

Physicochemical Fuel Properties and Carbonization Kinetics of Duduguru Coal

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Abstract

The growing demand for cheap electricity and the significant discovery of new coal deposits in developing nations like Nigeria has increased interest in coal-fired power generation. Conversely, comprehensive data on the fuel properties, emissions profiles, and environmental impacts of coal exploration and energy exploitation remain scarce. Therefore, this study examines the physico-chemical, microstructure, mineralogical, and thermo-kinetic properties of Duduguru (DDG) coal from a newly discovered deposit in Obi Local Government Area of Nasarawa State, Nigeria. The results showed that DDG contains high carbon and hydrogen but low oxygen contents, which accounts for its higher heating value (HHV) of 28.39 MJ/kg. Based on its volatile matter content and HHV, DDG is ranked as a high volatile bituminous C coal, which typically exhibits HHV in the range 26.70 MJ/kg - 30.20 MJ/kg. The microstructure and chemical analyses indicated that DDG consists of heterogeneous sized particles with a distinct white lustre. This could be ascribed to the presence of mineral and metal components such as quartz, kaolinite, and the metal elements (such as Ti and Fe) typically found in coal structure. Thermal analysis revealed that the change in heating rates and temperature significantly affected the thermal degradation behaviour and temperature profile characteristics of DDG. As a result, DDG experienced significant mass loss ($M_L = 39.32\%$ to 41.28%) and residual mass ($RM = 58.72\%$ to 60.68%). Furthermore, the results showed that the increase in heating rates from lower to higher values enhances the formation of a higher mass of residuals. In general, DDG is a high-rank coal, which may be suitable for steam generation for electricity or metallurgical coke for steel production.

Keywords: Carbonization; Kinetics; Fuel Characterisation; Duduguru; Coal; Nigeria.

1. Introduction

Coal remains an integral part of the global energy mix [1-2]. It is also the most abundant and widespread fossil-based fuel on the planet. Current estimates indicate that coal accounts for over 60% or 1 trillion tonnes of all fossil fuels that can be economically recovered on planet earth. Currently, coal accounts for 40% or ~ 8200 TWh (terawatt hours) of coal-fired electricity generation around the world [3]. With the increasing demand for cheap electricity around the world, it is estimated that coal-fired electricity generation could increase in the near future [4-5]. According to the International Energy Agency (IEA), the global demand for coal increased by 1% to ~7600 Mt in the year 2017 driven by robust power generation due to industrial output

and high electricity demand in India, China, and other developing nations such as Vietnam. Likewise, coal power generation worldwide expanded by 3% or 250 TWh, which represents 40% and 38% of additional power generation and global energy mix, respectively [6].

In the same vein, the discovery of large deposits in traditionally energy deficient nations in the developing world is projected to increase the utilization of coal in the future [7]. Due to the significant deficit in electric power generation, developing countries will require over 900 TWh to meet their energy demands. According to the World Bank, over 1 billion people lack access to stable electricity supply [8], resulting in a severe energy crisis that affects over 33% of economies including Nigeria [9]. In spite of Nigeria's position as the largest economy in Africa, the nation routinely experiences epileptic power supply in the form of perennial power cuts, black-outs, and load shedding resulting in epileptic power supply yearly [10-11]. These setbacks are ascribed to the nation's low power generation, inefficient distribution, poor infrastructure, among other problems [12]. Furthermore, the poor state of the electricity sector has significantly impacted the socio-economic growth and sustainable development of the nation [13-14].

Given these issues, the nation's energy policy seeks to diversify power generation from biomass, petroleum (including oil and gas) and hydropower which account for 83%, 16% and 1% of Nigeria's energy mix, respectively [15]. Likewise, Nigeria is rich in both renewable energy resources and other fossil fuels [16] such as coal. According to analysts, Nigeria's coal supplies are estimated at 2.75 billion tonnes of which a known reserve of 690 million (comprising 12% lignite, 49% sub-bituminous, and 39% bituminous), and 39% bituminous are located across the nation's sedimentary basins [17-19]. The strategic location of Nigeria's coal deposits in various geopolitical regions presents significant opportunities for the nation to locate and generate on-site coal-fired electricity. Furthermore, the integration of coal will create a structured energy mix that could potentially address energy shortage, stimulate socio-economic growth and catalyse infrastructural development, particularly in developing countries like Nigeria.

