

Microstructural, Morphological, and Compositional Analyses of Duduguru Coal from the Middle Benue Trough, Nigeria

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Abstract

In this study, the microstructural, morphological, and compositional properties of DDG coal newly discovered in the Middle Belt Trough (MBT) sedimentary basin of Nigeria were characterised using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) techniques. Sample preparation revealed that DDG coal is a brownish-black rock with a smooth multilayered surface. This surface texture suggests it has undergone significant geological processes, potentially reflecting a high degree of coalification and metamorphism. Microstructure analysis showed that DDG consists of fine and coarse aggregate particles ranging in size from 25-50 µm, which exhibit irregular and angular shapes. Morphological analysis revealed whitish or glossy particles, which indicate the presence of quartz, kaolinite and other clay-based minerals. Compositional analysis revealed eight (8) elements broadly comprising metals (Mg, Al, Ca, and Fe), non-metals (C, O, and S), and metalloids (Si), which could play crucial roles in the conversion efficiencies, product yield/distribution, and potential by-products/emissions during DDG conversion. In summary, the study observed that DDG coal is a potential candidate for thermochemical conversion and energy production due to its favourable properties. However, selected metals and metalloids could present operational challenges related to ash formation, slagging, and corrosion.

Keywords: Microstructure analysis; Morphological characterisation; Compositional analysis; Duduguru coal; Middle Benue Trough; Nigerian coals.

1. Introduction

Coal is a brownish-black and carbon-rich sedimentary rock [1] that is widely utilised for various applications such as energy production and materials utilisation [2]. The utility potential of coals typically categorised based on either rank (i.e., low-rank or high rank) or grade (i.e., lignite, bituminous, sub-bituminous, and anthracite), depends on its energetic, fuel, and material properties [3]. Its potential utilisation also depends on its microstructural, morphological, and compositional properties [4]. These properties play a crucial role in determining their suitability and efficiency for conversion and utilisation in various industries [5].

The discovery of vast coal deposits in developing countries such as Nigeria presents opportunities for utilisation in many industries [3]. Since the discovery of coal in Nigeria circa the

1930s [6], its utilisation in the country has been greatly limited to steel production [7-8]. However, there is potential for coal application in vital sectors such as electric power generation [9], materials synthesis [10], fertiliser production [11], and specialised materials such as zeolite [12], adsorbent materials [13], and composites [14], among others. The application of coal for electricity power generation is critical to socio-economic growth and infrastructural development [15], which is essential to addressing the widely reported energy challenges in Nigeria [16].

Recently, large coal deposits were discovered in Duduguru village, sited in the Obi Local Government Area of Nasarawa State [17]. The region located in the Middle Benue Trough (MBT) is one of the most prominent sedimentary basins in Nigeria [18] due to its numerous coal deposits [19-22]. In the past, various studies have examined the potential of Duduguru coal (DDG) [15,17,23]. However, there are limited studies in the literature on specific coal characteristics, such as the particle morphology and mineralogical composition of the newly discovered DDG coal from the MBT.

Therefore, the primary objective of this study is to comprehensively analyse the microstructural, morphological, and compositional properties of DDG coal through advanced analytical methods. The DDG coal structure and chemical characteristics are examined through scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) to explore its applicability in thermochemical conversion processes for energy recovery. SEM analysis provides comprehensive descriptions of coal particle morphology and microstructural characteristics [24], whereas EDX helps to identify key elements and minerals [25]. Furthermore, coal properties influence energy density, product yield, and emissions and pose challenges such as slagging, corrosion, and ash formation during thermochemical conversion processes like combustion [26-27].

Hence, a comprehensive understanding of such properties could help engineers and policy stakeholders to design, develop, and deploy processes and technologies for effective energy recovery and utilisation from coals. Overall, the paper examines and highlights the energy potential of DDG coal, along with potential restrictions posed by the existence of metals, non-metals, and metalloids in this structure. Lastly, it is envisaged that the paper will provide the research community with comprehensive knowledge of one of the newest coal resources in Nigeria. It also presents beneficial data for future exploitation and utilisation strategies for coal energy in the country.

