Coal and coal ash characteristics to understand mineral transformation: A case study from Senakin coal field, Indonesia

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Abstract. Coal ash is a material from coal combustion and recently is an economical source for the extraction of valuable elements such as rare earth elements and Yttrium (REY) and base metals. Eight coal samples from Senakin coal field were collected to identify their mineralogy composition in raw coal and coal ash. The ashing was carried out on coal samples at temperature 1000°C for 1 hour. The coal and coal ash samples were made polish section to identify the organic and inorganic constituents. In addition, the minerals in coal sample were identified by X-ray diffraction analysis. From this study, organic constituent consist of vitrinite (60 – 71%), liptinite (22 – 33%) and inertinite (5 – 9%). Minerals found in coal are kaolinite (25,78 – 46,90), pyrite (24,16 – 38,38%), quartz (6,07 – 12,80%), gypsum (4,81 – 10,34%), chlorite (2,56 – 8,40%), jarosite (0,72 – 4,63%), hematite (0,96 – 3,81%), calcite (0,37 – 5,07%), Mg-calcite (0,11 – 3,20%), dolomite (1,35 – 3,55%), and siderite (1,22%). Coal ash is composed of organic and inorganic components. Organic components found is unburned coal (3,09 – 8,55%) derived from maceral which does not burn out, mainly inertinite group. While inorganic components found are ferrous oxide mineral (40,36 – 54,55%), quartz (24,55 – 33,64%), mullite (5,09 – 7,82%), cenosphere (7,45 – 10,91%), pleiosphere (1,64 – 2,36%), pyrite (0,18 – 1,27%), and spinel (0,18 – 1,09%). Fe-oxide minerals are interpreted derived from Fe-bearing minerals in coal samples such as pyrite, hematite, and siderite. Kaolinite which is the dominant mineral in coal is transformed to cenosphere, pleiosphere, and mullite. Quartz in coal ash derived from quartz in the coal sample because ashing temperature does not exceed the melting temperature of quartz. Characterization of the coal ash component can be used as a reference for the extraction method of valuable elements.
1. Introduction
Coal has been a major energy supply in many countries including Indonesia in which 30% of the total energy will be occupied by coal in 2025. The need of coal has not been a problem because Indonesia owns abundance coal deposits of about 37.6 billion tonnes [1]. Spreading from Sumatera to Papua, the characteristic of Indonesia’s coal deposit is varied. Coal characteristic is one of key factors to determine the fly ash and bottom ash (FABA) characteristic beneficial for further utilization. Many studies have been conducted related to FABA utilization and coal characterization related to it, i.e., cenospheres characteristic and recovery [2-5], valuable elements [6-9], geopolymer [10-11], etc. However, coal characterization in Indonesia has not been mapped covering all coal deposit area, including Senakin at where coal deposit is of about 172 million tonnes [12]. Thus study on Senakin coal characterization in indeed important to be conducted in order to estimate the FABA characteristic that the recovery techniques of valuable materials can be predicted and designed appropriately.

2. Materials and Methods
Field observation and coal sampling were done in an active open pit mine in Sebuli area, Senakin coal field, Southeast Borneo (Figure 1). Eight coal samples (Table 1) were collected from main coal seam by using a ply-by-plly sampling method. Each ply sample was identified individually on the basis of coal lithotype and megascopic appearance referring to Diessel [13] classification system.

![Figure 1. Maps showing the sampling location from main coal seam, Sebuli area, Senakin coal field, Indonesia.](image)

Table 1. List of samples collected from main coal seam, Sebuli area, Senakin coal field, Indonesia.

| Seam                  | Sample | Lithology   |
|-----------------------|--------|-------------|
| Main upper coal seam  | MU-4   | Bright coal |
|                       | MU-6   | Bright coal |
|                       | MU-8   | Bright coal |
|                       | MU-10  | Bright coal |
| Main middle coal seam | MM-7   | Bright coal |
|                       | MM-6   | Bright coal |
|                       | MM-4   | Bright coal |
| Main lower coal seam  | ML-1   | Bright coal |
To obtain information about mineralogy in raw coal and transformation organic and inorganic components several analytical techniques were applied. They were:

2.1. Proximate analysis
Proximate analysis was conducted to evaluate ash yield, volatile matter, moisture, and fixed carbon. This analysis was conducted based on the [14] Standard Test Method for proximate analysis of coal and coke.

