Petroleum and Coal

Article **Open Access**

Microstructural, Morphological, and Compositional Analysis of Owukpa Coal from the Middle Benue Trough, Nigeria

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Received November 4, 2024; Accepted March 16, 2025

Abstract

In this study, the microstructural, morphological, and compositional properties of Owukpa (WKP) coal were examined through scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analyses. The results showed that WKP coal is characterised by largely irregularly shaped particles with smooth textured surfaces, curved edges, and compact structures ranging from 10 μm to 25 μm. The micron-sized particles of WKP indicate the coal sample is potentially thermally reactive and could undergo efficient thermochemical conversion for future energy recovery and utilisation. Likewise, the diverse shapes, smooth texture, and compactness of WKP coal particles could improve flow properties, safety, and product yield during thermal conversion. Conversely, the EDX analysis detected 7 elements namely; carbon, oxygen, aluminium, silicon, sulphur, titanium, and iron. Based on the weight percentage (wt.%), the major elements detected were carbon (80.47 wt.%) and oxygen (16.59 wt.%). This suggests that WKP has promising energy recovery potential due to its high carbon content, although the high oxygen could potentially increase CO_2 emissions. EDX also detected inorganic components namely; Si, Al, Ti, Fe, and S. The silicon (Si, 0.88%) and aluminium (Al, 1.43%) reveal that WKP contains silicates and clay minerals, which typically influence the composition and behaviour of ash during combustion. The metal elements titanium (Ti, 0.10%) and iron (Fe, 0.15%) which promote ash formation and catalytic activity during combustion, respectively were also detected in WKP. In conclusion, we find that WKP coal has energy recovery and utilization potentials, although future research is needed to understand its thermal conversion, degradation behaviour, and ash chemistry.

Keywords: *Owukpa coal; Microstructural analysis; Compositional analysis; Middle Benue Trough; Energy resources; Trace elements.*

1. Introduction

Coal is a fossilized, carbon-rich, and brownish-black sedimentary rock that is widely distributed across the globe [1]. The discovery and utilisation of coal dates back to primitive times when it was used by prehistoric men for cooking and heating [2-3]. Early historical records also report that Roman blacksmiths utilised coal for forging, metalworking, and jewellery production during the conquest of Britain [3-4]. However, it was during the Industrial Revolution of the late 1700s to mid-1800s that coal became an important fuel and energy source for powering steam engines and generating electricity [5-6]. In recent times, coal has become an important energy source used to power electricity grids worldwide owing to its abundance, cost-effectiveness, accessibility, and reliability [7]. According to key data for 2023, various energy analysts report that coal accounts for one-third or ~ 35-40% of global electricity generation [8-9]. According to the IEA, coal currently accounts for 24.5-26.9% of the energy mix worldwide [10]. Given these dynamics, coal is considered an important catalyst for socio-economic growth and infrastructural development [11-12]. Some analysts also suggest that coal could become a crucial energy source in developing nations like Nigeria [13-14].

Despite being Africa's most populous nation, Nigeria's socio-economic growth remains hampered by an energy crisis [15-16]. Annual reports indicate the nation is currently limited by poor electricity generation, transmission line losses, load shedding, and constant blackouts among other challenges [17-18]. However, the challenges of electricity generation and blackouts could be addressed by developing and deploying coal-fired electricity in Nigeria [14-19]. Likewise, coal-fired power generation poses numerous public health and environmental challenges [20-21]. Coal utilisation accounts for > 45% of global CO_2 and other greenhouse gas emissions which cause global warming and climate change [22-23]. Other pollutants emitted from coal-fired electricity generation at power plants include particulate matter, toxic aerosols, mercury, and fly/bottom ashes [24-25]. According to energy analysts and environmental campaigners, these emitted substances result in acid rain, respiratory illnesses, and neurological damage, among other challenges [20,26].

Access to cheap, constant, and reliable electric power is critical to socio-economic growth and infrastructure development [27-28]. Developing nations such as Nigeria with abundant coal deposits must explore the fuel and energy properties of such resources to ensure high efficiency and low emissions in future electricity generation. Therefore, the main objective of this paper is to critically characterise, and examine the microstructural, morphological, and compositional properties of Owukpa (WKP) coal from the middle Benue trough (MBT) sedimentary basin of Nigeria. Previous studies conducted on WKP have been limited to its combustion kinetic characteristics, thermal degradation behaviour, physico-chemical, and mechanical properties [29-31]. To the best of the authors' knowledge, this is the first study to highlight the microstructural, morphological, and compositional properties of WKP using SEM and EDX analyses. It is envisaged that this study will address the gap in the literature and provide insights into its potential applications, environmental burden, and strategic role in the energy future of Nigeria as well as guide policy, and foster strategies for coal utilization.

