Nuclear analytical techniques for identification of elemental composition of fine and coarse airborne particulate matter Collected in Bandung, Indonesia

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Abstract. Air pollution has received serious attention from both the government and the public in Bandung, Indonesia. Nuclear analytical techniques (NAT) such as X-ray fluorescence (XRF) have been implemented for the quantification of environmental pollutants, especially airborne particulate matter (APM). Sampling for fine APM (PM\textsubscript{2.5}) and coarse APM (PM\textsubscript{2.5-10}) have been conducted in Bandung using a GENT sampler, once a week for 24 hrs from 2015 to 2017. The samples were then analyzed for their mass, black carbon (BC), and elemental composition using gravimetric, smoke stain reflectometer, and ED-XRF, respectively. The results obtained indicate that the annual average of PM\textsubscript{2.5} is in the range of 17.85-20.90 \( \mu \text{g/m}^3 \) which has exceeded the Indonesian annual standard (15 \( \mu \text{g/m}^3 \)). While for PM\textsubscript{10} it is still below the standard. Compared to PM\textsubscript{2.5} and PM\textsubscript{2.5-10} in 2002-2004, the concentration has increased by around 35% and 29%, respectively. This situation needs serious attention especially because of the adverse effects of PM\textsubscript{2.5} on human health. The average BC fraction at PM\textsubscript{2.5} is around 19%. In this paper, chemical composition, annual concentration trends, and correlations between elements will be discussed in detail including the advantages and disadvantages of the NATs method. For the next stage, this information can be used to identify sources of pollutants.

1. Introduction
Increased urbanization and various economic activities such as transportation and industry have resulted in air pollution. Urban air quality in Indonesia, including Bandung, has shown a declining trend in the past two decades. This condition occurs because the source of pollutants has exceeded the carrying capacity of the environment so it cannot be neutralized naturally. This air pollution has a significant impact on human health, ecosystems, climate change and global warming [1]. The health risks associated with air pollution in urban areas have generally attracted a lot of attention in recent decades [2]. The main parameter of air pollution that has a significant impact on health is particulate matter (PM). Particulates present in the atmosphere are generally up to 50 \( \mu \text{m} \) in size, which varies in existence depending on size. Air particulates that are less than 2.5 \( \mu \text{m} \) (PM\textsubscript{2.5}), called fine particulates, are very dangerous because they can penetrate into the deepest parts of the lungs and heart, causing health problems including acute respiratory infections, lung cancer, cardiovascular disease and even death [3]. Fine particulates matter (PM\textsubscript{2.5}) are estimated to have a large contribution to mortality due to health problems related to air pollution. PM\textsubscript{2.5} generally consist of micro-sized particles originating from...
anthropogenic sources such as motor vehicles, biomass combustion, and fuel combustion. Besides PM$_{2.5}$, also known as PM$_{10}$ which is a particulate matter that is less than 10 µm in size, whereas total suspended particulate (TSP) is all suspended substances that are generally less than 50 µm in size.

One of the critical aspects in air quality management and air pollution control is the implementation of continuous air quality monitoring. In Indonesia, including Bandung, the identification of PM$_{2.5}$ and PM$_{10}$ are still very rare, because it requires advanced analytical techniques that are able to detect the concentrations of elements at the nanograms per cubic meter level. Nuclear analytical techniques (NATs) such as neutron activation analysis (AAN), X-ray fluorescence (XRF), and proton X-ray emission (PIXE) are suitable techniques because they have high sensitivity, simultaneous, selective, and the detection limit reaches the part per billion (ppb) level. NATs have been widely applied in the characterization of APM [4]. The advantages of NATs are expected to be a new breakthrough in the environmental field.

Air quality in Bandung in 2002-2004 was reported by Santoso (2008) which discussed in detail the chemical composition of the fine and coarse APM, including the identification of the source of the pollution. Motor vehicles, biomass burning, secondary sulfate, soil, and road dust are sources of pollutants. The results obtained also indicate that biomass burning contributes around 20% [5]. Kim Oanh (2006) reports the results of particulate monitoring conducted in 6 cities in Asia, including in Bandung. PM$_{2.5}$ and PM$_{10}$ concentrations in Bandung in the dry season reached 53 and 83 µg/m$^3$ respectively while in the rainy season they reached 38 and 62 µg/m$^3$, respectively [6]. Lestari 2009 reported that different source factors were obtained in the dry and wet season in Bandung. Secondary aerosols, vehicle emissions, electroplating industry, and biomass burning are sources for fine particulates. While the sources of coarse particulates during the dry season are the electroplating industry, aged sea salt, volcanic dust, soil dust, and lime dust. In the wet season, vehicle emissions and secondary aerosols are the main sources of fine particulates [7]. The objective of the current study is to provide an overview of the current status of Bandung’s air quality, including the level of mass concentration and elemental composition of the APM and its comparison with similar data from 2002-2004.