2.2. Petrography
Coal petrography was done based on [15] Standard Test Method for microscopical determination of the maceral compositions of coal. Maceral classification refers to ICCP classification 1994 [16-18]. Identification and classification of the organic and inorganic constituents of the coal ash were conducted using the classification described by Hower [19].

2.3. Mineralogy
Minerals identification in coal samples were conducted using petrography and X-Ray diffraction (XRD). XRD analysis was performed with 2θ from 2° to 60° in the analytical laboratory of the Geological Engineering Department, Universitas Gadjah Mada, Yogyakarta, Indonesia. There are 3 treatments of coal samples for XRD analysis, raw coal, ashing 370°C for 8 hours, and ashing 370°C for 14 hours.

3. Results

3.1. Raw Coal Characteristics
The raw coal from main coal seam have ash yield range 17.89 – 56.04 wt.%, moisture contents of 4.18 – 4.94 wt.%, volatile matter 27.45 – 40.86 wt.%, and fixed carbon contents of 11.85 – 39.06 wt.% (Table 2). Petrography analysis of coal polish section show that the samples dominated by vitrinite (63 – 71%, Table 3). Collotelinite and telnite are the most vitrinite maceral group and it is present in all the coal samples. The liptinite content range between 20 – 29%; sporinite, cutinite, and resinite are the most common liptinite group macerals observed. The inertinite content in in the 4 – 7% range and most is present as funginite and semifusinite. The maceral content, mineral matter, and ash yield of coal samples are presented in Table 3.

Table 2. Proximate analysis (wt.%) of raw coals from main coal seam, Sebuli area, Senakin coal field, Indonesia.

| Sample | M_{adb} | A_{adb} | V_{adb} | C_{adb} |
|--------|---------|---------|---------|---------|
| MU-4   | 4.22    | 19.25   | 37.47   | 39.06   |
| MU-6   | 4.43    | 25.71   | 36.07   | 33.79   |
| MU-8   | 4.94    | 37.20   | 33.70   | 24.16   |
| MU-10  | 4.67    | 56.04   | 27.45   | 11.85   |
| MM-7   | 4.18    | 20.80   | 40.54   | 34.48   |
| MM-6   | 4.19    | 23.29   | 38.44   | 34.08   |
| MM-4   | 4.42    | 17.89   | 40.63   | 37.06   |
| ML-1   | 3.30    | 19.92   | 40.86   | 35.92   |

M, moisture; A, ash yield; V, volatile matter; C, carbon; adb, air-dried basis
Table 3. Maceral content, mineral matter, and ash yield of raw coals from main coal seam, Sebuli area, Senakin coal field, Indonesia.

| Sample | Vitrinite (%) | Liptinite (%) | Inertinite (%) | Mineral matter (%) | Ash Yield (%) |
|--------|---------------|---------------|----------------|--------------------|---------------|
| MU-4   | 64            | 26            | 7              | 3                  | 19.25         |
| MU-6   | 64            | 29            | 5              | 2                  | 25.71         |
| MU-8   | 70            | 20            | 6              | 4                  | 37.20         |
| MU-10  | 70            | 21            | 5              | 4                  | 56.04         |
| MM-7   | 69            | 24            | 4              | 3                  | 20.80         |
| MM-6   | 71            | 23            | 4              | 2                  | 23.29         |
| MM-4   | 68            | 23            | 4              | 5                  | 17.29         |
| ML-1   | 63            | 27            | 6              | 4                  | 13.98         |

The coal petrography shows the main minerals in the coal are pyrite, clay minerals, and quartz (Figure 2). Pyrite is the most dominant in eight samples with abundance 45.4 – 76.9%. Clay minerals are present range from 11.1 – 36.3%. While quartz present in the 7.7 – 18.2% range. XRD analysis was able to identify minerals that were not identified in petrography analysis. The result of petrography and XRD analysis then normalized to determine mineralogy in coal samples. Minerals found in coal are kaolinite (25.78 – 46.90), pyrite (24.16 – 38.38%), quartz (6.07 – 12.80%), gypsum (4.81 – 10.34%), chlorite (2.56 – 8.40%), jarosite (0.72 – 4.63%), hematite (0.96 – 3.81%), calcite (0.37 – 5.07%), Mg-calcite (0.11 – 3.20%), dolomite (1.35 – 3.55%), and siderite (1.22%).

