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# Development and Testing of Sorption Isotherm Equipment for Nigerian Coal

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# Authors' contributions

This work was carried out in collaboration between all authors. Author OTE designed the study, all authors performed the statistical analysis and wrote the first draft of the manuscript and managed literature searches. Authors OTE, AD, OEE and EEB managed the Ultimate and proximate analyses of the study and literature searches. All authors read and approved the final manuscript.

### Article Information

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# ABSTRACT

Aim: The aim of the study is to investigate the methane content in Nigerian coal.

Study Design: Fabrication of Sorption Isotherm Equipment and Experimental Analysis.

**Place and Duration of Study:** Department of Petroleum and Gas Engineering, University of Port Harcourt Rivers State, between June, 2014 and January, 2015.

**Methodology:** This study includes the Fabrication of Isotherm Adsorption Equipment, Methane Sorption test, Proximate Analysis, Ultimate Analysis, and Estimation of Methane Content.

**Results:** The maximum moisture and fixed carbon was found in Sample D with moisture content of 3.35%, fixed carbon content of 60.41%; and it also has the minimum ash content of 5.61%. The maximum volatile matter was found in Sample B with volatile matter of 33.57%. The estimated methane content from the Meissner's method differs from those obtained from the adsorption Isotherm equipment by 22%.

**Conclusion:** The adsorption isotherm models obtained, showed that the total organic carbon content is not a significant factor in the adsorptive and dispersive behaviors exhibited by the shale

samples. It was also observed that pressure plays a major role in the adsorption process of methane gas from the coal samples.

Keywords: Coal bed methane; sorption isotherm; isotherm adsorption equipment; proximate and ultimate analysis.

## ABBREVIATIONS

- CBM Coal Bed Methane
- GHG Green House Gas
- FV Reference Gas Chamber
- SC Sample Chamber / Cell
- PT Pressure Transducer

#### **1. INTRODUCTION**

Nigeria as a developing country in Sub-Saharan Africa is gifted with numerous energy resources. Nigeria's energy resources are crude oil, natural gas, coal, tar sand, biomass and other renewable energy resources like geothermal, solar energy, tidal and wind energy, as well as large hydroelectric energy potential. These resources provide opportunities for the nation to thrive on a rich energy mix. The energy sector however, has not exploited this gift of nature. The progress in the use and development of oil and gas in the past fifty years has created an impression that hydrocarbon is the only viable energy medium available to the nation. This has negatively impacted on the comprehensive assessment and use of all Nigeria's energy resources that are available in the country. In order to ensure that Nigerians can enjoy a reliable and robust energy supply at a reasonable cost, it is essential that supply be generated from a variety of sources.

Coal is an alternative energy medium, which could be used with oil and gas to give the nation the desired energy mix that will ensure a reliable and affordable energy. Nigeria is the only country in West Africa producing coal at present and the producing coalfields are mostly located in the eastern and middle belt parts of Nigeria. Nigeria estimates its coal reserves at more than 2 billion tonnes, with approximately 650 million tonnes as proven reserves [1]. Although coal was the first energy resource to be exploited by Nigeria, a transition to oil and gas based fuels for power and transportation led to a decrease in coal production.

Methane (natural gas), while perhaps most closely related in our minds with petroleum, also

occurs in association with coal. Coalbed methane can be found almost anywhere there is coal. Considered a dangerous nuisance in the mining industry, it has potential as an abundant clean energy supply to help replace other diminishing hydrocarbon reserves. Recent developments in technologies and methodologies are playing a large part in harnessing this unconventional resource [2].

Because of its large ratio of surface area to volume, coal can store six to seven times as much gas as the same volume of rock in a natural gas reservoir. The gas content of a coal bed usually increases with depth and reservoir pressure. The deeper the coal, the less water is contained in the fractures, but water salinity increases with depth. Since water pressure prevents the gas from desorbing, the water must be removed to reduce the partial pressure in the coal seam so the gas can be released [3]. After desorption, the gas spreads throughout the coal bed. Significant coalbed methane production usually occurs only after substantial dewatering [4].

CBM can help fulfill a lot in the country, by giving a clean-burning fuel, increasing substantially, the natural gas reserve base; improving safety of coal mining, decreasing methane vented to the atmosphere from coal mines that might affect global warming and also improve access to abundant coal resource that is often too deep to mine.

Adsorption at various interfaces has concerned scientists since the beginning of this century. This phenomenon underlies a number of extremely important processes of utilitarian significance [5]. Its practical applications in industry and environmental protection are of paramount importance [6]. The adsorption of substrates is the first stage in many catalytic processes. Adsorption has application in water purification and recycling technologies. Among several methods, it is fast, inexpensive and a universal method [7].

