles (n=127) (a) and weekends (n=115) (b) in Jakarta site

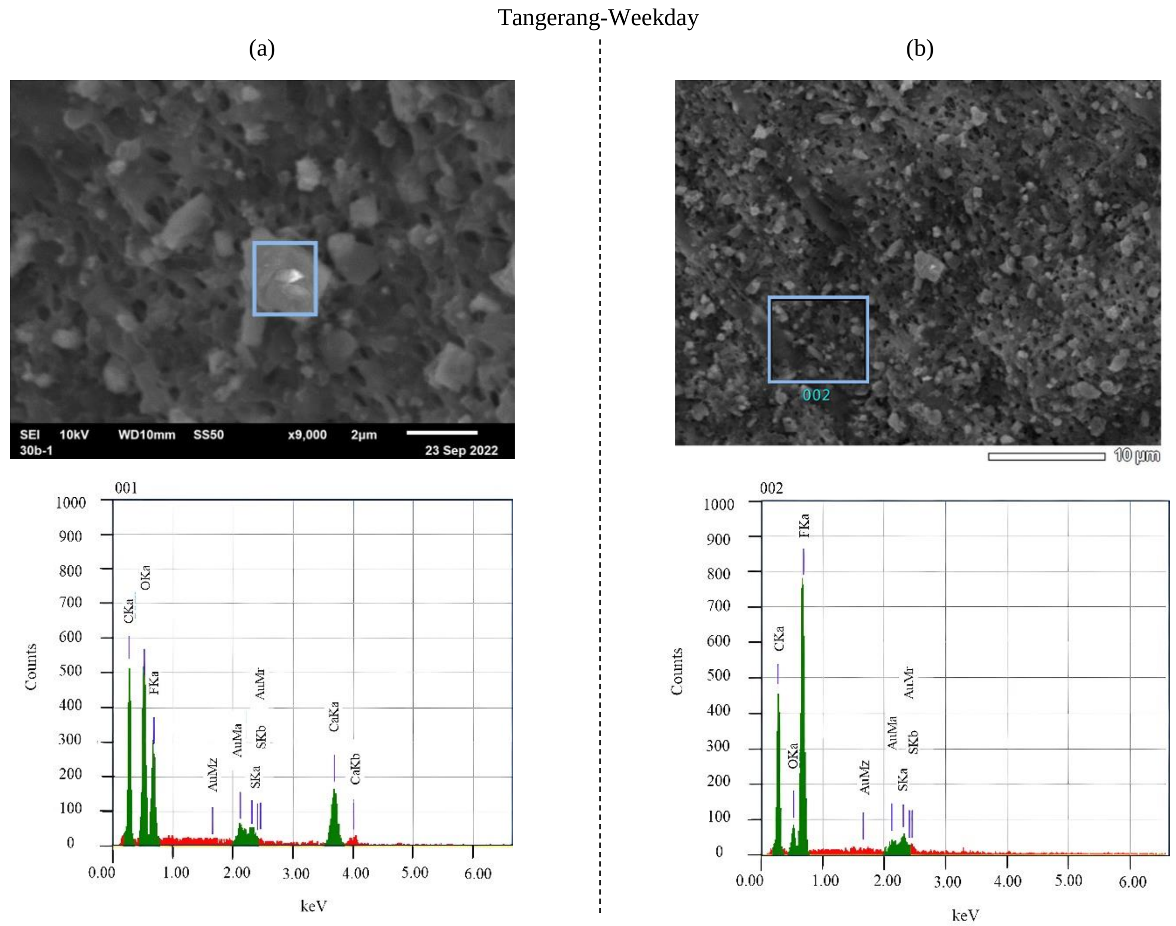

Figure 9. Morphology and EDX spectrum of $PM_{2.5}$ in Tangerang on weekdays at 9000x (a) and 3000x (b), on weekends on 9000x (c) and 8000x (d) magnification