The recent discovery of vast new deposits in Duduguru, Shankodi-Jangwa, Atito-Akpuneje, and Lafia-Obi have raised awareness on the potentials of exploiting Nigeria's coal for energy, chemicals and various other applications [20-23]. However, there is limited data on the fuel properties of the many newly discovered Nigerian coal deposits, particularly Duduguru located in Duglu village in Obi Local Government Area of Nasarawa State, Nigeria. Therefore, this study seeks to examine the fuel, carbonization, and kinetic characteristics of Duduguru coal from Nigeria. Typically, fuel properties are required to classify coal into various ranks and evaluate its suitability for various applications. It is envisaged that the findings of this study will present novel insights into the energy recovery potential of Duduguru coal from Nasarawa state located in the Middle Benue Trough in Nigeria.

2. Materials and methods

2.1. Physicochemical analyses

The physicochemical fuel properties of Duduguru (DDG) coal was examined based on ultimate (elemental), proximate, and calorific value analyses. DDG coal is a newly discovered coal sample from Duduguru village in Obi Local Government Area of Nasarawa State, Nigeria. The elemental analysis of DDG was determined by CHNS analysis (Model: vario MACRO Cube Analyser, Germany) to determine the carbon, hydrogen, nitrogen, and sulphur composition. The proximate analysis was carried out using a muffle furnace (Model: Ney Vulcan D-130, USA) based on the ASTM standards D3173-75 for moisture, ash, and volatile matter composition. Lastly, the calorific value was carried out through bomb calorimetry (Model: LECO AC350, UK) to determine the higher heating value (HHV) of the sample. All measurements were performed in duplicate to confirm the reliability of the results.

2.2. Microstructure and mineralogical analyses

The morphological and chemical composition of Duduguru coal was conducted by scanning electron microscopy (SEM) and energy dispersive x-ray (EDX) methods, respectively. Before the SEM/EDX analyses, the DDG sample was sputter-coated with gold (Au) using the Quorum

Q150R S equipment. Next, the sputter-coated sample was placed in the sample chamber of the SEM/EDX analyser and degassed to remove residual foreign matter and any oxidative gases. Next, the SEM/EDX microscope was initiated to scan for the microstructure and chemical composition of the sample. The operational settings of the SEM/EDX analyser were set at voltage 20 kV, working distance 5 mm, and a magnification of $\times 1000$. In the end, the SEM image was examined using the proprietary AZTEC EDX software from Oxford Instruments (UK). Next, the point ID and mapping program of the software was initiated to determine the elemental composition of DDG coal in weight per cent (wt. %).

2.3. Carbonization analysis

The carbonization characteristics of Duduguru (DDG) coal were examined through non-isothermal thermogravimetric analysis (TGA). The process was examined based on the multiple heating rate programmes. For each run, about 16 mg of the powdered DDG coal sample was heated from 30°C to 900°C based on different heating rates from 10°C/min to 30°C/min ($\Delta 10^\circ\text{C}/\text{min}$) using the thermogravimetric analyser (TGA Model: Shimadzu TG-50 Japan). During the tests, the TG system was flushed with ultra-pure (99.99%) nitrogen (N₂) gas to remove evolved gases from DDG and maintain an inert environment for carbonization. The flow rate of N₂ gas during the tests was 50 mL/min. On completion, the mass loss (%) and derivative mass loss data (%/min) were plotted as the TG and DTG graphs. Subsequently, the temperature profile characteristics (TPCs) of the DDG coal carbonization process were determined from the TG/DTG plots. The TPCs including; the ignition (T_i), midpoint (T_m), peak decomposition (T_p), and burnoff (T_f) temperatures along with the mass loss (M_L , %) and residual mass (R_M , %) were determined based on the procedural features of the Shimadzu Thermal analysis software (Version: Workstation TA-60WS). Based on the TPCs deduced, the carbonisation degradation kinetics of DDG was determined from the peak decomposition (T_p) values substituted into the Kissinger Kinetic Model (KKM). The fundamental theory and model equations of KKM are presented in section 2.4.