2. Experimental methods

The objective of the study is to examine the microstructural, morphological, and compositional properties of Owukpa (WKP) coal obtained from the Middle Benue trough (MBT) sedimentary basin of Nigeria.

2.1. Sample preparation

The rock sample of Owukpa (WKP) coal was provided by a mining and prospecting firm – Ehinehi Nigeria Enterprises Limited. The WKP sample was crushed and ground into a heterogeneous variable particle-sized powder using the dry miller (Model: MX-AC400TSK Mixer Grinder by Panasonic, Malaysia). Next, the pulverised WKP sample was sieved using the W. S. Tyler analysis standard sieve (mesh size 60, Cleveland OH, United States) to obtain homogeneous-sized particles < 250 μm before further analysis.

2.2. Microstructural and morphological analyses

The microstructural and morphological properties of WKP coal were examined through scanning electron microscopy (SEM). The analysis was conducted at the Surface Analysis Section of the University Industrial Research Laboratory (UiRL), University of Technology Malaysia (UTM), Johor Campus. For each test, a miniscule amount of WKP powder was deposited on epoxy tape pre-placed on grain mounts. Next, the sample was sputter coated with a thin layer of gold (Au) for 10 seconds using the automatic sputter coating apparatus (Model: Quorum Q150R S, United Kingdom). The sample was then transferred to the sample holding chamber of the SEM analyser (Model: JEOL JSM-IT300, Japan). It was then degassed to eliminate any extraneous materials that could affect the reliability and accuracy of the tests. Next, the sample was scanned using the SEM microscope to assess its microstructure and morphological properties using the point ID and mapping program technique. The SEM analysis was performed using 20 kV voltage, and 5 mm working distance settings. On completion, the SEM micrographs of WKP were analysed through the AZTEC software (Version: Oxford Instruments, United Kingdom). The SEM micrographs are presented in Figures 1 (a & b) at magnifications of ×400 and ×1000.

2.3. Compositional analysis

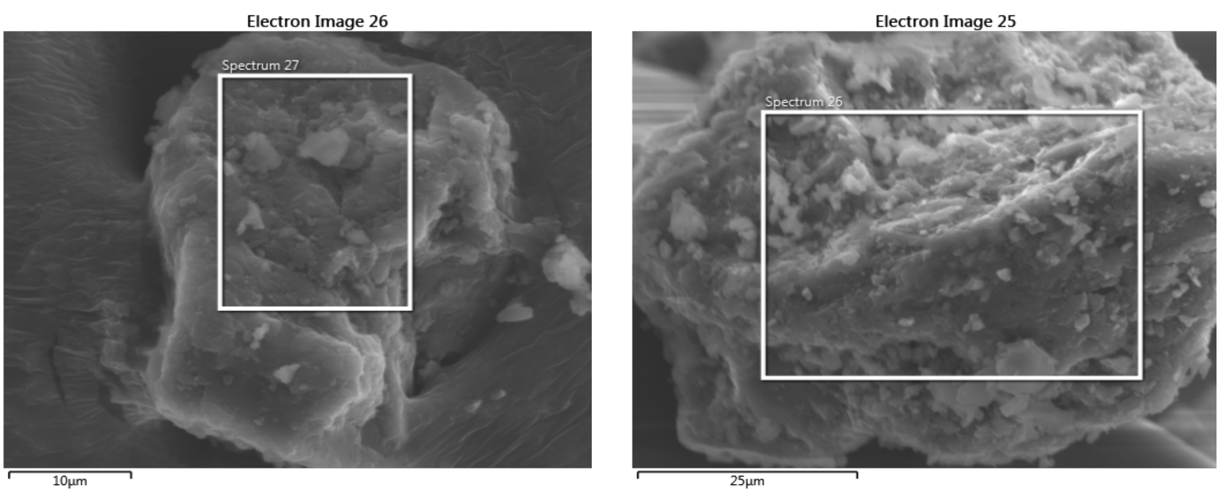
The compositional analysis of WKP coal was performed through an energy-dispersive X-ray (EDX) spectrophotometer attached to the SEM analyser (Model: JEOL JSM-IT300, Japan). For each test, the resulting WKP SEM micrograph was analysed with AZTEC EDX software (Oxford Instruments, UK) using the mapping feature of the device. Next, each mapped zone was computationally analysed to elucidate the elemental and mineral composition of WKP. The compositions were reported in weight percent (wt.%) to gain insights into sample compositions typically undetected by conventional elemental analysis.