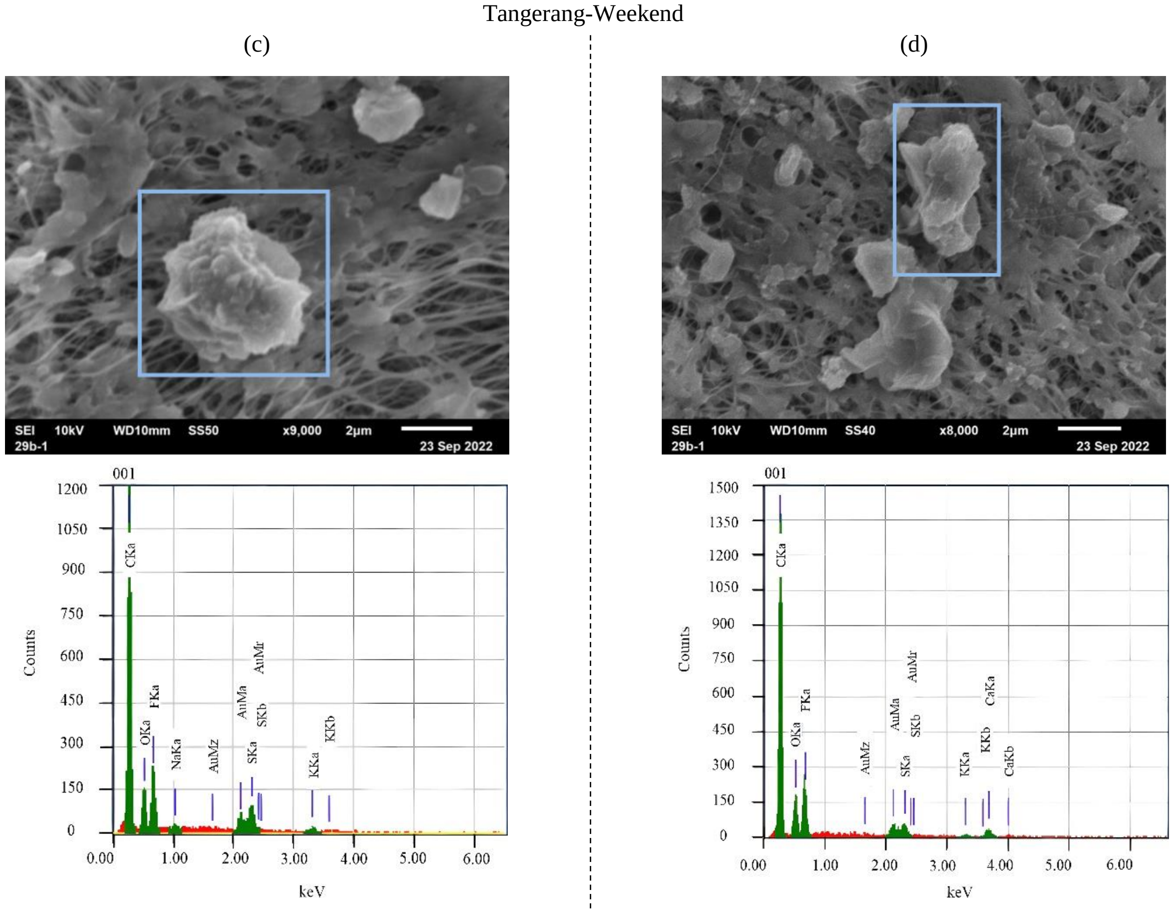

Figure 9. Morphology and EDX spectrum of $PM_{2.5}$ in Tangerang on weekdays at 9000x (a) and 3000x (b), on weekends on 9000x (c) and 8000x (d) magnification (cont.)

The EDX spectra for Tangerang provide detailed elemental composition data for specific areas, as shown in Figures 9(a), 9(b), and 9(d). Both weekday and weekend samples exhibited the presence of Teflon-related elements (C and F) and carbon species. On weekdays, significant levels of calcium (Ca) and sulfur (S) were observed, with Ca content measuring 16.2% (Figure 9(a)) and S content at 3% (Figure 9(b)). On weekends, Ca content increased to 33% (Figure 9(d)), while S content was 4% (Figure 9(d)).

The predominance of sulfur (S), calcium (Ca), and oxygen (O) on weekdays, and sulfur (S), sodium (Na), potassium (K), and oxygen (O) on weekends, suggests that the faceted morphology of the particles is influenced by emissions from coal power plants (Li et al., 2016). During weekends, the presence of soil dust particles, characterized by S-rich components along with K and Na from soot, indicates contributions from heavy-duty vehicles (Santoso et al., 2014) and biomass burning (Fatima et al., 2022). These particles exhibited near-spherical, amorphous, and aggregate forms. The elemental composition of $PM_{2.5}$ in Tangerang, including additional details, is further depicted in Figure 11(c).

3.3 Elemental characterization of $PM_{2.5}$ by ED-XRF

3.3.1 Elemental composition of $PM_{2.5}$ in Bandung

The elemental concentration of $PM_{2.5}$ at the Bandung site was calculated following the methodology explained in section 2.3.2. The elemental composition of $PM_{2.5}$ is illustrated in Figure 11(a) and is consistent with the results obtained from EDX analysis. The data reveals that both weekdays and weekends show significant concentrations of various elements on the Teflon filters, with sulfur (S), potassium (K), iron (Fe), sodium (Na), aluminum (Al), and silicon (Si) being the predominant elements. Additionally, heavy metals such as lead (Pb), zinc (Zn), titanium (Ti), and copper (Cu) were detected in the $PM_{2.5}$ samples. The major concentrations of Al, Si, and Ca were regarded as soil dust sources (Santoso et al., 2008b).