2.4. Carbonisation kinetic analysis

The Kissinger Kinetic Model (KKM) was adopted to examine the carbonisation kinetic properties of DDG coal in this study. The fundamental theory and model equations of KKM are derived from the general Arrhenius equation for thermally degrading carbon materials described as follows;

$$k(T) = A \exp\left(-\frac{E_a}{RT}\right) \quad (1)$$

where the terms; $k(T)$, A (min^{-1}), E_a (kJ mol^{-1}), and R ($\text{J mol}^{-1} \text{K}^{-1}$) denote the temperature-dependent rate constant, frequency factor, activation energy, and molar gas constant, respectively.

Therefore, the thermal degradation of DDG coal under non-isothermal, multiple heating rates, and oxidative combustion conditions can be described by the equation;

$$\frac{d\alpha}{dT} = \frac{A}{\beta} \exp\left(-\frac{E_a}{RT}\right) f(\alpha) \quad (2)$$

where the terms β denotes the heating rate ($\beta = 10^\circ\text{C}/\text{min}$, $20^\circ\text{C}/\text{min}$, $30^\circ\text{C}/\text{min}$) and $f(\alpha)$ denotes the function of reaction model for the combustion kinetics of DDG coal. Next, the separation and integration of variables were performed to deduce the governing model equations for the Kissinger kinetic model (KKM). Consequently, the peak decomposition (T_p) of the DTG plots was substituted into Eq 3 to determine the kinetic parameters for the thermal degradation of DDG. The KKM equation is given by as follows;

$$\ln\left(\frac{\beta}{T_p^2}\right) = \ln\left(\frac{AR}{E_a}\right) - \ln\left(\frac{E_a}{RT_p}\right) \quad (3)$$

Subsequently, Eq. 3 was adopted to compute the activation energy, E_a and frequency factor (A) from the straight plot of $\ln(\beta/T_p^2)$ against $1/T_p$ - where p is the peak temperatures for drying (T_{DRY}), and devolatilization (T_{DEV}) deduced from the DTG peaks for drying and devolatilization respectively.

3. Results and discussion

3.1. Physicochemical properties

Table 1 presents the physicochemical properties of DDG coal based on ultimate (elemental), proximate, and calorific value analyses. The results are all reported in dry basis (*db*) except for the moisture content, which is reported in as-received basis (*ar*).

Table 1. Physicochemical fuel properties of Duduguru (DDG) coal

Analyses	Element	Symbol (Unit)	Composition
Ultimate	Carbon	C (wt.%)	63.77
	Hydrogen	H (wt.%)	5.87
	Nitrogen	N (wt.%)	1.39
	Sulphur	S (wt.%)	0.66
	Oxygen	O (wt.%)	28.31
Proximate	Moisture content	M* (wt.%)	6.48
	Volatile matter	VM (wt.%)	81.71
	Ash content	A (wt.%)	0.54
	Fixed carbon	FC (wt.%)	17.75
Calorific	High Heating Value	HHV (MJ/kg)	28.39

As observed, DDG has high contents of carbon and hydrogen but low oxygen. Likewise, it contains high volatile matter along but low ash, nitrogen, and sulphur content. The results of the calorific analysis revealed that DDG has a higher heating value (HHV) of 28.39 MJ/kg. The high heating value of coals is typically ascribed to the high carbon, hydrogen, and low oxygen contents [24]. In comparison, the HHV of DDG is significantly higher than other Nigerian coals; Owukpa (26.51 MJ/kg), Amansiodo (27.48 MJ/kg) [18], Shankodi-Jangwa (27.22 - 27.37 MJ/kg) [20, 22]. The results indicate the high energy potential of DDG for future energy recovery applications. Furthermore, the HHV of DDG was adopted to predict the rank of the sample using the ASTM standard D388 [25]. Based on the HHV, DDG could be classified as high volatile bituminous C coal, which typically exhibits HHV in the range 26.70 MJ/kg - 30.20 MJ/kg. Based on the results of this study, DDG could be effectively utilised thermally for steam generation or metallurgical coke production for power generation or the manufacture of cement, iron, and steel. However, further tests are required to ascertain the rank, classification, and future applications of DDG.