## **1.1 Present Challenges**

- 1. Nigeria has about two billion tons of untapped coal waiting to be developed.
- Lack of awareness CBM deposits because currently emphasis has been on conventional gas resources, thereby neglecting CBM reserves.
- 3. With alternative energy discoveries in different parts of the world, Nigeria must harness all her energy potentials. Hence the need for quantitative and qualitative data for CBM recovery.
- Nigeria proven reserves of Natural gas is 186 trillion cubic meters (tcf), which is about 3% of the world total reserves. Hence there is need to increase our reserves.
- The recent IPP power projects and other gas to liquid (GTL) processes embark on in Nigeria have increase gas demand.

# 1.2 Literature Review

Coal seam can be classified in two major categories. The first is coalbed methane recovered in advance of mining operations, and the second involves primary CBM production in areas too deep to mine. In the first mode, the coal and methane resource are recovered in addition to potential GHG credits, making gas extraction economics less critical for project success. The second type of play must succeed without mining incentives or GHG credits. Like conventional natural gas wells, coal seam wells are drilled and stimulated, but coal's unique characteristics physical call for special techniques [4].

Scientific understanding of, and production experience with, coal-bed methane are both in the early learning stages. Much is yet to be learn about the controls on the occurrence and recoverability of coal-bed methane; the geologic, geochemical, engineering, technological, and economic factors, for example and about the environmental implications of developing the resource. The coal-bed methane industry is still relatively young, and few studies exist of the development and evolution of an individual coalbed methane play (a group of strata characterized by similar aspects of methane occurrence); thus, few models are available for planning the development of coal-bed methane resources on a broader scale. During coalification, large quantities of methane-rich gas are generated and stored within the coal on

internal surfaces. Because coal has such a large internal surface area, it can store surprisingly large volumes of methane-rich gas [8]. Evaluating the potential for CBM production relies heavily on laboratory core analysis and reservoir characterization. Field-level evaluation has evolved considerably since the early days of CBM development when models were adaptations of mining-industry techniques. Today, factors required to economically produce natural gas from coal seams are better understood. As new basins are explored, this understanding continues to evolve [2].

Nigerian coal ranges from lignite to bituminous in rank. The Anambra basin is located in the south east of Nigeria. This is the deposit that has economic viability and its area covered is very large. It covers about 1.5 million hectares and it is bounded on the west by river Niger, in the north by river Benue and on the east by Enugu Escarpment. The coal can be found in a single seam and the outcrops are along the east of the basin by the base of the Enuqu escarpment and drops into the middle of the basin. There is no evidence of the availability of coal at the west of the basin by river Benue. Outcrops of coal have been seen at Idah and Dekina which are in the northwest of the basin proving the existence of coal on the west and east of the basin [1].

### 1.3 Desorption

Desorption is the process by which methane molecules detach from the micropore surfaces of the coal matrix and enter the cleat system where they exist as free gas [9]. The desorption isotherm defines the relationship between the adsorbed gas concentration in the coal matrix and the free gas pressure in the cleat system.

The desorption isotherm is the link between the flow in the matrix system, where flow is controlled by concentration gradients, and flow in the cleat system, where flow is controlled by pressure gradients.

Gas content determination techniques generally fall into two categories:

- (1). Direct methods which actually measure the volume of gas released from a coal sample sealed into a desorption canister and
- (2). Indirect methods based on empirical correlations, or laboratory derived sorption isotherm gas storage capacity data.

# **1.4 GAS SORPTION ON COAL**

One of the reasons that coalbed methane resources are different from conventional natural gas resources is that sorption of gas is involved when producing coalbed methane. Sorption is the process, by which a gas is held on a microporous substance in a condensed or liquidlike phase.

Sorption processes are subdivided, according to the energies of interaction involved, into chemical (chemisorption) and physical sorption (physisorption).

In chemisorption, the sorbate is bound to specific sorption sites on the solid surface by electron transfer or electron sharing which makes this process highly specific compared to physisorption which is non-specific. Chemisorption typically involves monolayer while physisorption can involve either monolayer or multilayer surface coverage.

Physisorption occurs by van der Waals forces, which is attraction due to permanent and/or induced dipoles between the sorbate molecules and the atoms composing the sorbent surface. The enthalpy of physisorption is not much larger than the enthalpy of condensation of the sorbate and considered to be 1.0 to 1.5 times the latent heat of evaporation (compared to chemisorption where the enthalpy is >1.5 times the heat of evaporation) [10].

### 1.5 Methods to Determine Gas Sorption

For the determination of high pressure gas sorption isotherms of coals two different techniques are commonly used. The techniques differ in terms of the physical parameters used to determine the isotherms:

- (a). The manometric/volumetric method requires very accurate determination of cell and void volumes. Here the amount of gas sorbed is recorded by pressure readings (manometric method) or pressure and volume readings (volumetric method)
- (b). In the gravimetric method, the amount of gas sorbed is measured at constant pressure by means of a very accurate balance, with the sample either suspended mechanically or by magnetic coupling across the wall of a high pressure vessel.