2. Methods

2.1. Sampling

Sampling of ambient PM$_{2.5}$ and PM$_{10}$ were carried out on the roof of the office of West Java Provincial Environmental Protection Agency (S 06°55’10.9’’, E 107°36’39.1’’, Figure 1), using the GENT dichotomous sampler. In each sampling, 2 types of Nucleopore polycarbonate filters were used, namely fine and coarse filters, each of which had porous of 0.4 and 8 µm, respectively. Sampling was carried out at an altitude of about 20 m above the ground, for 24 hours and once a week during 2015-2017. This sampling produces APM samples with particulate sizes up to 2.5 µm in fine filters and between 2.5-10 µm in coarse filters.

2.2. Particulate matter analysis

Determination of mass concentration (PM$_{2.5}$ and PM$_{10}$) was done by gravimetric method using Mettler Toledo micro balance. Fine APM (PM$_{2.5}$) and coarse APM (PM$_{2.5-10}$) are obtained from the weighing of the sample weights on the fine and coarse filter, respectively. While, PM$_{10}$ is obtained from the total mass of particulates from fine and coarse filters. Before weighing, the filter must be conditioned in a room with humidity between 45-55% and a temperature of 18-25°C.

2.3. Black carbon analysis

Determination of carbon in the filter is based on the process of light reflection. The absorption and reflection of visible light by airborne particulate matter in the filter depends on particle concentration, density, refractive index and size. Because in atmospheric particles, BC is a component that has the highest absorption ability, so it is assumed that the absorption of light in the filter is caused by BC. The
The light reflection method is used in BC measurements, where the light coming from a light source is transmitted through the annular photocell to the surface of the sample filter, then the light is reflected back to the photocell, the length of the light path is twice the length of the transmission path. The amount of light absorbed will be proportional to the amount of BC in the filter. In this study BC was determined using a Digital Smoke Stain Reflectometer. Measurement of BC in the filter was done using the assumption that the average coefficient of particle mass absorption is 5.7 m$^2$/g [8].

![Figure 1](image.png)

**Figure 1.** Sampling site of airborne particulate matter in Bandung, Indonesia (source: Google Maps [9]).

### 2.4. Multi elemental analysis

Determination of the elements contained in airborne particulate matter is an important step in identifying sources of air pollutants. APM with a mass of less than 1 mg are very small mass to be analyzed using conventional methods. Therefore, nuclear analytical techniques which have high sensitivity, simultaneous, selective, and a very low detection limits are needed. This is the main reason for selecting the use of nuclear-based analytical methods for elemental characterization in APM. This method is suitable for analysis of relatively large sample sizes, sometimes reaching hundreds of filters and a small sample weight per filter; 100 - 600 µg. The method used for elemental analysis has been validated using SRM (Standard Reference Material) Air Particulate on Filter Media. The samples were analyzed using ED-XRF with 9 secondary targets (Fe, CaF$_2$, Ge, Zr, CeO$_2$, Mo, Ag, Al and one Barkla polarizing target (Al$_2$O$_3$)). Calibration was carried out using a standard single element MicroMatter, whereas for method validation, NIST SRM 2783 Air Particulate Matter was used in the filter media. Except for light elements, the ratio between measurement results and certificate values is in the range of 0.92-1.09% [4]. XRF analysis methods including the way of calibration and validation are explained in detail by [4,10].

### 3. Results and Discussions

#### 3.1. Particulate Matter Concentration

The results of 24-hour average mass fraction of the PM$_{2.5}$ and PM$_{2.5-10}$ in Bandung from 2015 to 2017 are shown in Figure 2, while the annual averages are presented in Table 1. The results obtained showed that the annual average concentration of 2015 (20.9 µg/m$^3$) was higher than in 2016 (17.85 µg/m$^3$) and 2017 (18.94 µg/m$^3$), due to the El Nino effect on 2015, this effect occurs periodically every 5 to 7 years which causes a longer dry season. In Figure 2 and Table 1, it can be seen that in general the concentration
of coarse particulate matter is higher than that of fine particulate, this is probably because natural pollutant sources have a relatively higher contribution than anthropogenic sources.