3.2. Microstructure and chemical properties

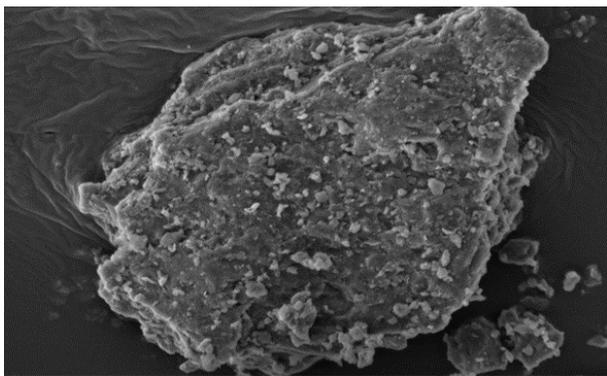


Figure 1. SEM Micrograph of Duduguru (DDG) coal

The SEM micrograph (magnification $\times 1000$) showing the microstructural and chemical properties of DDG are shown in Figure 1.

The results show that the morphology and microstructure of DDG revealed a solid but rough surface comprising various sized particles ranging from fine to coarse grains. As observed, the coarse particles are characterised by a distinct white sheen, whereas the small and finer particles showed a darker hue. According to previous studies [26-27], the white lustre is ascribed to the existence of quartz, kaolinite, and other metal-

based elements such as Ti and Fe typically found in the structure of coal. Therefore, the chemical composition of DDG was further examined by EDX analysis, as presented in Table 2.

As observed in Table 2, the EDX analysis revealed the presence of C, O, Mg, Al, Si, S, Ca, Ti, and Fe in the structure of DDG. The major elements detected were C and O with

atomic/weight compositions above 80% and 13%, respectively. The high carbonaceous nature of DDG is due to its rank classification as a high volatile bituminous class C coal. However, the O may be due to the presence of bonded oxygen groups in various forms as oxides of the metals Mg, Al, and Si typically present in coal samples.

Table 2. EDX chemical composition of DDG coal

Elements in coal	Symbol of element	Weight composition (Wt.%)	Atomic composition (wt. %)
Carbon	C	80.35	85.20
Oxygen	O	17.53	13.96
Magnesium	Mg	0.05	0.03
Aluminium	Al	0.46	0.22
Silicon	Si	0.52	0.23
Sulphur	S	0.59	0.23
Calcium	Ca	0.11	0.04
Titanium	Ti	0.21	0.06
Iron	Fe	0.17	0.04

The minor elements detected were; Mg, Al, Si, S, Ca, Ti, and Fe based on their low compositions detected below 1%. The elements Mg, Al, and Si, exist as oxides MgO, Al₂O₃, and SiO₂, whereas S and Ca exist as FeS₂ and Wollastonite. Lastly, Ti and Fe were detected in elemental form and could be responsible for the lustre of DDG as observed during SEM analysis.

3.3. Carbonization (pyrolysis) characteristics

The TG and DTG plots obtained based on the mass loss from the carbonisation studies are shown in Figures 2 and 3. Typically, TG analysis is performed to examine the thermal degradation behaviour and temperature profile characteristics (TPCs) of materials under various oxidative/non-oxidative along with isothermal/non-isothermal conditions. In this study, the effect of temperature and multi-heating rates was examined for DDG coal under non-oxidative (using nitrogen (N₂) gas flow) and dynamic conditions. The objective is to also simulate its thermal degradation behaviour and determine its characteristic temperature profiles during pyrolysis otherwise termed carbonisation.

