



Research Article

**Demineralization and Characterization of Coal Deposit
Found in Maiganga, Akko Local Government, Gombe
State, Nigeria**

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ABSTRACT

The characterization of coal provides valuable information on its composition, environmental effects, and applications. This study investigates the leachate elemental composition, physicochemical properties, morphology, mineralogy, functional groups, and thermal behaviour of the demineralised and demineralized coal deposits in Maiganga, Gombe State. Three modes of coal demineralization including mixed acid-alkali, acid, and alkali demineralization were tested. The quantified carbon indicates the hierarchy of leachates in order; HCl+ NaOH Washed > NaOH Washed > HCl Washed (70.38% > 64.42% > 60.39%). The microstructure images showed crystallites on the coal matrix indicating the presence of mineral phases found abundant in the demineralised sample compared to the demineralized coal. Also, the compositions of demineralised coal appeared in oxide form while that of demineralized coal is elemental because the chemical treatment effects during demineralization dissolve the oxides. The FTIR spectra demonstrate different aliphatic -CH bonds in both samples indicating the Maiganga coal as sub-bituminous coal with high carbon content. The phases in the demineralised coal sample are SiO₂, PbCO₃, CaO, and Mg₂Al₂SiO₅(OH)₄ and for the demineralized sample, the phases are Ca₄I₈ O₂₄, SiO₂ and PbCO₃. For efficient high-energy recovery the temperature of >848.2 °C is required as demonstrated by the two samples in thermal analysis, the Temperature Profile Characteristics (TPC) for the samples differ. Furthermore, the comprehensive combustion characteristics parameters indicated a higher and better combustion performance by the demineralized coal (2.4 x 10⁻⁷ min⁻² C-3) compared to the undemineralized coal (1.0 x 10⁻⁹ min⁻² C-3) sample. This finding demonstrated that mining the Maiganga coalfield may not pose threats to humans and the environment, and the coal is suitable for electricity generation.

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INTRODUCTION

Coal is a sedimentary rock composed of three types of materials: organic carbonaceous matter (macerals), non-combustible minerals from ash, and fluids (Behera *et al.*, 2018). It is a fossil fuel that is used in electricity generation, firing kiln for cement production, iron and steel production, raw material for numerous applications (Ryemshak and Jauro, 2013; Nyakuma *et al.*, 2021) and many other applications, however, there are high levels of ash and sulfur in raw coal (Meshram *et al.*, 2015). But low ash content is needed to increase handling and burning capacity, decrease handling costs, and improves combustion and boiler efficiency while low sulfur is required since high sulfur content is associated with acid mine drainage, chimney corrosion and other equipment such as air heaters (Usman *et al.*, 2019).

The effect of coal combustion on the environment includes smog, the greenhouse effect, and acid rain (Lu *et al.*, 2017; Silas *et al.*, 2018), the release of unfriendly trace elements into the environment (Okoro, Asadu, and Onoh, 2018) and other deleterious effects (Mukherjee and Borthakur, 2003). The removal of mineral matter bounded in the coal matrix by alkali and/or acid solution and dissolution in water (leaching) is effective (Behera *et al.*, 2017) for eco-efficiency, cleaner production, and resource recovery (Chareonpanich *et al.*, 2011). It was claimed that a successful sequential two-stage alkali-acid leaching eliminated these contaminants from coal to a great extent (Deb Barma *et al.*, 2021) and the dominant ash impurities in high-ash coal are silica (SiO_2) and alumina (Al_2O_3).

Demineralization of Enugu coal was reported (Okoro *et al.*, 2018), and the filtrate was analyzed with Atomic Absorption Spectrophotometer (AAS), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectrophotometer (EDX), X-ray Fluorescence (XRF), and Fourier Transform Infrared

Spectroscopy (FTIR) however, the mineralogy and physiochemical properties of the coal were not reported. The reduction of the ash and sulphur contents of the Nigerian Akunsa coal was investigated (Shi *et al.*, 2020) without reporting any characterization of the filtrate. In another study, multiple steps of proximate/ash composition analysis, hot aqueous leaching de-ashing with water and sodium carbonate were performed on Nigerian Lafia-Obi coal. The findings revealed that ash content was reduced to about half compared to the as-received coal (Adeleke *et al.*, 2011).