2. Experimental

This study set out to investigate the microstructural, morphological, and compositional analyses of Duduguru (DDG) coal, recently discovered from the Middle Benue Trough (MBT) sedimentary basin in Nigeria.

2.1. Sample preparation

The DDG coal was sampled from deposits in Duduguru village, located in Obi Local Government Area of Nasarawa State. The region is geographically situated in Nigeria's Middle Belt Trough (MBT) sedimentary basin. Consequently, about 50 kg of DDG coal was bagged in rock form and labelled before transporting it for further analysis in the Hydrogen & Fuel Laboratory Universiti Teknologi Malaysia. Figure 1 presents the rock sample form of the DDG coal. Before analyses, the rock samples were ground in a blender grinder (Model: Panasonic 400 MX dry miller Grinder, Malaysia) and subsequently sifted in a laboratory sieve (Mesh size 60, Tyler MS USA). The resulting powdered DDG coal samples were finally obtained as 250 micron-sized particles and stored in airtight sample bags before SEM and EDX analyses.

2.2. Morphological and microstructural analyses

In this study, scanning electron microscopy (SEM) was employed to examine the morphological and microstructural properties of DDG coal. Before the analysis, the powdered DDG coal was dispersed on adhesive carbon tape and deposited on the SEM grain mounts. The sample was then sputter coated for 10 seconds with a thin slice of gold (Au) using a sputter coating machine (Model: Quorum Q150R S, United Kingdom). Subsequently, the grain mounts were transferred to the SEM analyser (Model: JEOL JSM-IT300, Japan) and degassed in the

sample holder chamber. The DDG sample was then scanned to examine the morphological and microstructural properties of the DDG coal using the SEM analyser operated at 20 kV voltage and 5 mm working distance. On completion, the data was analysed on the AZTEC software (Oxford Instruments, United Kingdom) to elucidate the DDG characteristics using SEM micrographs presented at $\times 1000$ and $\times 2000$ magnifications in Figures 2 (a & b).

2.3. Compositional analysis

In this study, energy dispersive X-ray (EDX) spectroscopy was employed to examine the compositional properties of DDG coal. The EDX was fitted to the SEM analyser (Model: JEOL JSM-IT300, Japan) to determine the elemental/mineral composition (EMC) of DDG coal. EDX data was obtained from mapping and computing the outlined zones in the SEM micrographs using AZTEC EDX software (Oxford Instruments, United Kingdom). Lastly, the EMC was presented in weight per cent (wt.%) to acquire a comprehensive understanding of coal sample compositions normally concealed by conventional ultimate/elemental analysis.

3. Results

The primary aim of this study is to examine and highlight the microstructural, morphological, and compositional properties of DDG coal from the MBT sedimentary basin of Nigeria.

3.1. Sample appearance

Figure 1 presents the rock form of the DDG coal as recovered from Obi Local Government Area of Nasarawa State. As can be seen, DDG has a brownish-black appearance, which indicates it is characterised by a high content of carbon typically observed in coals at the matured coalification phase [31]. Its surface is largely smooth with a multilayered and varied texture, which indicates it has experienced geological processes and changes (e.g., accumulation of minerals and organic matter) in its depositional environment [28]. The smooth layered appearance may also indicate that DDG has experienced marginal degrees of weathering or fragmentation [29].



Figure 1. Rock form of DDG coal recovered from the Obi Mine, Nasarawa State, Nigeria.

Further analysis revealed that glossy interruptions exist on the surface layers of DDG. Selected sections revealed minor colour or textural changes that are indicative of the organic or mineral content of the coal [30]. Such variations could assist in elucidating coal rank or classification, as high-rank coals exhibit uniform colours or texture with minimal interruptions [3,31]. The brownish-black appearance, smooth layered surface, and glossy interruptions of DDG also

indicate that it may have experienced an extensive degree of metamorphism and geological processes [32-33]. Nonetheless, it is important to state that comprehensive petrographic, microscopic, and chemical analyses remain the most reliable approach for determining the rank, classifications and compositional properties of newly discovered coals.