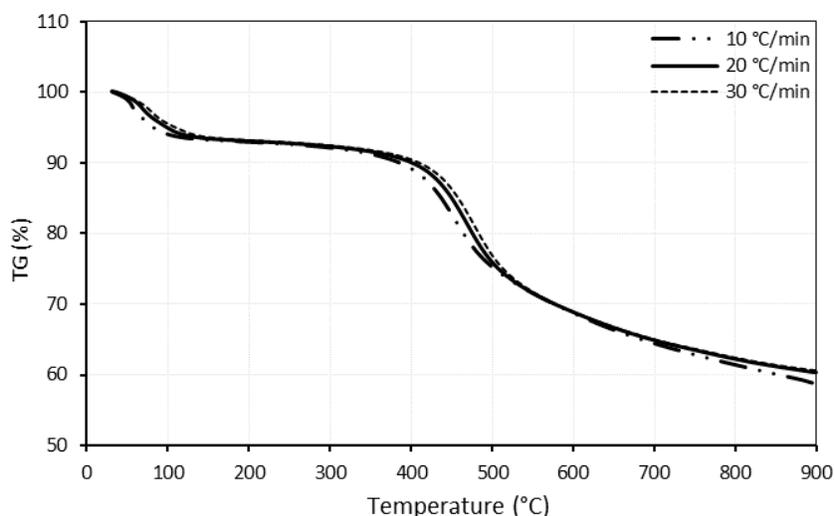


Figure 2. TG (%) Mass loss plots for carbonisation of DDG coal

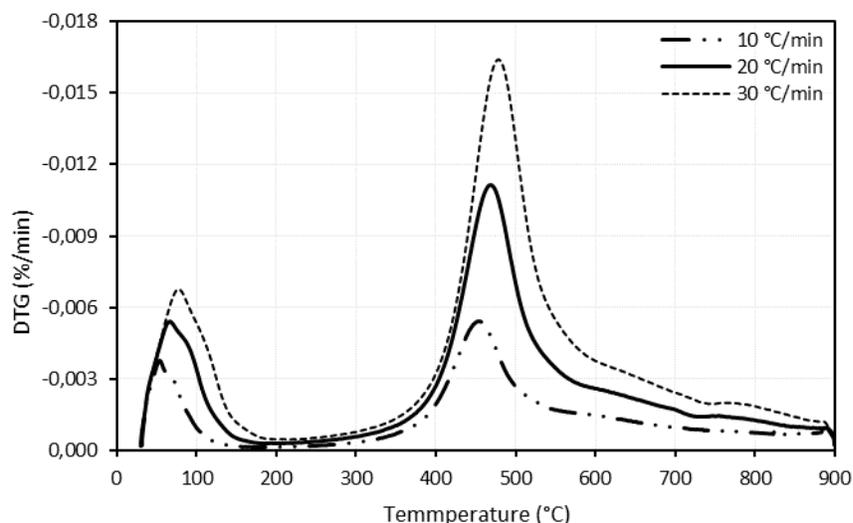


Figure 3. DTG (%) Mass loss plots for carbonisation of DDG coal

As observed, the effect of dynamic heating from RT to 900 °C resulted in the loss of mass of the sample during TGA. Furthermore, the results show that the increase in temperature and heating rates significantly influenced the thermal degradation behaviour of DDG coal, thereby resulting in mass loss from the initial condition of 100% to approximately 40%, on average for the heating rates. Typically, the higher heating rates tend to increase in the mass loss of coal during pyrolysis as reported by Shi et al., [28]. This observation is ascribed to an increase in the evolution of volatile matter, which occurs at higher heating rates during coal thermal analysis. Furthermore, the results showed that the increase in heating rates resulted in a shift of the TG plots to the right-hand side indicating distinct changes in the TPCs, as presented in Table 3. The shift in TG plots is reportedly ascribed to thermal lag that alters the time required for thermally degrading samples to attain equilibrium during TGA [29-30].

Table 3. TPCs for DDG coal based on TG plots

Heating rate (°C/min)	Onset temp. (°C, T_{ons})	Midpoint temp. (°C, T_{mid})	Endset temp. (°C, T_{end})	Mass loss (M_L , %)	Residual mass (R_M , %)
10	374.24	460.20	561.40	41.28	58.72
20	381.18	470.39	562.89	39.64	60.36
30	388.67	478.42	570.61	39.32	60.68

Table 3 shows that the onset (T_{ons}), midpoint (T_{mid}), and endset (T_{end}) temperature profiles for DDG all increased as the heating rates increased from 10 °C/min to 30 °C/min. As stated earlier, the shift in the TG plots to the right-hand side is as a result of the thermal lag, which results in distinct changes in the TPCs of the samples. In this study, the onset (T_{ons}) temperatures occurred in the range 374.24°C to 388.67°C or 381.36°C on average; whereas the midpoint (T_{mid}) temperatures were from 460.20 °C to 478.42°C or 469.67°C on average and lastly the endset (T_{end}) temperatures were from 561.40°C to 570.61°C or 564.97°C on the average. Furthermore, the change in heating rates also affected the mass loss (M_L) and the mass of residuals (R_M) after the TG analysis. As observed, the mass loss (M_L) decreased from 41.28% to 39.32%, which resulted in the mass of residuals (R_M) in the range from 58.72% to 60.68%. The results showed that the increase in heating rates from lower to higher values enhances the formation of a higher mass of residuals (R_M). Due to the non-oxidative nature of the thermal environment during TGA, the mass of residuals (R_M) could be reasonably used as an indication of the coke formed whereas the mass loss (M_L) denotes the volatiles (gas or liquid products) produced by the sample during carbonisation. Hence, the results indicate that the thermal degradation of DDG coal under non-oxidative or carbonisation conditions could

result in an average mass of volatiles of 40.08% and 59.92% coke. However, the coke potential of DDG needs to be determined by ASTM techniques.

