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Authors' contributions

This work was carried out in collaboration between both authors. Author OYA designed the study, managed the literature searches, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author ANO managed the analyses of the study and supervised the overall output of the study. Both authors read and approved the final manuscript.

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ABSTRACT

Proximate and ultimate analysis have been carried out on cretaceous coal samples from Emewe Efopa in the Northern Anambra Basin of Nigeria to determine its mineralogical composition and chemical characteristics. The analysis indicates that the coal, on average, contains 25.5% ash, 6.33% moisture, 27.33% volatile matter, 40.83% fixed carbon. It also contains 0.72% nitrogen, 0.04% phosphorus, 63.92% organic carbon, 0.91% sulphur, 3.25% hydrogen. Geophysical investigation using vertical electrical sounding (schlumberger array) reveals that the study area has six geoelectric layers: sandstone, claystone, shale, coal, claystone, claystone respectively. The coaly layer has a resistivity of 31104Ω. The integration of the different studies carried out in the study area has aid in the ranking of the coal deposits to be a low ranked sub-bituminous type.

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Keywords: Anambra basin; coals; geochemical analysis; geophysical investigations; sub coal.

1. INTRODUCTION

The area of study (Emewe Eforpa) belongs to the Northern Anambra Basin as represented in Fig. 1. The Cretaceous Anambra basin covers an area of about $40,000 \text{ km}^2$ in southeastern Nigeria [1]. It is located at the southwestern part of the Benue Trough (Fig. 1). It is the structural link between the Cretaceous Benue Trough and the Tertiary Niger Delta [2]. The area has attracted numerous studies such as several authors [3-9], recorded that Nkporo Shale consists of dark fossil shales and mudstones with occasional thin beds of sandy shale and sandstone. Thin bands of shaly limestone may be present. Fig. 1. The Cretaceous Anambra basin covers
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According to Nwajide and Reijers [8], investigation of the Anambra Basin's fill in terms of sequence stratigraphic analysis has only recently started. They stressed that repeated allocyclic incursions of the sea into the Anambra Basin resulted in characteristic basin-wide

genetic sequences or parasequence sets. Only two allocycle events, however, have so far been recognized in the Anambra Basin in contrast to the better-studied Niger Delta with a more complete log record, where at least eleven events are reflected by a repetitive pattern of transgressive-regressive lithologies [statistical analysis of the literature review shows that more than ninety percent of the studies so far in the basin is in the southeast section [10-13]. genetic sequences or parasequence sets. Only
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Geologic exploration for coal led to the discovery of coal in the Mamu Formation (formally called the "Lower coal measures) of the Anambra Sedimentary Basin of south Eastern Nigeria, with test drillings and later mining operations in Enugu. The formation became the subject of many reports. statistical analysis of the literature review
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Coal beds are widely deposited within the sedimentary succession of Benue Trough of Nigeria [14].

Fig. 1. Map of sedimentary basins of Nigeria showing the location of Anambra basin [16]

Quality assessments have been Carried out on coal samples from Okaba in the Anambra Basin of Nigeria principally to determine its mineralogical composition, chemical characteristics and its cokability or otherwise. The Okaba coal, (close proximity to the study area) on the average, contains 4.53% clay minerals, 6.18% pyrite, 2.94% carbonate and 1.66% quartz and (by difference from 100) contains 84.70% macerals [15]. Chemical analysis indicates that the coal, on the average, contains 12.51% moisture, 11.48% ash, 47.49% volatile matter and 28.53% fixed carbon. It contains 71% sulphur, 76.36% organic carbon, 5.95% hydrogen, 0.81% nitrogen, 10.46% oxygen and 0.05% phosphorus, and has a low free swelling index (FSI) of 0.5 [15]. Significant amongst these coal deposits are those within the Mamu Formation in the Okaba area of the Lower Benue Trough.

The scope of this study includes field geological mapping of the area on a scale of 1:25,000, petrographic and megascopic observation of the rock types, geochemical analysis of rock samples collected and geophysical investigation of coal in order to determine the depth and thickness of the coal bed and other rock layers in the study area.