No study compared the demineralized and undemineralized Maiganga coal since before use, the coal needs to be demineralized to make it environmentally safe in application. By efficiently reducing ash content and removing deleterious minerals from coal, difficulties with gasification, carbonization, liquefaction, combustion efficiency, and airborne pollutant emissions are avoided. According to (Behera *et al.*, 2018), the cleaned coal is used in the generation of ultra-clean fuel for various applications such as; in an internal combustion engine (ICE), in direct firing in the gas turbine with reduced pollution emissions, or as fuel when mixed with LNG and used in a diesel engine. Moreover, the demineralization of coal improves low-grade coal by eliminating the mineral content, resulting in properties that are like those of high-rank coal. Furthermore, for optimal coal consumption, characterisation and quality are critical in determining their appropriate applications (Khan and Bibi, 2016; Ryemshak and Jauro, 2013). The Maiganga coal lies within the Gombe sandstone, adjacent to and above the Pindiga formation meanwhile, the Gombe sandstone is the formation that hosts most of the coal deposits found in Gombe State, especially that of Maiganga (Rawat and Yadav, 2020). The evaluation of coal resources for energy production for various industries can

improve the economics of the local communities, the state government and the country at large.

The present work studied the effect of chemical demineralization on the Maiganga, Gombe coal following the characterization and comparison of the raw and demineralized samples by ultimate and proximate analyses, Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy, Energy Dispersive X-ray Fluorescence Spectrophotometer, X-ray diffractometer, and thermogravimetric analysis.

MATERIALS AND METHODS

Sample preparation

The coal sample was obtained at Maiganga mining coalfield, Kibuwa community which covers an area of about 20,129.47 Acres (48.16 Km²) (Amosu *et al.*, 2021) located between Latitude 10° 02' to 10°05' and Longitude 11 ° 06' to 11 ° 08' in Akko Local Government Area of Gombe state, Nigeria. The coal sample was washed with distilled water several times and dried in a vacuum oven at 110 °C for one hr. Using a sieve shaker, the cleaned coal is

RESULTS AND DISCUSSION

Result of leachate elemental composition

Flue gas is generated when coal is combusted and it is estimated to be composed of SO₂: 0.2%, NO: 0.1%, CO₂: 10% (Zhou *et al.*, 2012). These

pounded in a pestle and mortar and screened through 75 mm sieves.

Coal demineralization process

Demineralization of coal was performed in three samples of 1 L reaction flask containing 50 g of prepared coal sample added to 500 mL HCl and NaOH. For the acid-alkali procedure, NaOH demineralization was initially carried out followed by the HCl. The acid, alkali and acid-alkali coal samples are denoted as HCl Washed, NaOH Washed and HCl + NaOH Washed coals. The stirring operation at 400 rpm and reaction temperature of 65 °C for 2 hr were used, after leaching the hot coal slurry is cooled, filtered with man filter paper No. 1 and washed with distilled water several times, the procedure is modified from literature (Pan *et al.*, 2021). The demineralized residual coal retained on the filter paper was dried in an oven at 100 °C for 2 hr and then utilized for analyses while the leachate was analyzed for sulphur, nitrogen and carbon using an Atomic Absorption spectrometer (AAS). The coal residue from HCl + NaOH Washed was used to perform the other characterizations.

compositions constitute environmental problems hence, the elements Carbon, Sulphur, and Nitrogen were analyzed based on the AAS analysis after demineralization. Figure 1 shows the AAS result of the leachate.