Next, the degradation mechanism for the non-oxidative thermal analysis of DDG was examined by derivative thermogravimetric analysis (DTG). Figure 3 presents the DTG plots for DDG coal at various heating rates (10, 20, and 30°C/min) during TGA from RT to 900 °C.

As observed, the DTG plots for each heating rate was characterised by two sets of endothermic peaks with the temperature ranges from RT to 200°C and from 200°C to 600°C. As observed, the size of the peaks increased with increasing heating rate with smallest and largest peaks observed at 10°C/min and 30°C/min, respectively. Furthermore, the DTG plots indicate that the thermal degradation of DDG occurred in three (3) stages, namely; Stage I: from RT to 200 °C; Stage II from 200°C to 600°C, and finally stage III from 600°C to 900°C.

The degradation of DDG during Stage I could be ascribed to the drying or the loss of surface-bound moisture in the coal structure. In this study, the average mass loss in this stage was 5.69 %, which is in good agreement with the observed moisture content of 6.48% (Table 1). Stage II resulted in a significantly higher mass loss for the sample or an average of 25.46%, which is typically ascribed to the loss of volatile matter during thermal degradation of carbonaceous materials such as coal. As observed, the mass loss during this stage of TGA was significantly lower than the reported content of volatiles. This observation could be ascribed to the low reactivity of the DDG coal sample, resulting in only a partial loss of volatile matter. Finally, stage III resulted in an average mass loss of 8.93% thereby forming coke, fixed carbon, and some ash after thermal degradation of the DDG sample.

The effects of temperature and change in heating rates on the DTG plots was also examined through the temperature profile characteristics (TPCs), as presented in Table 4. The TPCs examined were the peak temperatures for drying (T_{DRY}) and devolatilization (T_{DEV}) along with the average rates of thermal degradation of the sample during the TGA carbonisation process.

Table 4. Temperature profile characteristics for DTG plots

Heating rate (°C/min)	Drying peak temp. (T_{DRY})	Rate (%/min)	Devolatilization peak temp. (T_{DEV})	Rate (%/min)
10	53.88	1.25	455.07	1.57
20	67.03	1.90	468.59	3.57
30	76.83	2.43	478.25	5.20

As observed, the peak temperatures for drying (T_{DRY}) and devolatilization (T_{DEV}) both increased with increase in the heating rates from 10 °C/min to 30 °C/min during the TGA carbonisation process. The findings reveal that the TPCs for DDG were significantly influenced by the change in TGA parameters. Furthermore, it can be reasonably inferred that heating the DDG sample between 53.88°C and 76.83°C or an average of 65.91°C could altogether remove surface-bound moisture from the sample. Similarly, the findings indicate that heating the samples between 374.24°C and 570.61°C can effectively devolatilize the sample based on the devolatilization (T_{DEV}) values from 455.07 °C to 478.25°C. Overall, the findings indicate that DDG is not highly reactive and will require higher temperatures to undergo complete devolatilization or thermal degradation into gas or liquid fuels during thermal conversion. In addition, the findings indicate that DDG is not low-rank coal since such coals are characterised by high reactivity during thermal conversion. Lastly, the DDG characteristics indicate it is also suited for coke production or solid products from carbonisation.

3.4. Kinetic degradation characteristics (Kissinger)

Kissinger Kinetic Model plots for the non-isothermal and multi-heating rate TGA carbonization of DDG are shown in Figures 4 and 5. The Kissinger kinetic plots for the drying and devolatilization stages were calculated from the maximum drying and devolatilization peaks deduced from Table 4.