![Figure 2. 24-hour averages for PM$_{2.5}$, PM$_{2.5-10}$ and PM$_{10}$ ($\mu$g/m$^3$) in Bandung, Indonesia.](image)

The average level of mass and the major components of fine and coarse APM in Bandung, 2015-2017 are shown in Table 2, while the comparison with 2002-2004 data is presented in Table 3. Comparison with 2002-2004 data was chosen because the sampling and analysis methods in both periods (2002-2004 and 2015-2017) were carried out in the same procedure. The concentrations of PM$_{2.5}$, PM$_{2.5-10}$ and PM$_{10}$ for 2002-2004 were 14.03, 17.64 and 31.67 $\mu$g/m$^3$, respectively. Compared to 2002-2004 data, after 13-15 years, mass concentrations of fine and coarse APM increased by 49%, 27%, 35%, and 49%, 38%, 6% for 2015, 2016, 2017, respectively. An increase in PM$_{2.5}$ level is expected due to population growth, number of motorized vehicles and industrial activities. Based on data from the Central Bureau of Statistics, the total population of the city of Bandung in 2010 was 2,394,873; while in 2017 there were 2,412,458. In Indonesia, the increase in vehicles for 10 years reached 3 to 4 times. The number of motorcycle vehicles in 2002 was 17,002,130 and in 2012 it became 76,381,183 (increase by more than 400%), while the car vehicles from 3,403,433 to 10,432,259 (increase by more than 300%). In Bandung, the increase in the number of vehicles also occurs with the same tendency as the national level, this is one of the main reasons for the increase of PM$_{2.5}$ in addition to the increase of population and industrial activity in the city of Bandung. The increase of coarse APM in 2015 and 2016 which is reached more than 35% is most likely due to the large number of road infrastructure developments including the fly over in the city of Bandung. Increasing the concentration of particulates needs serious attention, especially for fine particulates which are very dangerous to human health because they are able to penetrate deeply into the lung. In addition, the PM$_{2.5}$ generated from the GENT sampler tends to underestimate because the sample collection point is closer to 2.2 $\mu$m and not 2.5 $\mu$m [11]. Therefore, the measured PM$_{2.5}$ level should be greater than what is observed. PM$_{2.5}$ concentrations that has exceeded the annual standard of either the Indonesian or WHO standards will pose a major health risk to the surrounding community. For this reason, an appropriate strategy is needed to reduce PM$_{2.5}$ concentrations in Bandung.
Table 1. Annual averages for PM$_{2.5}$, PM$_{2.5-10}$ and PM$_{10}$ (µg/m$^3$) in Bandung, Indonesia

|       | 2015       | 2016       | 2017       |
|-------|------------|------------|------------|
|       | Average    | SD         | Min        | Max        | Average    | SD         | Min        | Max        | Average    | SD         | Min        | Max        |
| PM2.5 | 20.90      | 5.31       | 8.52       | 30.01      | 17.85      | 6.57       | 6.21       | 30.16      | 18.94      | 10.01      | 4.51       | 55.71      |
| PM2.5-10 | 26.29     | 7.84       | 15.36      | 43.10      | 24.42      | 6.90       | 11.71      | 37.72      | 18.72      | 7.41       | 5.40       | 38.99      |
| PM10  | 47.19      | 11.50      | 23.87      | 68.17      | 42.27      | 10.94      | 20.74      | 64.97      | 37.66      | 14.39      | 11.87      | 80.06      |

3.2. Black carbon concentrations

Black carbon concentrations in PM$_{2.5}$ for the 2015-2017 period are presented in Figure 2 and the average BC is shown in Table 3. BC is generally emitted into the atmosphere due to incomplete combustion. To avoid inaccuracies in reflectance and absorbance for large particle sizes, the BC measurements are only applied to fine APM. Black carbon is a form of impurity from carbon that results from incomplete combustion of fossil fuels or biomass burning. Black Carbon has a complex effect on climate change, which is warming of the atmosphere as well as cooling the earth's surface. Reducing BC pollution is believed to be a good strategy in reducing or minimizing the effects of global warming.