3.2. Morphological and microstructural properties

According to the literature [32-33], the quality, conversion, utilisation and emissions potentials of coal samples are greatly impacted by their morphological and microstructural properties. In this study, the DDG properties were examined through SEM analyses. Figures 2 (a) and (b) present detailed SEM micrographs of DDG at magnifications of $\times 1000$ and $\times 2000$ representing 25 μm and 50 μm sized particles, respectively. As can be observed, the DDG coal particles are mainly in the 25-50 μm size range. The micrographs reveal the sample comprises a mixture of fine and coarse aggregate particles, each displaying irregular, angular shapes normally associated with pulverised coal, albeit the surfaces have principally smooth textures. The heterogeneous shapes of the DDG coal particles could be due to the impact of mechanical fractionation or handling during coal preparation, as highlighted in Section 2.1. Consequently, the DDG coal particles experienced fragmentation into smaller and non-uniform particles from the rock form obtained from sampling. On the other hand, the smooth texture of the particle surfaces suggests relatively low roughness, which could impact synergy or interfacing with different materials (e.g., biomass fuels, binders or ab- or absorbents) during further processing or utilisation.

Furthermore, the SEM micrographs revealed tiny pores on the particle surfaces of DDG coal. Such microscopic-sized pores have the potential to improve the surface area required for thermal and chemical reactions, which suggests that DDG coal could be efficiently used for specialised applications [34]. For example, the process of filtration, adsorption, and catalysis typically requires materials with highly reactive or porose surfaces [35-36]. However, the size and distribution of pores on the DDG coal require further analysis using Brunauer-Emmett-Teller (BET) or particle size (PS) analyses to highlight DDG coal particle utilisation potential for such specialised applications in the industry. The morphological analysis reveals that DDG coal contains whitish or glossy particles, which, according to the literature [37-38], could indicate the existence of quartz, kaolinite and other clay-based minerals. Overall, the SEM analysis provided vital insights into the morphology and microstructural/surface properties of DDG coal, which are crucial for detailed comprehension of its thermal and chemical performance during processes and applications in industry.

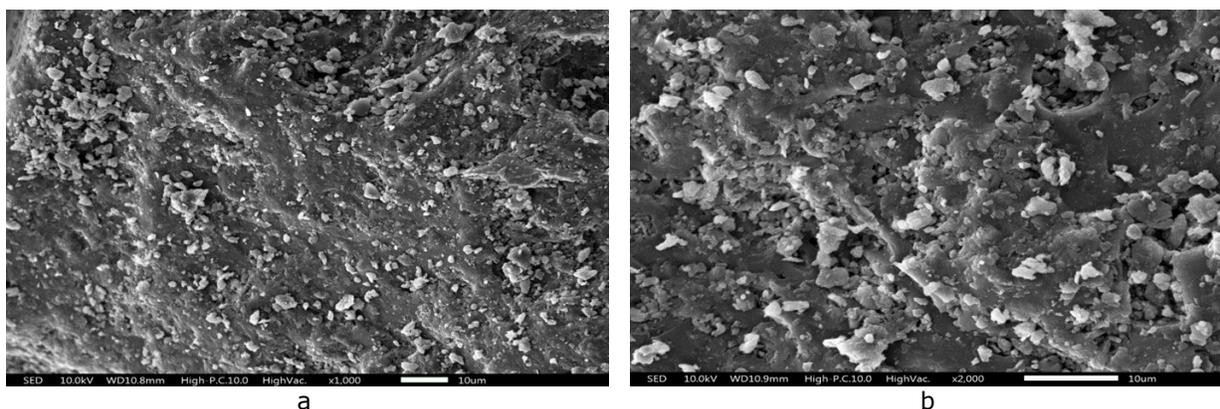


Figure 2. SEM micrographs for DDG coal at magnifications of $\times 1000$ and $\times 2000$.