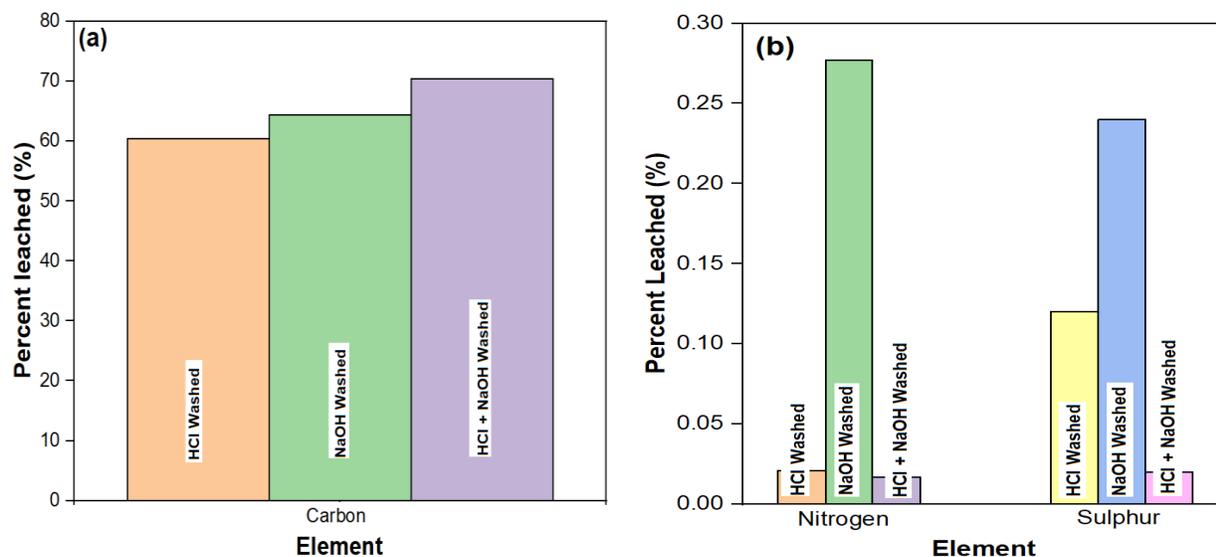
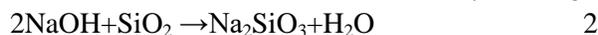


Figure 1: AAS results of (a) Carbon leachate (b) Nitrogen and sulphur leachates

The quantified carbon (in percent) indicates high to low hierarchy of leachates in order; HCl + NaOH Washed > NaOH Washed > HCl Washed (70.38>64.42>60.39). This result signified that the combined effect of the acid and alkali in demineralizing the Maiganga coal sample with

$$\text{Degree of demineralisation (\%)} = \frac{\text{Ash content (raw coal)} - \text{Ash content (leached coal)}}{\text{Ash content (raw coal)}} \times 100\% \quad 1$$

The degrees of demineralization calculate for alkali demineralization is 60% which signified a good demineralization performance. The results revealed a significant reduction in ash concentration of around 62.5%. The capacity of alkali to infiltrate the interior of the coal matrix and react with silica, alumina, and clay-bearing



Effective mineral removal is observed with alkali demineralization compared to acid demineralization this finding agrees with other study (Dhawan and Sharma, 2019). The alkali The proximate analysis revealed that eliminating mineral matter from coal demineralization has a high potential as indicated by other studies (Manoj and Elcey, 2010; Manoj *et al.*, 2009; Okoro, Asadu, and Maxwell, 2018; Usman *et*

respect to carbon surpasses the individual acid and alkali demineralization. The degree of demineralization determines the reduction in mineral content in chemical leaching of coals. The percentage of demineralization is (Dhawan and Sharma, 2019).

minerals, resulting in hydrated alkali-bearing silicate, aluminate, and aluminosilicate complexes, also is reason for significant alkali demineralization, similar reason was reported (Behera *et al.*, 2018). Demineralization mechanism follows as;

demineralization showed higher nitrogen and sulphur leachate compared to the other two treatments.

Results of proximate and ultimate analyses *al.*, 2019) and is appropriate coal for solid fuel combustion. The proximate and ultimate analyses of the undemineralization and demineralization coal samples are presented in Table 1.

Table 1: proximate and ultimate analyses of the undemineralization and demineralization

Fuel property	Proximate analysis		Ultimate analysis		
	HCl + NaOH Washed demineralized coal (%)	undemineralized coal(%)	Fuel property	HCl + NaOH Washed demineralized coal (%)	undemineralized coal (%)
Moisture content	1	5.8	Carbon	73.15	69.48
Ash content	5	8	Hydrogen	4.34	5.22
Volatile matter	20	43.91	Oxygen	3.04	10.56
			Nitrogen	0.61	1.22
Fixed carbon (FC)	74	42.16	Sulphur	0.08	0.13

After the demineralization, the carbon content increased when compared to the undemineralized coal however, the other fuel properties decreased for the ultimate analysis. Low sulphur content as depicted by the demineralized coal signifies that the solid fuel can be used to generate power with reduced emissions to the environment (Behera *et al.*, 2017). Calorific value required for generating electricity falls within the range 2700 - 9500 Kcal/Kg (Usman *et al.*, 2019). The Maiganga undemineralized coal has the calorific value of 5179.80 kcal/kg while demineralized coal calorific value is higher 5210.38 kcal/kg, this result depicts that the demineralization of the coal is beneficial since the higher is the calorific value signifies the better the quality of the coal (Behera *et al.*, 2017) and a major term for heat generation capacity (Usman and Maitera, 2015).