3.2. Coal Ash Characteristics

3.2.1. Coal Ash Organic Components. Petrographic analysis of polish section shows that coal ash is also composed of organic material in the form of unburned coal (UC) (Figure 3) or maceral that does
not burn out. The abundance of inertinite in the coal samples around 4 - 7% while the abundance of UC in coal ash range 4.00 - 8.55% (Table 4) which shows the percentage that is similar.

**Figure 3.** Reflected light photomicrographs showing unburned coal (UC) particles.

**Table 4.** Percentage of organic and inorganic components of coal ash samples based on petrography analysis.

| Sample | Unburned coal (%) | Glass (%) | Quartz (%) | Fe-oxide minerals (%) | Pyrite (%) | Spinel (%) | Mullite (%) |
|--------|-------------------|-----------|------------|-----------------------|------------|------------|-------------|
| MU-4   | 6.36              | 9.82      | 30.00      | 46.00                 |            |            | 7.82        |
| MU-6   | 8.55              | 10.55     | 32.91      | 40.36                 | 0.18       | 1.09       | 6.36        |
| MU-8   | 7.82              | 10.36     | 27.64      | 47.09                 | 0.18       | 0.18       | 6.73        |
| MU-10  | 3.09              | 13.09     | 33.64      | 44.00                 |            |            | 6.18        |
| MM-7   | 6.91              | 10.18     | 26.55      | 51.27                 |            |            | 5.09        |
| MM-6   | 4.00              | 9.09      | 24.55      | 54.55                 | 1.27       |            | 6.55        |
| MM-4   | 7.27              | 1.27      | 28.36      | 46.36                 |            |            | 6.73        |
| ML-1   | 6.00              | 9.45      | 25.64      | 51.09                 |            |            | 7.82        |
3.2.2. Coal Ash Inorganic Components

- **Quartz** ($SiO_2$). Quartz is one of the inorganic components found in coal ash. The abundance of quartz ranging from 24.55 – 33.64% (Table 4). Quartz (Figure 4. a) in petrographic analysis is characterized by colorless colors with white angular - sub rounded form.

- **Mullite** ($Al_6Si_2O_{13}$). Mullite (Figure 4. d) abundance of coal ash ranged from 5.09 – 7.82% (Table 4). Based on the petrographic analysis mullite has black color with a stretchy shape.

- **Fe-oxides minerals**. Fe-oxide minerals (Figure 4. b) is the most abundant components of coal ashes. The abundance of Fe-oxide mineral in coal ashes between 40.36 – 54.55% (Table 4) with characteristic red color with subangular - angular shape based on petrographic analysis.

- **Glass** (amorphous $SiO_2$). The glass component can be distinguished based on its morphology in the form cenosphere and pleiosphere. The Cenosphere (Figure 4. c) has a perfectly round morphology that is similar to a sphere, while the pleiosphere (Figure 4. f) is characterized by round morphology which is not perfect. The abundance of glass in coal ash samples ranged between 9.09 – 13.09%. The cenosphere abundance in coal ash samples ranged from 7.45 – 10.91% and pleiosphere abundance ranged from 1.64 – 2.18%.

- **Spinel**. Spinel (Figure 4. e) is only found in MU-6 and MU-8 samples (Table 4). Spinel types are found to be interpreted as Fe spinel which is supported by the abundance of Fe

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**Figure 4.** Reflected light photomicrographs inorganic components; a. Quartz (Qz); b. Fe-oxides mineral; c. Cenosphere; d. Mullite; e. Spinel (Spl); f. pleiosphere.
element in the coal sample. Petrographic analysis shows spinel characteristics are dark in appearance and it is generally a sphere composed of black crystals with dendritic texture.