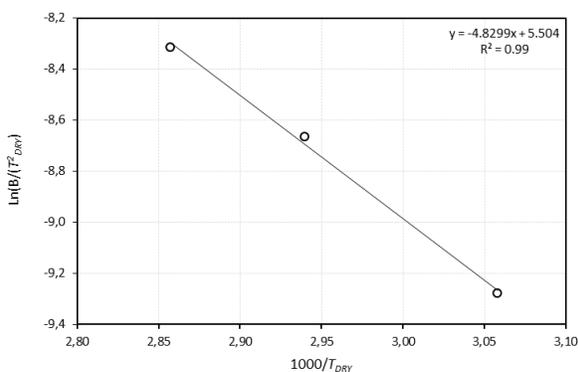


Figure 4. Kissinger kinetic plots for Drying DDG Coal

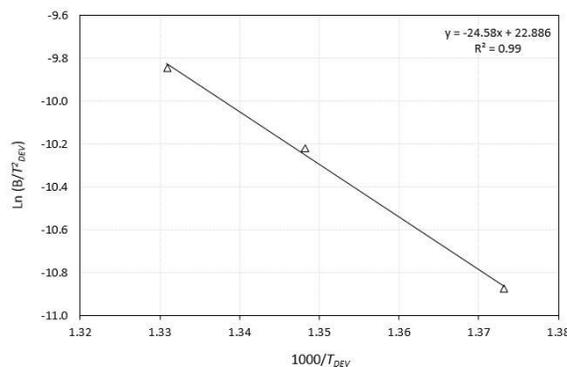


Figure 5. Kissinger kinetic plots for Devolatilization of DDG Coal

Based on the plot in Figure 4, the activation energy, E_a for drying DDG coal is 40.16 kJ/mol, whereas the frequency factor, A is $1.19 \times 10^{03} \text{ min}^{-1}$. Likewise, the kinetic parameters for devolatilization were computed based on Figure 5. Therefore, the activation energy, E_a for the devolatilization of DDG is 204.36 kJ/mol, whereas the frequency factor (A) is $1.54 \times 10^{11} \text{ min}^{-1}$. The values for the kinetic parameters for drying and devolatilization of DDG were computed at high $R^2 = 0.99$, respectively. In comparison, the kinetic parameters; activation energy, $E_a = 40.16 \text{ kJ/mol}$ and frequency factor, $A = 1.19 \times 10^{03} \text{ min}^{-1}$ for drying DDG coal is higher than the $E_a = 28.86 \text{ kJ/mol}$ and $A = 5.97 \times 10^{00} \text{ min}^{-1}$ deduced for Owukpa coal. Furthermore, the values of $E_a = 204.36 \text{ kJ/mol}$ and $A = 1.54 \times 10^{11} \text{ min}^{-1}$ for the devolatilization of DDG is higher than the $E_a = 57.29 \text{ kJ/mol}$ and $A = 9.86 \times 10^{00} \text{ min}^{-1}$ deduced for Owukpa coal in the literature.

Overall, the results indicate that DDG is not a highly reactive coal, as earlier deduced, under the thermal conditions examined in this study. Based on the physicochemical and thermal properties of DDG described in this study, it can be reasonably inferred that DDG is a high-rank coal. Hence, DDG could be potentially utilized for steam generation in power generation or metallurgical coke production for the manufacture of cement, iron, and steel in the industry.

4. Conclusions

The physicochemical, calorific value, microstructure, thermal, and kinetic fuel properties of the newly discovered Duduguru (DDG) coal from Nigeria were characterised and presented in this study. The results showed that DDG has a high calorific value of 28.39 MJ/kg, which can be ascribed to its high carbon, hydrogen, and low oxygen contents. Based on its volatile matter content, DDG could be ranked as a high volatile bituminous C coal. The microstructure and chemical analyses of DDG indicated it is characterised by fine to coarse grain-sized particles with a distinct white sheen, which could be due to the presence of quartz, kaolinite, and other metal-based elements. The thermal analysis results indicated that the variation of temperature and heating rates during the TGA carbonisation significantly influenced the thermal degradation behaviour and temperature profile characteristics. Hence, DDG experienced significant mass loss (ML = 39.32% to 41.28%) and residual mass (RM = 58.72% to 60.68%). Overall, the results indicate that DDG is a high-rank coal with potentials for use in steam generation or metallurgical coke production for power generation and the manufacture of cement, iron, and steel.

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