2. GEOLOGIC SETTING OF THE BASIN

The studied area lies in the Anambra Basin. The basin is a NE-SW trending syncline that is part of the Central African Rift System which developed in response to the stretching and subsidence of major crustal blocks during a lower Cretaceous break-up phase of the Gondwana supercontinent [17]. The movements were reactivated by further plate activity in lower Tertiary soon after the intermittent Upper Cretaceous rifting [17]. The separation of the African and South American plates left the Benue Trough as an Aulacogen, a failed arm of an RRR Triple Junction [18,19,9].

The basin is an expensive west and Central African rift system in which it opened as an extensive sinistral wrench complex [20-22]. Based on the work done by Murat [5], Southern part of the Benue Trough was interpreted to have a longitudinal fault with its eastern half subsiding preferentially to become the Abakaliki depression. There was the differential in the rate of subsidence in the Southern Benue Trough e.g high in Pre-Albian time, low in lower Cenomanian and very high in Turonian; the

latter was an important phase of platform subsidence [23].

The period of subsidence in Southern Benue Trough corresponds to the time of the initiation of the Anambra Basin, which started during the Coniacian and reached its peak at the Santonian thermotectonic event [24]. Several authors [5,25- 27,8,28,29] demonstrated that the Santonian tectonic pulses dating back to 84Ma, was associated with intensive magmatism. Folding and faulting which resulted in Abakaliki area becoming flexurally inverted to form the Abakaliki Anticlinorium.

Deposition of sediments in the Anambra Basin commenced during the Campanian, with Nkporo Shale, Enugu Shale and Owelli Sandstone which is regarded as the Nkporo Group, constituting the basal beds of the Campanian period. The campanian was a period of short marine transgression and regression, the shallow-sea later became shallower due to subsidence, thereby resulting in a regressive phase during the Maastrichtian which deposited the flood plain sediments and deltaic forests of Mamu Formation that was regarded as the Lower Coal Measures. Mamu Formation is overlain by the Ajali Formation which is regarded as the false-bedded sandstone [6] and followed by Nsukka Formation which is Fluvio-deltaic sediment [6] (Fig. 2).

Coal mining in Nigeria dates back to 1915 when sub-bituminous coals in the Mamu and Nsukka Formations (Maastrichtian, Danian) were first exploited. Exploration in the coal basins also led to the discovery of extensive lignite deposits in the Oligocene–Miocene Ogwashi-Asaba Formations [4]. Initial mining of the subbituminous coals was concentrated in the Enugu area while other mines in the Owukpa and Okaba areas focused on the Mamu Formation with the discovery of smaller deposits around Ezimo, Ogboyaga and Inyi in the middle of the last century. Total subbituminous coal reserves in the north-south trending coal belt were assessed to be about 1.5 billion tons [30]. Lignite deposits occur in numerous seams in an eastwest belt which passes through Ihioma and Azagba Ogwashi to the SW of the coal belt. Lignites reserves were estimated to total about 300 million tons but are largely unexploited [30]. Sub-bituminous coal production, mainly in the Enugu area, reached an annual output of Ca. 700,000 tons in the 1980s [30], but has since declined until recently when efforts to revive it

Fig. 2. Stratigraphic section of the Anambra Sedimentary Basin, modified after [25] Fig. Stratigraphic section the

have been proposed by the Nigerian Government. This has encouraged detailed studies of the geology, physical and chemical characteristics of the coals. have been proposed by the
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3. MATERIALS AND METHODS

Sedimentary rocks of the Mamu formation Sedimentary rocks of the Mamu formation
exposed at Emewe Efopa and its environs were mapped on a scale of 1: 25, 000. The method of investigation involves both field and laboratory work. Bedding characteristics in terms of textures, structures and lithology were studied in the field. Traverses were undertaken along stream channels, road cuts, footpaths and along untarred roads to reach the locations of outcrops. Fresh rock samples were collected at each location after removing the weathered surface using the geologic hammer. The dimensions of the outcrop were measured and global positioning systems (GPS) was used to take the coordinates (latitude and longitude) of the locations of coal and associated rock types in the study area. The samples at each location were described based on physical parameters such as colour, grain size, and sedimentary structures (bedding and other features). textures, structures and lithology wer
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surface using

3.1 Geophysical Studies

Vertical electrical sounding method of geophysical survey was carried out in the study area to determine the thickness and lateral Vertical electrical sounding method of
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extent of the coal and other rock types in the study area.