Result of SEM images

On the coal matrix, a SEM image shows a scattered network of distributed small crystallites, indicating the existence of mineral phases (Behera *et al.*, 2017). In other studies, it was reported that (Okoro *et al.*, 2018) The presence of lithophytes such as magnesium, aluminum, silicon, calcium, titanium, and siderophile, such as iron, is indicated by the bright luminosity, while the presence of chalcophiles such as lead, chromium, cadmium, arsenic, mercury, copper, zinc, and other metals is indicated by the dark luminosity. Figure 2 shows the SEM micrographs at different magnification the undemineralized and demineralized coal.

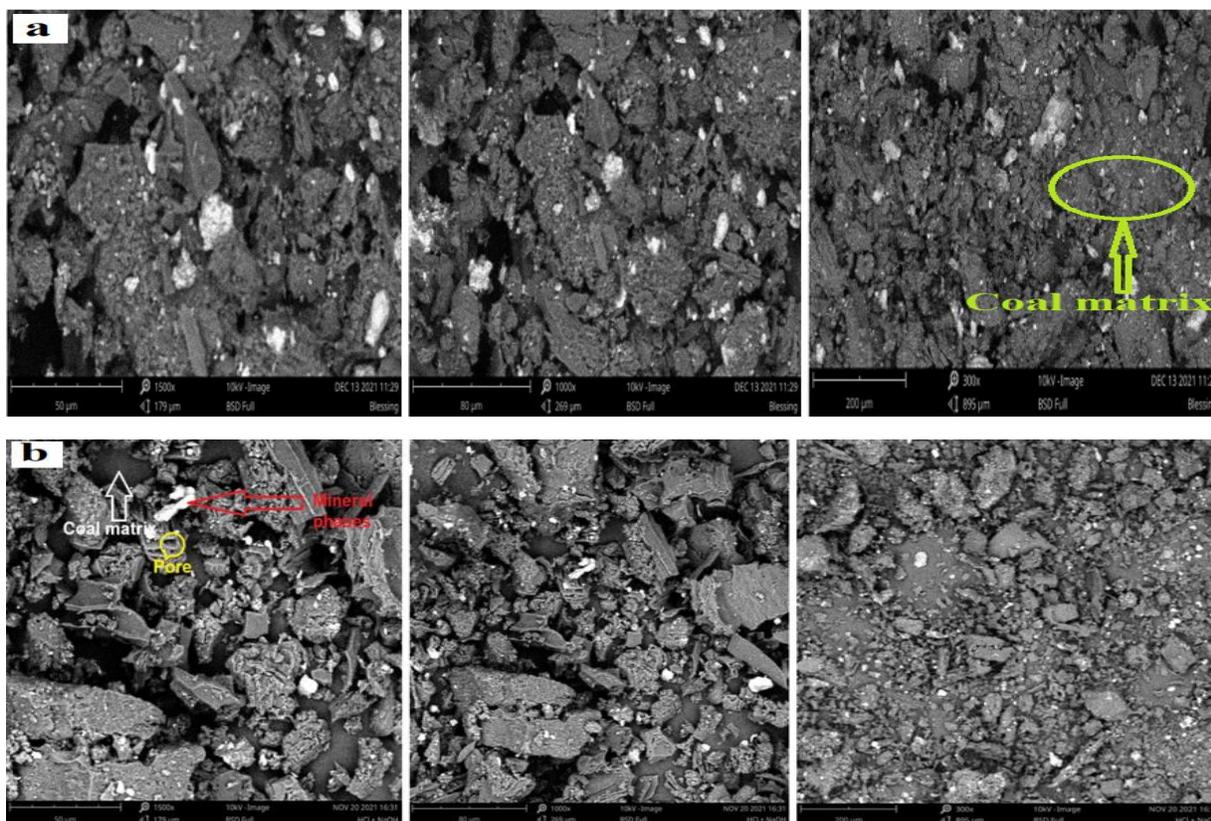


Figure 2: SEM micrographs at different magnification (a) Undemineralized coal at 50, 80 and 200 μm (b) Demineralized coal at 50, 80 and 200 μm .