3.3. Compositional properties

The EDX spectrophotometer was employed to examine the composition of non-metals, as well as metals and their related minerals in coals [25, 39], such as DDG. Table 1 presents the detailed elemental composition of DDG coal determined based on the EDX procedure described

in Section 2.3. As can be observed in Table 1, the EDX analysis detected a total of 8 elements in the sample comprising metals, metalloids, and non-metals in the coal composition. The composition of metal elements includes Mg, Al, Ca, and Fe, whereas the non-metal elements are C, O, and S, and the single metalloid detected was Si.

Table 1. EDX elemental composition of DDG coal.

Element	Symbol	Wt. %
Carbon	C	85.40
Oxygen	O	12.27
Magnesium	Mg	0.10
Aluminium	Al	0.40
Silicon	Si	0.50
Sulphur	S	0.82
Calcium	Ca	0.33
Iron	Fe	0.18

The presence of these groups of elements could have a significant impact on coal conversion and utilisation [40-41]. For example, coal combustion and its emissions are greatly influenced by the compositional properties of the selected samples [42]. The C content of DDG coal, which is > 80%, is the main source of energy, accounting for the higher heating value (HHV) of the coal. However, the C content also contributes to the production of carbon dioxide (CO₂) [3], which is a well-known greenhouse gas (GHG) and by-product of the combustion process [43]. The O content of DDG, which is > 10%, also has an impact on the HHV and conversion efficiency of coal during combustion [44]. The oxygen content of coal is also an important dynamic in the production of C, S, and N oxides such as carbon monoxide (CO), sulphur oxides (SO_x) and nitrous oxides (NO_x) [45]. The emission of CO and SO_x have significant impacts on the environment, resulting in the occurrence of acid rain and water pollution [46].

The metal elements detected in DDG were Mg, Al, Ca, and Fe, which could be due to the presence of minerals and compounds such as magnesia (MgO), alumina (Al₂O₃), wollastonite (CaSiO₃), and pyrite (FeS₂), respectively. Mg has an impact on the characteristics of coal ash [47], which helps to lower the slagging tendency in the equipment used for coal combustion [48]. Al has an impact on the ash fusion temperature of coal, which could result in slagging and corrosion [49]. The EDX analysis also detected Si, which, although it lowers the tendency for the formation of slag, tends to increase the stability or refractoriness of ash [50]. Ca could result in the formation of slag, although it also neutralises acid gases, thereby reducing sulphur-based problems [51]. Erarslan and Örgün [52] also reported the presence of Ca and Ca-Al silicates, such as wollastonite, anorthite, and nacrite in coal. Similarly, Li [53] reported the detection of wollastonite along with quartz, magnesium oxide, and calcium zeolite in coal using various analytical techniques. Lastly, Fe has an impact on the handling and density of coal ash, which causes corrosion and particulate matter emissions [54].

4. Conclusions

The study examined the microstructural, morphological, and compositional analyses of DDG from the MBT sedimentary basin of Nigeria using various analytical techniques. The DDG coal obtained as a brownish-black rock mass was pulverised, sieved, and analysed using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX). The SEM analysis revealed the DDG coal comprises a mixture of fine and coarse aggregate particles in the 25-50 µm size range, each displaying irregular, angular shapes along with surfaces characterised by smooth textures. The morphological analysis revealed whitish or glossy DDG particles, which indicate the presence of quartz, kaolinite and other clay-based minerals. On the other hand, EDX identified 8 elements comprising metals (Mg, Al, Ca, and Fe), non-metals (C, O, and S), and metalloids (Si) in DDG. Overall, the study finds that the pulverised DDG coal particles are suitable for thermochemical conversion and energy recovery due to the presence of combustible elements. However, selected metal and metalloid elements could also pose challenges due to ash formation, slagging, and corrosion.

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