The vertical electrical sounding was carried out at Emewe Efopa. Instruments used include: Terameter, current electrodes (AB), potential electrode (MN), battery, code (meter). The vertical electrical sounding was carried
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The schlumberger configuration was adopted fo

The schlumberger configuration was adopted for the following reasons:

- **❖** The Schlumberger array provides for high signal to noise ratio.
- ❖ It has excellent vertical resolution and good depth sensitivity [31].
- * The manpower and time required for making Schlumberger sounding are less compared to that required for another array such as wenner profiling.
- \div It permits the acquisition of data within a very short time.
- Stray current in industrial areas and telluric currents that are measured with long spread affect Schlumberger array less than it affects wenner array. Stray current in industrial areas and telluric
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The effect of near-surface, lateral in-
- **The effect of near-surface, lateral in-**
homogeneities affect schlumberger homogeneities measurements less than it affects the wenner measurements.
- The interpretation techniques are more fully developed and more diversified for schlumberger sounding curves than wenner sounding curves.

3.1.1 Limitations

- Substantial lengths of cable energized with the current at high voltage present a safety hazard
- ❖ Long current electrode cables are required, the recording instruments need to be very sensitive, and the array may be difficult or confusing to coordinate amongst the field crew

During the field data acquisition the following precautions were observed;

- \clubsuit The connection was carefully made.
- Care was taken to ensure that the electrodes penetrated well into the ground to have good contact.
- Caution was taken while laying the wires for effective current flow.
- ❖ The terameter was off after each successive reading and on when taking next reading. This was done to prevent the readings from interfering.
- * Readings were not taken under sources such as high tension, metallic pipelines in

readings. order not increase or interfere with the

❖ Distance AB/2 being greater than or equal to five times the distance MN/2 was maintained throughout the survey.

The resistivity values of the spacing of the different electrodes were converted to apparent resistivity values using apparent resistivity formula for schlumberger array. The mathematical expression is below: to five times the distance MN/2 wa
maintained throughout the survey.
The resistivity values of the spacing of the
different electrodes were converted to apparer
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R\alpha \frac{L}{A} \tag{1}
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R = \frac{\rho L}{A} \tag{2}
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Where ρ = Proportionality constant called the electrical resistivity, $L =$ Unit length of material, A = Cross-sectional area.

Stray current in industrial areas and telluric order not increase or interfere
currents that are measured with long
the readings.
The effect Schlumberger array less
than it affects wenner array, the film of the times the The apparent resistivity values were plotted against half electrode spacing on logarithmic coordinate to obtain the sounding curves from which resistivities and thicknesses of the layers were determined. Apparent resistivity values and thicknesses obtained from both partial curve matching and the method of asymptotes were used for computer iterative modelling using interprets one- dimensional sounding inversion. This is done to identify the different geoeletric units. The geoeletric layers were further correlated with available lithologic logs from borehole within the area of study and based on the correlation; lithology was inferred for another different sounding. The interpretation was done using the curve types, the resistivity values of the geoelectric units, the thicknesses, in conjunction with the regional geology of the study area. Where ρ = Proportionality constant called the electrical resistivity, $L =$ Unit length of material, A = Cross-sectional area.
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Fig. 3. Illustration of Schlumberger array electrode spacing method

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Fig. 4. Topographic map of the study area

Fig. 5. Geologic map of the study area

Bulk fresh coal sample of approximately 100 mg was adequately collected from three locations in Bulk fresh coal sample of approximately 100 mg
was adequately collected from three locations in
the study area, transported to the laboratory for geochemical analysis. Various preparations and analysis carried out on the coal samples took place at Geological Laboratories in Kogi State University, Anyigba and Federal University of geochemical analysis. Various preparations and
analysis carried out on the coal samples took
place at Geological Laboratories in Kogi State
University, Anyigba and Federal University of
Technology, Akure. All samples were crushed in a rotary mortar and sieved through a 0.425 mm sieve size.