3. Results and discussions

The objective of the study is to examine the microstructural, morphological, and compositional properties of Owukpa (WKP) coal.

3.1. Microstructural and morphological properties

SEM analysis provides critical insights into the microstructural and morphological properties of coals [32-33], which influence their quality, combustion behaviour, and environmental impact [19,34]. These properties aid in optimizing processes, maximizing utilization, and advancing cleaner coal technology [35-36]. Figures 1 (a) and (b) show the SEM micrographs of WKP coal magnifications of ×400 and ×1000 representing 10 μm and 25 μm sized particles, respectively.



(a) (b)

Figure 1. SEM Micrographs of WKP coal.

As observed, the SEM micrographs reveal that the WKP coal particles are largely in the micron size range from 10 – 25 microns. Further analysis reveals that the particles are compact and irregularly shaped with smooth textured surfaces and curved edges. Based on the foregoing, the microstructure of WKP coal particles could have important implications for their pretreatment, processing, and utilisation [37]. For example, the micron-sized distribution of the WKP coal particles could enhance thermal reactivity and conversion efficiency during thermochemical processes such as pyrolysis, gasification and combustion for energy recovery and utilisation [38-39]. The varied shapes and smooth texture of the coal particles could also enhance the flow properties, and operational safety, as well as decrease the generation of coal dust during processing, handling, and transportation [40-41].

The compact nature of the WKP implies a high density of the coal particles which could impact the product yield and distribution during thermal conversion and energy utilisation [42-43]. On the other hand, the morphological properties of the surfaces of WKP coal particles are characterised by whitish or glossy-hued layers, which suggest the presence of quartz [44]. The layers are highly coated and the staked layers indicate the presence of kaolinite clay-like minerals, as similarly reported in the literature [45]. To further examine the elemental / mineral composition of WKP, EDX analysis was conducted as presented in Section 3.2.

3.2. Compositional properties

The compositional analysis of WKP coal was examined using EDX analysis as outlined in Table 1 and Figure 2. The results are presented in weight (wt.%). The analysis detected 7 elements namely carbon, oxygen, aluminium, silicon, sulphur, titanium, and iron. As observed, WKP has high carbon content (C, 80.47%) indicating that it has good energy recovery potential, although the high oxygen content (O, 16.59%) could decrease the calorific value, energy density, and exacerbate the emission of carbon dioxide (CO_2). The carbon and oxygen are strong indicators of the organic matter, macerals, and volatile matter content of the coal sample [34]. According to Ge [46], carbon is the life spine of the structure of any coal sample, whereas oxygen is the most copious heteroatom. In addition, these constituents provide information on the rank, history, and prospective performance as well as quality and applicability of coals for thermochemical conversion and energy recovery [34].

Table 1. EDX Compositional analysis of WKP coal.

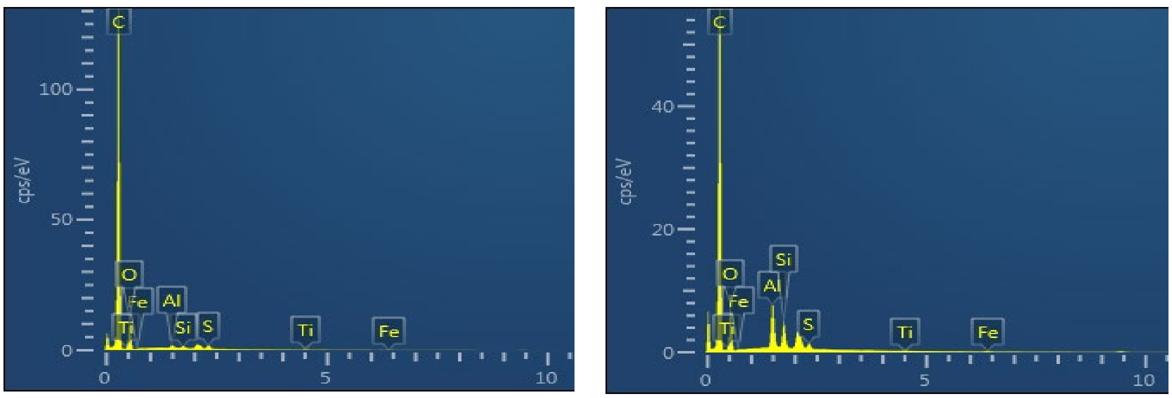
Element	Symbol	Wt.%	Element	Symbol	Wt.%
Carbon	C	80.47	Sulphur	S	0.41
Oxygen	O	16.59	Titanium	Ti	0.10
Aluminium	Al	1.43	Iron	Fe	0.15
Silicon	Si	0.88			