4. Discussion
Coal ash is generally composed of a mixture of organic and inorganic components, both inherited directly from the coal and neoformed during ashing process. The following discussion will explain some of those materials transformation.

4.1. Transformation of organic component
Unburned carbon (UC) in coal ash indicates inefficiency in combustion. The nature and relative proportions of the macerals in the raw coals can significantly impact the UC content of coal ashes. The maceral composition is mainly an inertinite group in coal have significant effect on the proportion of UC produced in coal ash. In various ranks of coal, generally the inertinite maceral is more difficult to burn than other maceral groups. Percentage of inertinite maceral in coal ash is relatively same as in the original coal [20-22]. The abundance of inertinite in the coal samples around 4 - 7% while the abundance of UC in coal ash range 3.09 – 8.55% which shows the correlation between inertinite contents and UC contents.

4.2. Transformation of inorganic components
The major crystalline minerals in the coals are kaolinite, pyrite, and quartz. Minor proportions of chlorite, gypsum, Mg-calcite, siderite, dolomite, jarosite, hematite, and calcite also found in coals. In most coal ashes, inorganic components comprise the majority of the material. Similar to UC, certain inorganic components may represent neoformed glass or mineral components [23].

4.2.1. Quartz. Quartz (Figure 4. a) is the most common and recognizable mineral to pass through from the raw coal to the ashes. Quartz in coal ash derived from origin coal [24]. Crystalline quartz can be found in coal ash because it does not melt when it is heating at a temperature below 1400 - 1500°C [19].

4.2.2. Mullite. Mullite is a new mineral (neoform) formed from the coal ashing process. This mineral is generally derived from kaolinite in origin coal [24]. Mullite and another Al-Si minerals may be formed by solid-state reactions [25] or may crystallize from the melt [26]. The transformation of kaolinite into mullite takes place at temperature of 950 - 1000°C [27]. The decompositions of kaolinite starts at around 600 K due to release of water leading to the formation of metakaolinite and at around 1100k metakaolinite converts into mullite [28]. Petrographic analysis of mullite shows stretchy shape that produced from the initial melting of kaolinite when ashing process at high temperatures [24].

4.2.3. Glass (Cenosphere & Pleiosphere). Cenosphere formation is similar to the glass blowing process [29]. In fact, silica-containing melts (as in the case of cenospheres), present high viscosity and form glass easily upon cooling. On the other hand, if the temperature is high enough and the fall distance is enough, any droplet of melt will become spherical due to surface tension forces [30]. The gases emitted from a char particle during combustion, or from ash particle during melting, inflate the melted inorganic mineral matter and if cooling is fast, amorphous cenosphere can be formed. Gases capable to inflate spherical particle can come from decomposition of calcium and magnesium sulfates, kaolinite, calcium carbonate, dolomite and pyrite oxidation [31-32] all those reactions can occur at temperatures below 1000°C.

4.2.4. Spinel. Petrographic analysis shows spinel can form dendritic assemblages in glass (Figure 4. e). Spinel is common in coal ashes which is rich in Fe and sometimes found associated with magnetite [19]. The iron in the secondary minerals comes from the decompositions of Fe-sulfides and other Fe-
bearing minerals in the coal [26]. Fe-bearing mineral in the raw coal samples present as pyrite (FeS2), siderite (FeCO3), and hematite (Fe2O3).

5. Discussion
Based on its characteristics, coal ashes from Senakin coal consist of organic and inorganic components. Organic component presents as unburned coal that derived from partially burned resistant macerals such as inertinite group maceral. Inorganic components of coal ashes consist of quartz, mullite, Fe-oxide minerals, glass, and spinel. Quartz derived from the primary quartz mineral that pass through from the raw coal to the ashes because the heating temperature is lower than 1400 - 1500°C. Mullite produces from the crystallization of Al-Si melt during the transformation of kaolinite minerals or other Al-Si bearing minerals at temperature of 950 - 1000°C. Fe-oxides minerals and spinel derived from the transformation of Fe-bearing minerals (pyrite, hematite, siderite) during combustion of raw coal. Amorphous glass produced from the fast cooling of melted inorganic mineral matter. Characterization of the coal ash component can be used as a reference for the extraction method of valuable elements.

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