3.2 Chemical Analysis

Inorganic analysis frequently used is categorized into two major types namely: the proximate and the ultimate analysis. The proximate analysis is the most common analysis run on coal and consists of the following measure, moisture, ash, volatile matter, fixed carbon.

Ultimate analysis; ultimate analysis was also carried out on the samples to determine the composition of the coal in weight percent of carbon, hydrogen and oxygen (the major components) as well as sulphur, phosphorus, pyrite.

4. RESULTS AND DISCUSSION

4.1 Geophysical Investigation

The interpretation method used for the geophysical investigation represented above is an integration of 1D and 3D inversion methods using the IPI2win interpretation software. Both quantitative interpretation methods were applied

Bulk fresh coal sample of approximately 100 mg to determine the thicknesses and true
strategiven and the proximistic of the layers based on measured
depectentical analysis. Various preparations and "geoelectric units. The resistivities of the layers based on measured field values and subsequently help to identify geoelectric units. The sounding interpreted six geo-electric units (Table 2). The first laye about 0.25 m thick, which is the sandstone layer with a resistivity of 1799Ω m. The second layer of about 0.332 m thick, has a resistivity of 373Ω m and with a depth of 0.582 m interpreted as claystone lithology. The third layer has a thickness of about 0.715 m reaching a depth of 1.3 m has a resistivity of 2149 Ω m interpreted as shale. The fourth layer is about 2.85 m thick, depth of 4.15 m and has a high resistivity of about 31104Ω m interpreted as the layer that contains coal in the study area (Emewe- Efopa) due to the high resistivity of this layer which happens to be the layer that has the highest resistivity in the study area, it is interpreted as the coal layer. The fifth layer has a thickness of about 8.58 m, 12.7 m in depth with a resistivity of 106Ω m interpreted as clay stone which is the last layer whose base could not be reached. to determine the thicknesses and true
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4.2 Geochemical Analysis

4.2.1 Proximate analysis

The ash content in the samples varies from 24.5% in coal sample A has the minimum to 27% in coal sample B has the maximum (Table 3). Ash is the inorganic component of coal or the noncombustible residue left after coal is burnt to 700-750ºC. In other words, ash is the residual noncombustible matter in coal that comes from silt, clay, silica, or other substances. The ash due to the high resistivity of this layer which
happens to be the layer that has the highest
resistivity in the study area, it is interpreted as
the coal layer. The fifth layer has a thickness of
about 8.58 m, 12.7 m in

Fig. 7. Graphical interpretation of VES of the study area showing the different 7. Graphical layers (H (H-k type curve, using IPI2win software)IPI2win software)

content of the coal samples is inversely proportional to the moisture content. That is samples with high ash content has low moisture content as vice versa. This high ash content indicates that Emewe Efopa coal deposit is a low ranking type and will not burn well as an industrial fuel.

The moisture content in the coal samples ranges from 5.0% as the minimum in coal sample B to 8.0% as the maximum in coal sample A (Table 4). The moisture content is high in sample A simply because of the moisture absorbed by the coal from the possible underlain clay deposit. All

content of the coal samples is inversely the coal samples crumble and disintegrate on
proportional to the moisture content. That is exposure to the air due to their high moisture
content as vice versa. This high ash conten exposure to the air due to their high moisture content. The moisture content of coal also affect the calorific; that is, its combustibility. The higher the moisture content of coal the less it's calorific value and vice versa. The moisture content of coal also determines its rank. The lower the moisture content of coal, the higher the rank. For instance high-rank anthracite has little moisture value and vice versa. The moisture content of coal also determines its rank. The lower the moisture content of coal, the higher the rank. For instance high-rank anthracite has little moisture content of about 3-5% while lo bituminous coal has a higher moisture content of greater than 10%. The moisture content of 6.33% of Emewe Efopa coal deposit has low calorific value and sub-bituminous. the coal samples crumble and disintegrate on exposure to the air due to their high moisture content. The moisture content of coal also affect the calorific; that is, its combustibility. The higher the moisture content of c bituminous coal has a higher moisture content
greater than 10%. The moisture content
6.33% of Emewe Efopa coal deposit has l
calorific value and sub-bituminous.