The presence of pores in coal is known (Song *et al.*, 2016) and might have been improved due to the demineralization in agreement with other studies (Manoj *et al.*, 2009; Rizkiana *et al.*, 2019). The raw coal (Figure 2a) morphology showed irregular shapes of spheres, tablets etc. The compositions of demineralized coal are elemental (not in oxide form), the acid effect during demineralization desolutes the oxides, this finding agrees with the literature (Baruah *et al.*, 2006; Behera *et al.*, 2018; Khan and Bibi, 2016). Most of the undemineralized coal appeared in

and largely the coal matrix also, it can be observed that the crystallites are found in abundance compared to the demineralized coal (Figure 2b) because of chemical reaction during the demineralization.

Result of chemical composition

oxide form Fe, Si, Al, S, Ca, Ce, Mg, Ti and Mn were detected as major elements while the trace elements were V, Cu, P, As, K, Cr, Pb, Ga, Ni, Cl, Br, Sr, Ta, W, Ag, Nb, I, Zn, Th. Figure 3 shows the elemental compositions, major elements ($\% > 0.5$) and minor elements ($\% < 0.5$).

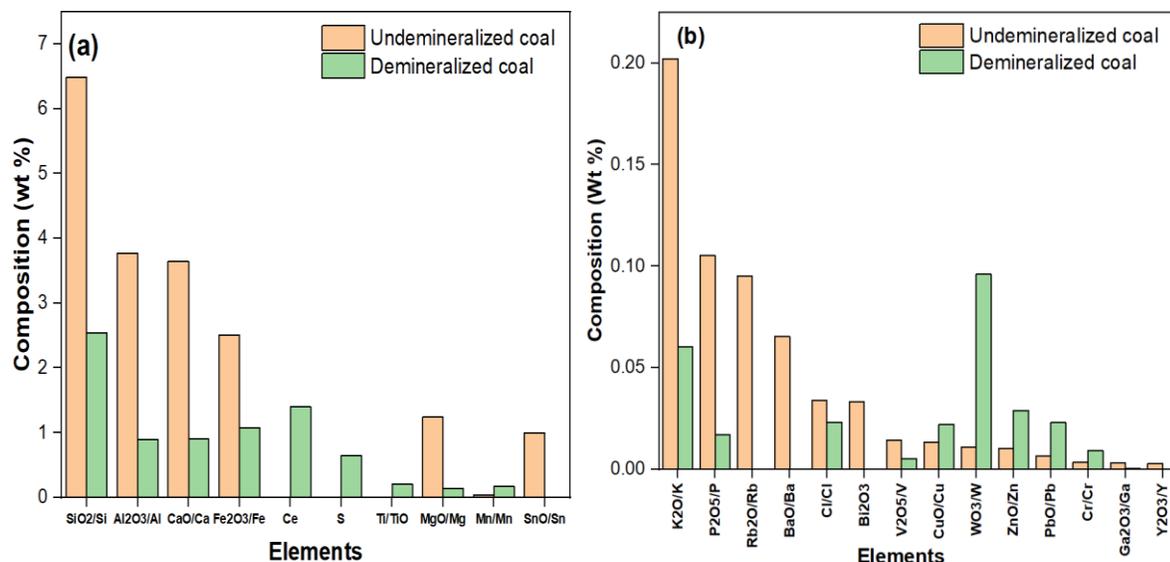


Figure 3: Elemental composition by EDXRF of un/demineralized coal (a) Major and (b) Minor elements

Potentially Hazardous Traces Elements (PHTEs) are Pb, Ni, Cd, Mn, Cr, these elements are found as traces and below the impactful level to the environment as indicated by Table 2 hence, the

Mainganga coalfield activities does not pose threats to human and the environment, similar conclusion was reported (Tikpangi, 2016).

Table 2: Elements and traces in the undemineralized and demineralized coal

Undemineralized	PHTEs (%)	Demineralized	PHTEs (%)	Regulatory level (Baruah <i>et al.</i> , 2006) (Tikpangi, 2016)
NiO	0.0092	Ni	0.0	1.8
MnO	0.0343	Mn	0.1655	0.5
CdO	0	Cd	0	2
Cr ₂ O ₃	0.00342	Cr	0.0092	0.2