In Figure 2, BC concentrations are in the range of 1.1 to 5.7 µg/m$^3$ with an average of 3.6 µg/m$^3$ which contributes 19% to PM$_{2.5}$. The main sources of BC are anthropogenic combustion sources including biomass combustion, motor vehicles and industrial emissions such as coal combustion. Compared to 2002-2004 data, the average BC level has decreased by 15%. This indicates the reduction in BC emission sources originating from both motor vehicles and biomass burning. This is in line with various regulations that have been issued by the local government related to the prohibition of open burning (biomass burning), as well as improving the quality of motor vehicles in order to reduce emissions, one of which is through the implementation of vehicle emission tests. The average BC in Bandung (3.6 µg/m$^3$) is lower compared to Kuala Lumpur, Malaysia (4.5 µg/m$^3$) [12], but higher than the results found in Mumbai India (3.58 µg/m$^3$) and Ulaanbaatar Mongolia (3.29 µg/m$^3$) [13].

![Figure 3. The 24-hour concentration of BC (µg/m$^3$) in Bandung, Indonesia](image)

3.3. Multi elemental concentrations

Multi-element concentrations measured by ED-XRF with 9 secondary standards are presented in Table 2. The results obtained indicate that the chemical composition of fine particulates is dominated by S and BC which are anthropogenic sources, while coarse particulates are dominated by crustal elements such as Si, Al, Ca, and Fe. A good correlation and the same trend between these elements shown in Figure 3.
and Figure 4, can then be used to estimate the source of these particulate pollutants. A good correlation between S and BC can indicate coming from vehicle sources or industrial activities, while the Al, Si, and Ca correlations originate from soil dust or road dust.

Table 2. The mean and standard deviations of mass and major components of fine and coarse APM (ng/m³) in Bandung, 2015-2017

|                | Fine fraction (PM₂.₅) | Coarse fraction (PM₁₀) |
|----------------|-----------------------|------------------------|
|                | Average  | SD    | Min   | Max   | Average | SD    | Min   | Max   |
| Mass           | 19019    | 7798  | 4507  | 55712 | 22795   | 7981  | 5403  | 43105 |
| BC             | 3609     | 962   | 1069  | 5681  |          |       |       |       |
| Na             | 262.9    | 229.0 | 5.7   | 933.3 | 220.9   | 172.8 | 12.2  | 858.3 |
| Mg             | 26.2     | 21.4  | 1.4   | 119.4 | 141.0   | 55.7  | 20.8  | 300.7 |
| Al             | 70.7     | 39.8  | 1.7   | 168.9 | 606.4   | 215.9 | 48.5  | 1215.2|
| Si             | 129.4    | 49.6  | 32.0  | 266.9 | 1044.6  | 356.9 | 198.8 | 1935.9|
| S              | 1226.1   | 591.8 | 166.5 | 2868.4| 623.1   | 255.7 | 133.0 | 1326.3|
| K              | 197.3    | 86.0  | 48.1  | 571.9 | 155.2   | 55.8  | 32.5  | 351.3 |
| Ca             | 97.6     | 33.8  | 25.5  | 230.7 | 888.3   | 294.4 | 221.9 | 1648.8|
| Ti             | 7.7      | 2.8   | 2.0   | 18.6  | 50.8    | 17.5  | 8.4   | 87.4  |
| V              | 0.5      | 0.3   | 0.01  | 1.5   | 1.27    | 0.74  | 0.01  | 3.89  |
| Cr             | 0.9      | 0.6   | 0.01  | 2.3   | 3.3     | 1.6   | 0.4   | 10.4  |
| Mn             | 4.9      | 5.7   | 0.4   | 51.7  | 17.1    | 6.2   | 3.9   | 42.2  |
| Fe             | 112.2    | 54.4  | 27.8  | 516.5 | 735.1   | 249.0 | 144.8 | 1279.9|
| Ni             | 1.09     | 0.55  | 0.05  | 2.52  | 1.28    | 0.71  | 0.01  | 2.94  |
| Cu             | 2.87     | 2.40  | 0.02  | 14.52 | 5.41    | 4.17  | 0.29  | 30.55 |
| Zn             | 34.3     | 15.9  | 8.7   | 97.5  | 35.7    | 18.5  | 7.7   | 115.8 |
| Pb             | 19.0     | 16.0  | 0.3   | 82.8  | 12.7    | 12.8  | 0.6   | 75.1  |