In Bandung, the elevated levels of sulfur (S) and potassium (K) are linked to open burning and biomass combustion, as local residents frequently burn trash and wood for cooking and heating. Notably, the sulfur content is higher on weekends (3,256 ng/m^3) than on weekdays (1,798 ng/m^3), which is likely due to increased local vehicle emissions around the sampling site, as detailed in the gravimetric analysis of Bandung. Additionally, natural sources such as the Tangkuban Perahu volcano, located approximately 30 km north of Bandung in the Lembang area, may also contribute to the elevated sulfur levels (Santoso et al., 2008b). Interestingly, despite the local government's prohibition of leaded gasoline since July 2006, which led to a significant reduction in Pb concentrations over the years (from 12.69 ng/m^3 in 2012) (Santoso et al., 2014). The $PM_{2.5}$ in Bandung still shows a relatively high lead content of 100 ng/m^3. This anomaly suggests that further investigation into the sources and persistence of high Pb levels in Bandung is warranted.

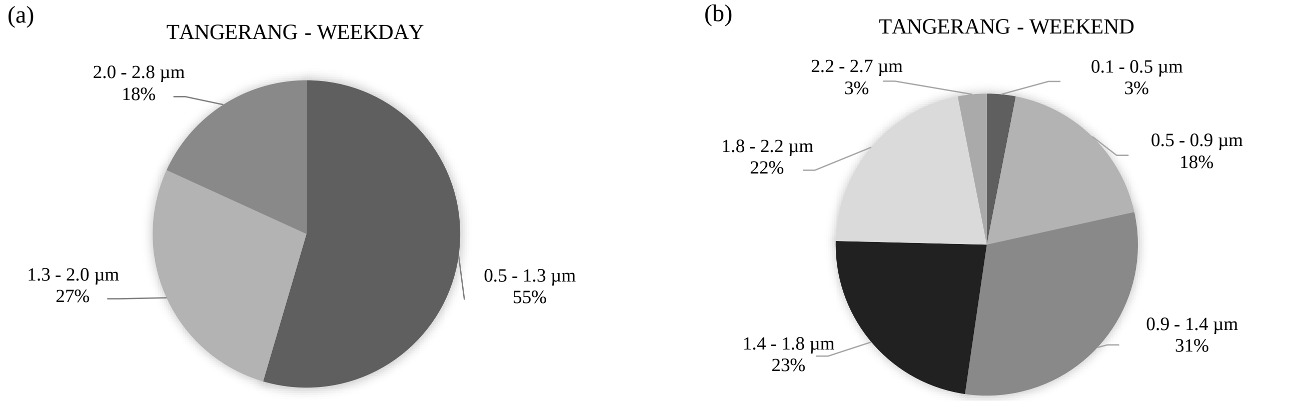

Figure 10. Size distribution of $PM_{2.5}$ collected on weekdays with total selected particles (n=142) (a) and weekends (n=91) (b) in Tangerang site