Result of FTIR spectra

The peaks at 1072 and 1383, cm^{-1} assigned to Si–O stretching vibrations indicates the presence of quartz and cerrusite, the absorption at 1320–1210 cm^{-1} is stretching vibration (C–O) of phenol and ether (Bora *et al.*, 2021; Rawat and Yadav, 2020; Silas *et al.*, 2019), the stretching band of aromatic compounds can be observed at 1615 cm^{-1} peak (Boral *et al.*, 2021), 2800–3000 cm^{-1} is due to the stretching vibration aliphatic and alicyclic ($-\text{CH}_3$, $-\text{CH}_2$ and $-\text{CH}$) groups carbon where different aliphatic $-\text{CH}$ bonds is

associated to sub-bituminous coal (Manoj and Elcey, 2013; Rodrigues *et al.*, 2020). According to (Nyakuma *et al.*, 2021), sub-bituminous coals have the potential to be liquefied and used to make synthetic crude oil and tar. The observed peak at 1604 cm^{-1} (hydrogen-bonded C=O) indicates high carbon content in coal and 2356 cm^{-1} can be assigned to the asymmetric stretching vibrations of the isocyanate (C=N=O) group (Akinyemi *et al.*, 2020). Figure 4 is the FTIR spectra of undemineralized and demineralized coal.

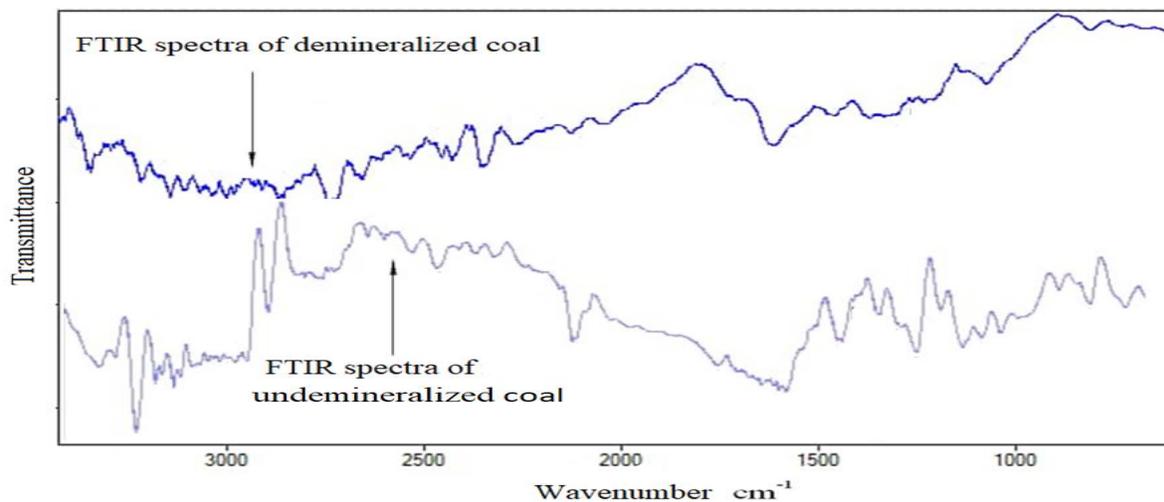


Figure 4: FTIR spectra of undemineralized and demineralized coal

The strong absorption peak at 3346 is ascribed to hydroxyl (-OH) group stretching vibration and may be presence of moisture, phenol and alcohol (Jiang *et al.*, 2019). Also, associated with -OH are the indication of clay minerals including montmorillonite, kaoline, illite, and halloysite (Sonibare *et al.*, 2010). The peak at 1435 cm^{-1} is ascribed to stretching vibration of carbon and oxygen (Song *et al.*, 2016). The

Result of phases

Based on the mineral study of the undemineralized coal, the sample is amorphous materials with characteristic of amorphous halo centered at $2\theta = 23^\circ$, and a reflection of the plane (002) as shown in Figure 5a and the

peaks between 900 and 700 cm^{-1} are attributed to various bands related to aromatic, out-of-plane C-H bending, and -OH groups with varying degrees of substitution, whereas the change in total areas of the alkane group of raw and demineralized coals indicated the replacement of aliphatic side chains by nitro (-NO_2) groups of aromatic compounds found in the range of $1550\text{--}1490\text{ cm}^{-1}$ (Boral *et al.*, 2021).

phases analysis are quartz low (SiO_2), cerussite (PbCO_3), lime (CaO), and amesite ($\text{Mg}_2\text{Al}_2\text{SiO}_5(\text{OH})_4$) with the corresponding diffraction angles of 26.6° , 25° , 32.02° and 36.6° respectively.