### **1.6 Sorption Isotherm**

Sorption isotherm is used to express the equilibrium between a free gas and a sorbed phase which gives the sorbed gas content as a function of the free gas pressure at a constant temperature. The gas content obtained from the analysis of isotherm always gives the sorption capacity or the highest volume of gas a coal can hold. The sorption capacity is a reliable measure of the actual gas content only when coal is fully saturated with gas or when it is holding as much gas as it possibly can. The isotherm serves different types of purposes, it can provide the measure of sorption capacity, the isotherm is useful in evaluating the maximum possible gasin-place; and it gives an estimate of the actual gas-in-place, if the coal is fully saturated. It can also be used as a comparison by which gas content from direct method is compared in order to evaluate whether the coal is saturated with gas or not. Since the gas-in-place is given as a function of pressure, the isotherm can also provide an estimate of how much of the gas can be recovered. Because it represents the sorbed fluid property, the isotherm is an important input to reservoir flow simulator that are used to estimate gas rates, reserves and composition [11].

#### 2. MATERIALS AND METHODS

The different procedures used in the experiments on the coal samples are analyzed and documented.

# 2.1 Fabrication of Isotherm Adsorption Equipment

Isotherm adsorption Equipment is a high pressure, high volume adsorption system that is designed for the evaluation of gas adsorption isotherm for coal sample.

There is no fully developed coal laboratory in Nigeria where coal analysis such as sorption isotherm can be carried out. This necessitated the need for local fabrication of Isotherm Adsorption Equipment which can be used to carry out the measurement on coals from Nigerian coal field.

# 2.2 Proximate Analysis Procedure

To carry out the proximate analysis of coal samples, the following equipment are required:

- Oven
- Muffled Furnace
- Desiccator
- Weighing Balance
- Crucibles
- Tongs
- Coal samples

#### 2.2.1 Coal samples and preparation

Coal samples from different fields were received from Nigeria coal fields (See Table 1).

#### Table 1. Coal samples and location

| Coal sample | Location |
|-------------|----------|
| Sample A    | Okpara   |
| Sample B    | Okaba    |
| Sample C    | Ezimo    |
| Sample B    | Benue    |

The coal samples were crushed and grounded into fine power before it was used for proximate analysis.

# 2.2.2 Experimental determination of moisture content

The sample was dried with sun to remove the extraneous moisture. Approximately 1 g of the sample was weighed in a crucible and placed in an electric hot oven with temperature of 110°C. The crucible and the sample were allowed to stay for 1 hrs 30 mins before it was removed and placed in the desiccator to cool for some minutes. The cooled crucible and the sample were weighed again. The changes in weight were reported as the moisture on a percentage basis. It was calculated using;

% Moisture (M) = 
$$\frac{Y-Z}{Y-X} \times 100$$
 (1)

Where,

- X= weight of empty crucible, in grams (g)
- Y= weight of crucible + coal sample before heating, in grams (g)
- Z= weight of crucible + coal sample after heating, in grams (g)

Y -X = weight of coal sample, in grams

Y-Z = weight of moisture, in grams (g)

# 2.2.3 Experimental determination of ash content

The weight of the empty crucible was taken. Approximately 2 g of the coal sample was weighed with the crucible and placed into a muffle furnace at 450°C for 30 minutes. The temperature of the furnace was raised to 700°C and the sample was left for 1 hr. Then, the crucible was taken out and placed in the desiccator to cool before weighing. The percentage Ash content was calculated by;

% Ash = 
$$\frac{Z-X}{Y-X} \times 100$$
 (2)

Where

X= weight of empty crucible in grams (g)

- Y= weight of coal sample + crucible in grams (g) before heating
- Z= weight of coal sample + crucible in grams (g) after heating

# 2.2.4 Experimental determination of volatile matter

To determine the volatile matter of the samples, a covered crucible was utilized. The empty crucible and the cover were weighed. Then, the crucible was conditioned by heating in the muffle furnace at 800°C for 1 hour. The crucible was cooled and its weight was taken again. Approximately 2 g of the coal was weighed with the covered crucible and placed inside the muffle furnace at 925°C for 7 minutes, after which the crucible was removed and placed in the desiccator before re-weighing. Thus,

% Volatile matter (VM) = 
$$\frac{Y-Z}{Y-X} \times 100 - M$$
 (3)

Where

X = weight of empty crucible, in grams (g)

- Y = weight of crucible + coal sample before heating, in grams (g)
- Z = weight of crucible + coal sample after heating, in grams (g)

Y - X = weight of coal sample, in grams (g)

Y- Z = weight of volatile matter + moisture, in grams (g)

#### 2.2.5 Determination of fixed carbon

It was obtained by subtracting the sum of the moisture, ash and volatile matter and is given as:

#### Fixed Carbon (FC) = 100 - (M + VM + A) (4)

Where

- M = Moisture
- VM = Volatile Matter
- A = Ash content of coal

Elizabeth et al.; BJAST, 10(2): 1-14, 2015; Article no.BJAST.18445

# 2.2.6 Determination of Vitrinite reflectance of the coal

This was determining using the formula by Rice [12].