Table 2. Model parameter for VES curve (A type)

Table 3. Ash percentage analysis results

Average Ash % = 24.5 + 27.0 + 25.0/3 = 25.5 %

Table 4. Moisture content analysis results

Average moisture % = 8 + 5 + 6/ 3 = 6.33%

Average volatile matter % 26+29+27/3= 27.33%

Volatile matter content in the samples ranges from 26% in the coal sample A as the minimum to 29% in the coal sample C as the maximum Table 5. Volatile matter in coal refers to the components of coal, except for moisture, which is liberated at high temperature in the absence of air. Volatile matter includes light hydrocarbon compounds such as carbon dioxide $(CO₂)$, methane (CH_4) , that were yielded by the decomposition of each layer of dead plant material by aerobic or oxygen- requiring bacterial during coalification (coal forming process). The importance of volatile matter in coal are: It's proportionality increases flame length and help in easier ignition of coal, its set minimum limit on the furnace height and volume, its influences secondary air requirement and distribution aspect, it also influences secondary air support.

The fixed carbon content in the samples varies from 39.0% in the coal sample B as the minimum to a maximum of about 42.0% in sample C (Table 6). The fixed carbon content of a coal is the carbon found in the material which is left after volatile matter are driven off.

4.2.2 Ultimate analysis

Organic carbon content in the coal samples ranges from 63.00 in sample B has the minimum to 64.50 in sample C has the maximum (Table 7). The moisture content ranges from 5% to 8% and on the other hand, the volatile matter ranges from 26% to 29% (Tables 4 & 5). Comparing these values with those of bituminous coal that has 4.31% moisture, 31.26% volatile matter and 83.47% carbon and anthracite that has 3.15% moisture, 21.63% volatile matter and 91.44%

carbon. Emewe-Efopa is therefore be assigned a low ranked sub-bituminous coal.

The sulphur content in the samples varies from 0.81% in sample B to 0.99% in sample B while the phosphorus content ranges from 0.04% in sample B to 0.05% in sample C (Table 7). The sulphur and phosphorus contents in the coal samples are directly proportional to the organic carbon content (Table 7). That is, samples with low sulphur and low phosphorus have low carbon while those with high sulphur and high phosphorus have high carbon. Though, the sulphur content of 0.91% on the average in Emewe Efopa coal falls within the ratings of <1.0% given by Ward [32] for coke making, its pyritic nature (3.30%-7.20%) makes it unsuitable for coke production. Like sulphur, phosphorus also constitutes an impurity in coal as its presence is not required in coke-making. The phosphorus content of 0.04% on average in Emewe Efopa coal is above the safe limit of 0.03% given by Bustin, et al. Gray, et al. [33] and therefore rendered the coal unsuitable for coke production.

The nitrogen content in the samples varies from 0.63 in sample A to 0.82 in sample C, while the hydrogen content ranges from 3.01 in sample B to 3.62 in sample in sample A (Table 6). Comparing the nitrogen and hydrogen contents of Emewe Efopa coal with bituminous coal and anthracite, shows that Emewe Efopa coal is ranked sub-bituminous coal.

However, these characteristics contrast with Newcastle (England) bituminous coal that contains 4.13% moisture, 0.20% ash, 31.26% volatile matter and 64.23% fixed carbon and South Wales (Britain) anthracite which according to Gray, et al. [34] contains 3.51% moisture, 1.32% ash, 21.63% volatile matter and 73.90% fixed carbon. These values have indicated that Emewe Efopa coal is neither a bituminous coal nor anthracite but sub-bituminous coal.

Coal moisture, volatile matter, and ash content are rank dependent parameters and they determine coal coking qualities [35]. Okaba coals are poor in those qualities and are therefore not suitable for coke making, which is also comparable with Emewe Efopa Coals. Jauro, et al. [36] also stated that the volatile matter apart from its uses in coal ranking is one of the most important parameters used in determining their suitable applications. Metallurgical coals according to Peng [37] are