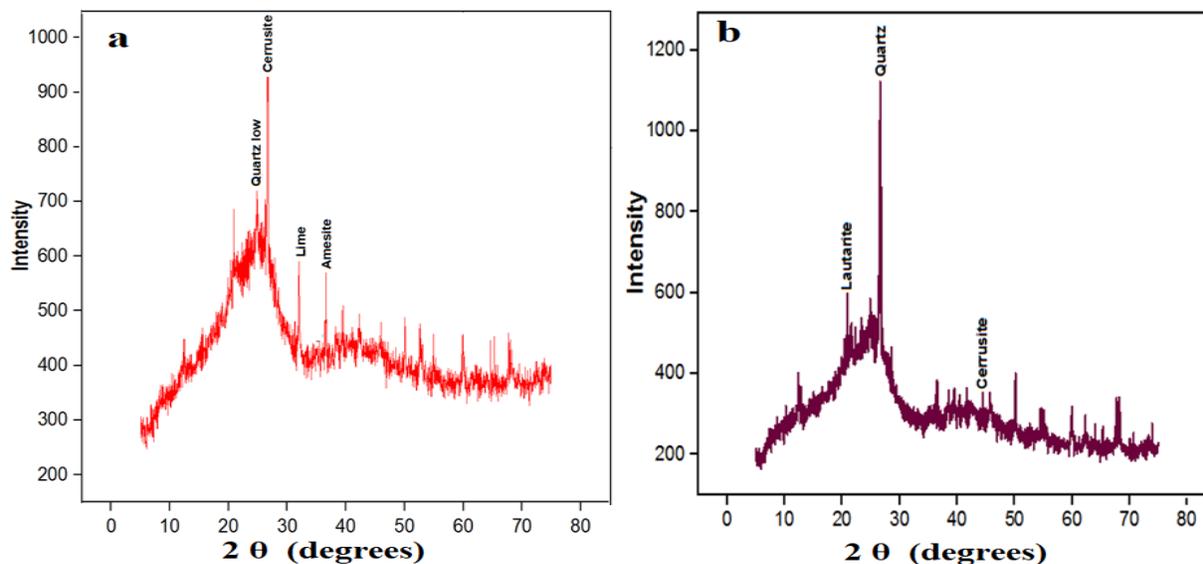


Figure 5: XRD patterns of (a) Undemineralized coal (b) Demineralized coal

The various demineralized coal sample showed the phases Lautarite ($\text{Ca}_4\text{I}_8\text{O}_{24}$), Quartz (SiO_2) and cerrusite (PbCO_3). Few crystals that are similar to graphite existed in the coal as indicated by the Gaussian peaks which consists of γ – band (at 22° , the (002) band exist) and Π -band (d_{002}). γ – band ranges from $5\text{-}33^\circ$ where the areas under the curve left of (002) band constitute the aliphatic chain and the right side constitute the aromatic chains. Π -band is in the region from $40\text{-}50^\circ$ (100) band. Furthermore, the high background intensity observed indicate the amorphous carbon found in coal, this finding agrees with the literature (Lu *et al.*, 2017; Song *et al.*, 2016).

TGA result

The Maiganga coal samples is thermo-chemically reactive under oxidative condition since TG-curve indicate $>90\%$ weight loss and $<10\%$ residual mass. Furthermore, the TG-curve shape signified a steady thermal degradation where Mass Loss (M_L , %) of 94.6 % and Residual Mass (R_M , %) of 5.4 % for the undemineralized coal and the Temperature Profile Characteristics (TPC) were deduced (ignition T_{it} , peak loss, T_{max} , and burnout temperature T_{ot}). The combustion behaviors of demineralized and undemineralized coal through the Thermal decomposition (TG) Figure 5a and the Derivative Mass Loss (DTG) Figure 6 are presented where the data were plotted against temperature.