Compared with 2002-2004 data (Table 3), it can be shown that in 2015-2017, BC concentrations have decreased, but sulfur (S) and potassium (K) concentrations have increased. In general, S is a key element of vehicle and industrial emissions, while K is from biomass burning. The increase in S concentration can be due to the increasing use of coal both for power plants and industries as well as the high concentration of Sulfur in vehicle fuels. Meanwhile, the increase in K is probably due to open burning activities that are still being carried out in Bandung and its surroundings, especially to reduce the volume of household waste. The increase in Zn and Mn elements also needs attention because these elements are indicators of metal industry pollutants. Meanwhile, reducing the Pb concentration can show the success of the unleaded gasoline program implemented in Indonesia since July 2006. Even though unleaded gasoline has been applied, the problem of heavy metal lead pollution in ambient air still occurs as in Tangerang Indonesia [14] and Dhaka Bangladesh [15]. In the coarse fraction APM, Si is a major chemical species followed by other elements namely Ca, Al and Fe. These elements (Si, Ca, Al, Fe) are crustal elements which generally have a significant contribution to the coarse APM. These APM data can then be used to determine multi-element correlation which is very useful for estimating pollutant sources. For example, if there is a good correlation between S against BC, K, Al, and Si, it can be used as an indicator of emissions from brick kiln sources around Bandung. These data can then be used to estimate pollutant sources using receptor model. The results obtained from this study will then be used to determine the type of pollutant source both qualitatively and quantitatively.
Figure 4. (a) BC and S concentrations of PM$_{2.5}$ (b) Al, Si and Ca concentrations of PM$_{2.5-10}$. 
Table 3. The mean and standard deviations of mass and major components of fine and coarse particulate (ng/m³) in Bandung during 2015-2017 and 2002-2004

|          | 2015-2017          | 2002-2004[5]         |
|----------|---------------------|----------------------|
|          | PM$_{2.5}$ | PM$_{2.5:10}$ | PM$_{2.5}$ | PM$_{2.5:10}$ |
|          | Average | SD         | Average | SD         | Average | SD         | Average | SD         |
| Mass     | 19019   | 7798       | 22795   | 7981       | 14030   | 6860       | 17640   | 9420       |
| BC       | 3609    | 962        | 229.03  | 172.76     | 76.49   | 48.79      | 260.36  | 209.75     |
| Na       | 262.87  | 39.85      | 606.45  | 215.89     | 89.69   | 88.61      | 727.75  | 459.82     |
| Mg       | 26.22   | 140.97     | 45.57   | 55.71      | 150.03  | 73.06      | 1049    | 684        |
| Al       | 1226    | 592        | 256     | 55.81      | 123.78  | 95.37      | 122.25  | 54.29      |
| Si       | 97.62   | 33.79      | 294.36  | 17.47      | 52.72   | 25.14      | 583.58  | 240.31     |
| S        | 7.72    | 50.84      | 2.77    | 17.47      | 9.84    | 14.60      | 43.96   | 21.40      |
| K        | 197.28  | 155.22     | 86.01   | 55.81      | 123.78  | 95.37      | 122.25  | 54.29      |
| Ca       | 97.62   | 33.79      | 888.35  | 294.36     | 52.72   | 25.14      | 583.58  | 240.31     |
| Ti       | 49.55   | 17.13      | 5.65    | 6.22       | 2.09    | 1.89       | 13.22   | 9.96       |
| V        | 249.04  | 356.86     | 0.47    | 0.74       | 0.89    | 0.99       | 1.96    | 1.57       |
| Cr       | 59.25   | 45.39      | 0.86    | 1.61       | 6.92    | 14.49      | 14.14   | 14.14      |

4. Conclusions
The annual average of PM$_{2.5}$ in Bandung during 2015-2017 is in the range of 17.85-20.9 µg/m³ and has exceeded Indonesian standards (15 µg/m³). Compared to 2002-2004 data, PM$_{2.5}$ level has increased by 35%. Although the annual average of PM$_{10}$ is still below the standard, but PM$_{2.5}$ levels that have exceeded the standard and tend to increase from time to time, serious attention and appropriate strategies are needed in order to reduce health risks for the community. The characteristics of chemical composition both in fine and coarse APM can be used as a preliminary study in identifying the source of pollutants as well as to assess the effectiveness of an air quality program.

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