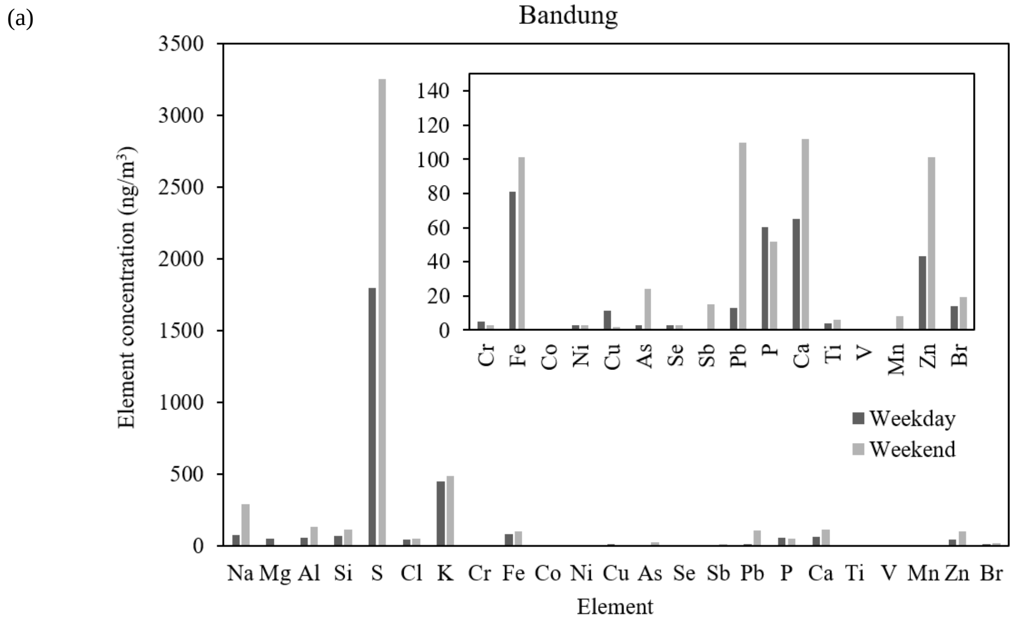

Figure 11. The elemental concentration of $PM_{2.5}$ in Bandung (a), Jakarta (b), and Tangerang (c) on weekdays and weekends with the highest $PM_{2.5}$ concentration during the sampling period.

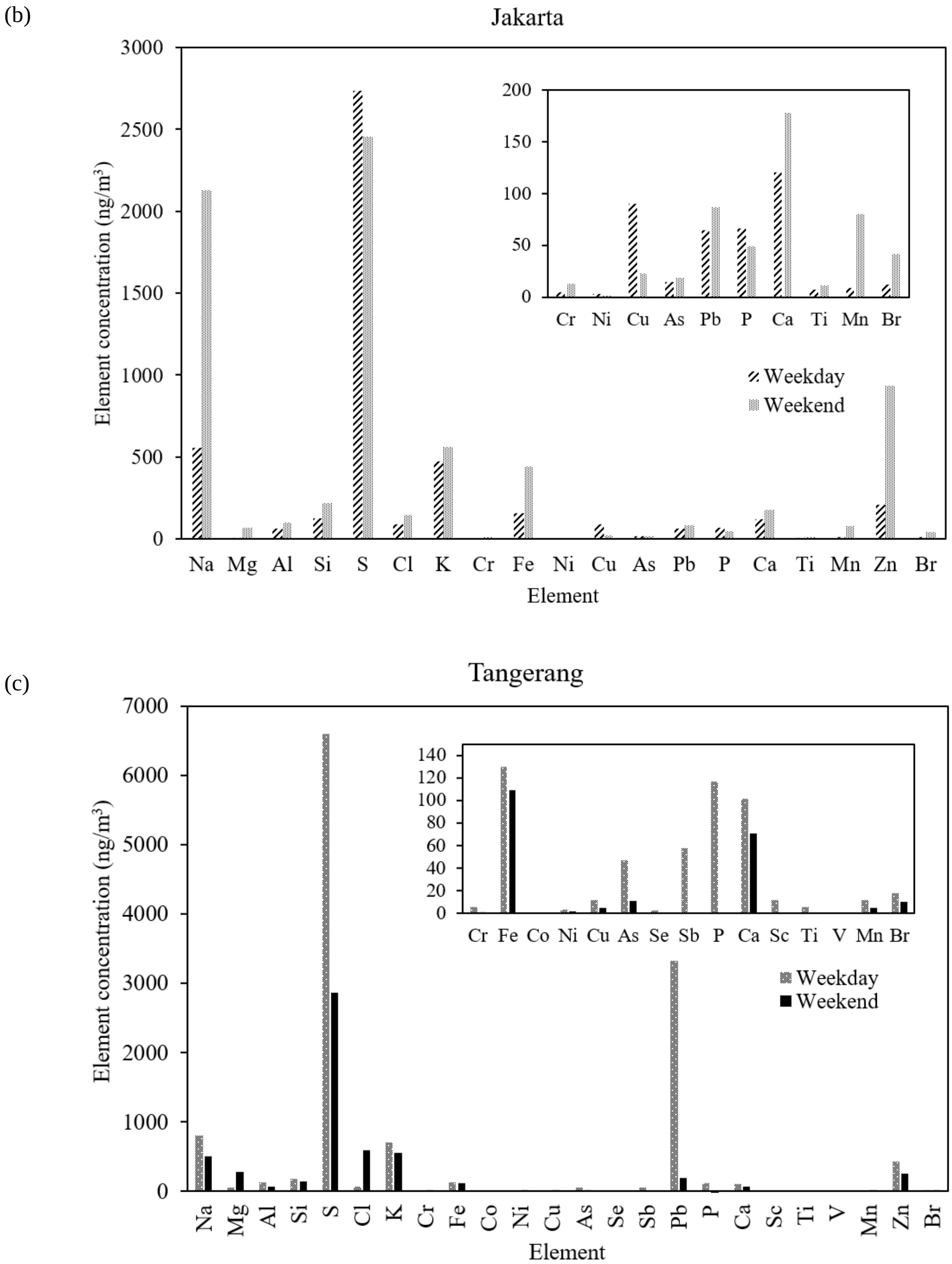

Figure 11. The elemental concentration of $PM_{2.5}$ in Bandung (a), Jakarta (b), and Tangerang (c) on weekdays and weekends with the highest $PM_{2.5}$ concentration during the sampling period (cont.).