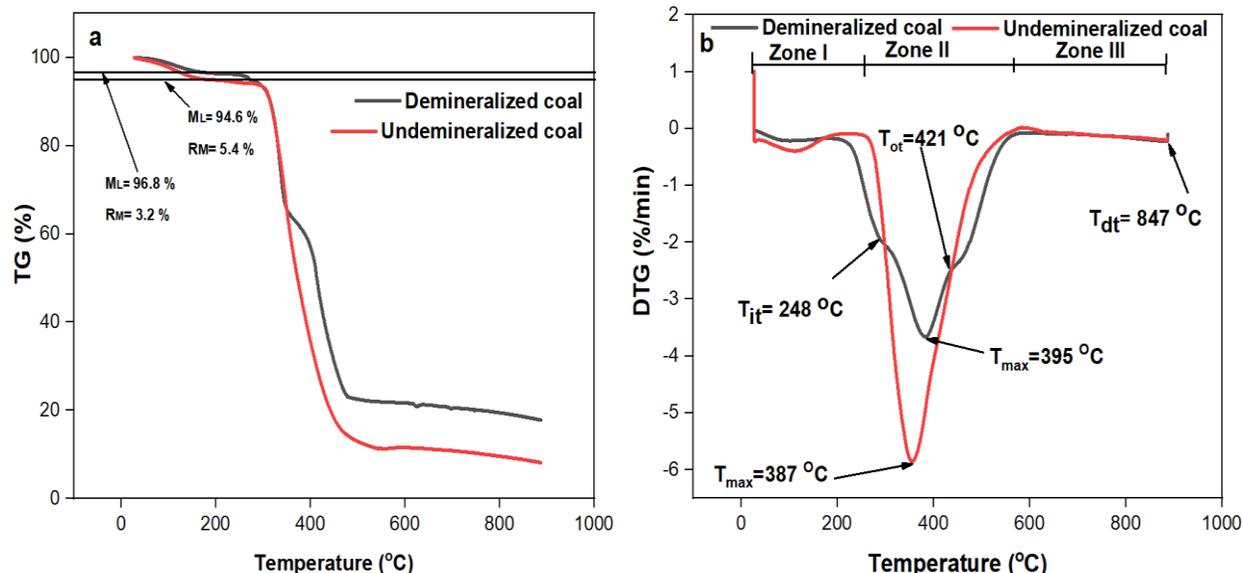


Figure 6: Plots for (a) TG combustion profile and (b) DTG combustion profile of Maiganga coal

The TG-Temperature plot depicts that there is organic fraction (maceral) degradation with increase of heat from 0-1000 °C signifying the conversion (breaking reaction) of the coal sample resulting to energy recovery or coal products. The TG-plot indicates a general thermal degradation of maceral or organic components of the coal from about 30-848.2 °C and >848.2 °C is required to decompose the Maiganga coal to obtain an efficient and high-energy recovery.

The DTG-temperature plot for demineralized shows three zones of thermal effect on the coal, SZone I reveal weight loss due to loss of moisture content (30-200 °C), Zone II, weight

$$S = \frac{K_{max}K_{mean}}{T_{ot}T_i^2} \quad 4$$

Where, the maximum (K_{max}) and average (K_{mean}) are the combustion rates. Table 3 shows the calculated values of combustion

Table 3: Calculated values of combustion performance

Coal sample	T_i (°C)	T_{max} (°C)	T_{ot} (°C)	K_{max}	K_{mean}	$S /(\text{min}^{-2}\text{C}^{-3})$
Undemineralized	285	387	426	-5.8	-3.07	1.0×10^{-9}
Demineralized	248	395	421	-3.7	-2.04	2.4×10^{-7}

loss due to organic content or volatile matter (200-589 °C) and Zone III, weight loss due to residual organic degradation or slow coke formation (589-847 °C). The reactivity of coal deduced from the DTG plots for the undemineralized coal which shows the three stages in the combustion process; drying at 30-200 °C, devolatilization at 200-500 °C and coke formation at 500-848.2 °C.

The comprehensive combustion characteristics parameter S , was used to compare the combustion characteristics of the undemineralized and demineralized coal sample. Coal sample with a higher S value have better combustion performance (Ge *et al.*, 2015):

performance for the undemineralized and demineralized coal sample.

The S-parameter further indicate that the demineralized coal sample is suitable for heat generation capacity and agrees with the calorific value, elemental and chemical finding.

CONCLUSION

This study showed that the Maiganga coal deposit is a Low-Rank Coal (LRC) and suitable for electricity generation. From the proximate and ultimate analyses, it was observed that demineralization could improve the fuel property also, the combined effect of the acid and alkali in demineralization with respect to carbon is better than the separate acid and alkali demineralization as indicated by AAS results. The presences of crystallites are found in

abundant in the undemineralized coal compared to the demineralized coal due to chemical reaction during the demineralization. Furthermore, the elemental result depict that the Maiganga coalfield activities does not pose a threat to human and the environment because all the PHTEs are below the regulatory levels. FTIR spectra showed different aliphatic –CH bond that is associated to sub-bituminous coal. Quartz and cerrusite are the similar phases found between the undemineralized and the demineralized coal while the demineralized coal has higher combustion performance and it is suitable for heat generation capacity.

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