The analysis also the detected various inorganic components in WKP. The presence of silicon (Si, 0.88%) and aluminium (Al, 1.43%) suggest the presence of silica (SiO_2) and alumina (Al_2O_3), respectively. The findings suggest WKP coal contains silicate, aluminosilicate, and clay minerals, which play an important role in the composition and behaviours of ash during coal combustion. Other studies have reported the presence of minerals and aluminosilicates in the structure of various coals [32,47]. The aluminosilicate minerals in coals strongly influence the combustion behaviour as well as the performance of its emissions control and conversion systems due to their ability to form slag from the ash [48-49].

The EDX analysis also highlighted the presence of the metal elements titanium (Ti, 0.10%) and iron (Fe, 0.15%) in the WKP coal structure. The two *d*-block or transition metal elements can markedly impact the reactivity and products of coal thermal conversion. The Ti promotes ash formation and reactivity [50-51], whereas the coal conversion efficiency, particularly during combustion, is enhanced by the catalytic activity of Fe [52-53]. Together, Ti and Fe could substantially influence ash melting behaviour, potential emissions, secondary waste products, and the total performance of coal fuels [34,53-54]. Kimura [55] also reported the presence of Ti, and Fe along with other inorganic elements/minerals in coals extracted from the *Ishikari* coal field in the *Ashibetsu* region of Japan. The study also reports that Ti possibly occurs as anatase (i.e., titanium dioxide or TiO_2) as well as in the form of mineralised clays. On the other hand, the study reported that Fe occurs as carbonate-based minerals such as ankerite and siderite, as well as pyrite (FeS_2) in the coal samples [55].

Lastly, EDX detected sulphur (S) in WKP coal, which could be due to the mineral pyrite (FeS_2) typically found in low-ranked coals [56]. According to Wang [56] sulphur exists in either organic or inorganic form, with the latter (which exists as crystalline FeS_2 ore) posing the most significant challenge to conversion efficiency and emissions control during coal utilisation. High sulphur content has a significant influence on the spontaneous combustion characteristics of coals. In this study, the S content of WKP is 0.41%, which although low could still impact the SOx emissions and environmental impact of the coal during combustion. In conclusion, the EDX analysis highlights that WKP has fuel conversion and energy recovery potentials, although the presence of inorganic species necessitates measures to ensure effective ash management and emission control in the future.

Figure 2 shows the EDX spectrum for WKP coal. As observed, the spectrum shows strong peaks or signals for C, Si, and O, but weak versions for Ti, S, Al, and Fe. The data confirms that the major elements in WKP are C, Si, and O, which highlight the carbon-rich nature and presence of silicate minerals in coals. The weaker signals of Ti, S, Al, and Fe point to the trace nature of these inorganic elements in the coal. Although present in small quantities, the elements play an important role in the conversion efficiencies, reactivities, products, and emissions resulting from coals. Lastly, the results show that the metal elements could be extracted from coals for application in other sectors in the future.



(a) (b)

Figure 2. EDX spectrum for WKP coal.

4. Conclusions

The study examined the microstructural, morphological, and compositional properties of Owukpa (WKP) coal from the middle Benue trough in Nigeria. The microstructural and morphological properties of WKP determined through SEM revealed the WKP coal particles are largely irregularly shaped with smooth textured surfaces and curved edges with compact structures in the micron size range, which could enhance the thermal reactivity and conversion efficiency of WKP for energy recovery and utilisation. The WKP coal particles possess varied shapes and smooth textures, which could improve flow properties and operational safety. The compact nature of the coal particles implies a high density, which could impact product yield and distribution during thermal conversion and energy utilization. The coal sample contains seven elements: carbon, oxygen, aluminium, silicon, sulphur, titanium, and iron as deduced from EDX analysis. The high carbon content suggests WKP possesses good energy recovery potential, whereas the oxygen content could adversely affect calorific value and exacerbate CO_2 emissions. The Ti and Fe detected in WKP could markedly impact the WKP ash melting behaviour, emissions, secondary waste products, and overall fuel performance during thermal conversion. Therefore, WKP is a potential fuel for energy recovery and utilisation. Future studies could examine the thermal conversion, degradation behaviour, and ash chemistry of WKP to predict its energy recovery and emissions profile for the design and development of power plants.

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