commonly divided into three categories according to their content of volatile matter: lowmedium- or high-volatile content. Lower volatile coals (31-33%) are strongly expanding and create strong pressure during coking. If used alone, many low-volatile coals make a strong coke, due to high fixed carbon and other coking characteristics, but could break the wall of a coke oven if used alone. Emewe Efopa can be said to be an example of lower volatile coal since its content of volatile matter on average is 27.33%. On the other hand Okaba coals are good examples of high volatile coals, (>36%) and tend to have the opposite tendencies. They contrast, exert less wall pressure and make a weak coke if used alone. Mid-volatile coals (33- 36%), as might be expected, have properties between these two extremes. Low volatile coals expand to the point of sticking in the oven or breaking the oven walls. In the rating of coking coals for bedding by Bustin, et al. [33], volatile matter content of between 31.0-33.0% is graded as good, 33.0-36.0% as medium, and greater than 36.0% as poor. Volatile matter yield of coal, when determined, is used to: establish the rank of coals, indicate coke yield on carbonization, provide the basis for purchasing and selling, and establish burning characteristics.

Peng [37] also recommended an ash content of 6-8% and moisture content of 6% for good coking coal thereby making Emewe Efopa coal with moisture content of 6.33% and ash content of 25.5% on average fairly suitable for coke production due to its good moisture content.

Ultimate analysis (Table 9) shows that Emewe Efopa coal, on the average, contains 63.92% carbon, 3.25% hydrogen, 0.91% sulphur, 0.04% phosphorus, 0.72% nitrogen. These values are also similar to those of Wyoming (USA) subbituminous coal and Okaba (Nigeria) subbituminous coal (Table 7), indicating that Emewe Efopa coal is sub-bituminous coal. Blackmore [38] stated that Dietz (USA) coal that is rank
sub-bituminous contains 73.66% carbon, sub-bituminous contains 73.66% carbon, 5.19% hydrogen, 0.98% nitrogen. This composition is also similar to Emewe Efopa coal. Fatoye, et al. [15] Likewise stated that Okaba (Nigeria) coal that is ranked sub-bituminous contains 76.36% carbon, 5.95% hydrogen, 0.81% nitrogen, 0.71% sulphur, 0.05% phosphorus. This composition is also similar to Emewe Efopa coal in the same sub-bituminous rank. Sulphur is commonly present in most Nigerian coal in the form of pyrite and marcasite [30]. Sulphur in the form of pyrite has a serious

effect on the quality of iron but the sulphur (pyrite) can, however, be removed by an additional appropriate amount of limestone, desulphurization of the pig iron and washing [14]. The higher the sulphur content the more limestone is required for washing. The effectiveness of washing coal depends on the sophistication of the preparation plant and the nature of the sulphur. With conventional cleaning, most of the pyrite sulphur can be removed, but the organic sulphur is bound to the coals and is not removed [37]. Zimmerman [39] gave 0.05-0.06% as a safe limit while Gray et al, (1978) gave an even limit of 0.03%. Though the sulphur content of Emewe Efopa coal (0.91%) falls within the coke ratings for coke making, its pyrite nature makes its unsuitable for coke production.

Table 6. Fixed carbon results (Source; this study)

Table 7. Analysis results of hydrogen, sulphur, organic carbon, phosphorus, nitrogen (Source; this study)

Table 8. Summary table of proximate analysis result (Source: this study)

O: Okaba (Nigeria) Sub- bituminous coal [15]; X: Saar (Germany) sub-bituminous coal [34]; Y: Newcastle (England) bituminous coal [34]; Z: South Wales (Britain) anthracite [34]; A-C: This Study

O: Okaba (Nigeria) sub-bituminous coal [15]; X: Wyoming (USA) sub-bituminous coal [34]; Y: Newcastle (England) bituminous coal [34]; Z: South Wales (Britain) anthracite [34]; A-C: This study

5. CONCLUSION

Sediments typical of Mamu Formation exposed at Emewe Efopa and its environs in the Northern Anambra basin are composed of sandstone, mudstone, claystone and coal. The coal exposure/outcrop was subjected to geophysical and geochemical investigation. Vertical electric sounding method of geophysical exploration was able to help delineate the depth and lateral extent of the coal deposit and associated rocks in terms of layers. Geochemical analysis of the coal was used to rank the coal deposit as subbituminous. This study has helped to show extensively the importance of integrating field
petrographic studies, geophysical and studies, geophysical and geochemical methods of exploration of coal deposits in the study area.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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