3.3.2 Elemental composition of $PM_{2.5}$ in Jakarta

The elemental concentration of $PM_{2.5}$ at the Jakarta site was calculated following the methodology explained in section 2.3.2. The elemental composition is detailed in Figure 11(b). The analysis indicates that sulfur (S), sodium (Na), potassium (K), zinc (Zn), and iron (Fe) are the most concentrated elements. Other elements, including aluminum (Al), magnesium (Mg), silicon (Si), copper (Cu), arsenic (As), lead (Pb), phosphorus (P), calcium (Ca), manganese (Mn), and bromine (Br), were detected but in lower concentrations (below 300 ng/m³). The high levels of Al, Si, Ca, and Fe are attributed to soil dust sources (Santoso et al., 2013). Sulfur concentration is notably elevated at 2,734 ng/m³ on weekdays, likely due to the high density of motor and diesel vehicles in the area (Santoso et al., 2013), as discussed in the gravimetric section. The reduced sulfur levels on weekends are associated with decreased vehicle activity due to the closure of offices. Arsenic (As) is present, possibly originating from coal-burning power plants located approximately 11 km from the sampling site. The medium levels of sodium

(Na) and chlorine (Cl) are attributed to sea salt, as evidenced by the presence of NaCl minerals in Jakarta's $PM_{2.5}$ morphology (Hopke et al., 2020), as evidenced by the presence of NaCl mineral in Jakarta's morphology in Figure 7. Although lead (Pb) is detected, its concentration in Jakarta is lower compared to Tangerang, suggesting that Pb may be transported from Tangerang by wind (Santoso et al., 2011). The presence of lead (Pb) and copper (Cu) on weekdays is likely due to brake wear in urban environments (Khobragade and Ahirwar, 2023), while manganese (Mn) and zinc (Zn) are typically associated with crustal sources (Liu et al., 2018).

3.3.2 Elemental composition of $PM_{2.5}$ in Tangerang

The elemental concentration of $PM_{2.5}$ at Tangerang site was analyzed according to the methodology explained in section 2.3.2. Figure 11(c) reveals that the highest concentrations of elements include sulfur (S), lead (Pb), sodium (Na), potassium (K), and zinc (Zn), with variations observed when compared to Jakarta and Bandung. The high Sulfur concentrations observed on weekdays are primarily attributed to emissions from heavy-duty vehicles, given that the sampling site is situated along a busy inter-provincial route. On weekends, the reduction in vehicular traffic leads to a notable decrease in Sulfur levels, approximately 50% lower than on weekdays. Additionally, the high Sulfur content in Tangerang is further linked to emissions from coal-burning power plants located around 40 km north of Tangerang, as evidenced by the presence of arsenic (As) from these combustion sources (Wang et al., 2018). The exceptionally high lead (Pb) concentration in Tangerang is attributed to non-formal lead smelting industries in the vicinity (Santoso et al., 2011). This source of Pb contributes significantly to the elevated levels observed at the site.

4. CONCLUSION

CONCLUSIONThe morphological analysis reveals distinct characteristics in $PM_{2.5}$ particles across different sites. At Bandung, $PM_{2.5}$ particles exhibit amorphous surfaces with irregularly agglomerated and flaky shapes. Jakarta's $PM_{2.5}$ particles are noted for their semi-crystalline, tabular, and elongated forms, along with puff-like structures. In Tangerang, the particles are primarily irregularly faceted and agglomerated. EDX spectra from all sites indicate the presence of carbonate minerals (C, O, Ca) in $PM_{2.5}$. Calcium-rich particles are specifically observed in Bandung, while Sulfur-rich particles are detected in Jakarta and Tangerang. Additionally, fingerprint elements corresponding to specific pollution sources have been successfully identified through ED-XRF analysis. The integration of SEM-EDX and ED-XRF methodologies provides a comprehensive understanding of the $PM_{2.5}$ composition, offering valuable insights into the particle characteristics. However, further analysis could be conducted to deeply analyze each pollution source at each site.

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