The formula is as follows:

$$R_0 = -2.712 \times \log(VM) + 5.092$$
 (5)

### 2.3 Ultimate Analysis Procedure

This shows the various elemental chemical components such as carbon, hydrogen present in the coal.

The relationships below, that exist between proximate and ultimate analysis was adopted.

## 2.4 Estimation of Methane Content

The methane was estimated based on [9] who observed that the amount of methane gas ( $V_{CH4}$ ) is related to volatile matter, calculated on dry ash free basis, by the following equation:

$$V_{CH_4} = -325.6 \times \log \frac{V_M}{37.8} \tag{6}$$

Most of the gas in the coal is adsorbed on the internal surface of micropores and varies directly with pressure and inversely with temperature. The relationship between the volume of adsorbed gas with pressure and temperature based on the moisture and ash content of coal samples was estimated by Kim's empirical equation. This was also employed in our methane content calculation.

$$V = \left[ \left( \frac{100 - M - A}{100} \right) \right] \times \frac{V_w}{V_d} \left[ K(P)^N - b \times T \right] (7)$$

Where

V = Volume of methane gas adsorbed (cc/g)

M = Moisture content (%)

A = Ash content (%)

$$V_w/V_d = \frac{1}{(0.25 \times M \times 1)}$$
 (8)

Where

 $V_w$  = Volume of gas adsorbed on wet coal (cc/g)  $V_d$  = Volume of gas adsorbed on dry coal (cc/g)

$$K = 0.8 \left(\frac{FC}{V_m}\right) + 5. \tag{9}$$

Where,

FC = Fixed carbon (%)

VM = Volatile matter (%)

- N = Constant, depends on the composition of coal (for most bituminous coals, N = 0.39 0.013 × K)
- b =Adsorption constant due to temperature change (cc/g/°C).

$$T = 2.5 \times \frac{h}{100} + T_o \tag{10}$$

Where,

 $T_0$  =ground temperature

h = depth(m)

## 2.5 Experimental Procedure for Sorption Isotherm

The Methane sorption experiment was carried out in a self-fabricated set up which was shown above in Figs. 1 and 2. The sorption tests were carried out up to maximum pressure of 800psi.

### 2.5.1 Procedure for gas sorption

The process flow diagram of fabricated set up represents reference gas chamber (FV), sample chamber (SC); pressure transducer (PT). The pressure gauge helps to measure the pressure in

| Table 2 Relationship  | between ultimate | analysis and | proximate | analysis |
|-----------------------|------------------|--------------|-----------|----------|
| Table 2. Relationship | between unmate   | analysis and | proximate | anaiyəiə |

| %C    | = | 0.97C+ 0.7(VM - 0.1A) - M(0.6-0.01M)                        |
|-------|---|---|
| %H    | = | 0.036C + 0.086 (VM -0.1xA) - 0.0035M <sup>2</sup> (1-0.02M) |
| %N2   | = | 2.10 -0.020 VM  |
| where |   |   |
| С     | = | % of fixed carbon   |
| A     | = | % of ash  |
| VM    | = | % of volatile matter  |
| Μ     | = | % of moisture   |

Elizabeth et al.; BJAST, 10(2): 1-14, 2015; Article no.BJAST.18445



Fig. 1. Fabricated reference and sample cell





Fig. 2. Fabricated sorption isotherm equipment for Nigerian coal

the sample cell and the reference cell. A micro filter is contained in the sample cell not visible in Fig. 1. The sample was placed inside the sample chamber to prevent loss of samples during the application of vacuum. The entire fabricated set up was submerged in a constant temperature water bath. The amount of adsorbed volume of gas was estimated at a constant temperature which was a function of gas pressure at equilibrium. The equilibrium at each isotherm point was achieved at a constant pressure within 6-8 hours. Real gas law was applied to measure the adsorbed volume. To calculate the compressibility factor in real gas equation, Peng-Robinson Equation of State was utilized. The fixed volume and the void volume were measured using Helium. After conducting the experiment at constant temperature, pressurized gas was totally depressurized. The whole setup was maintained at constant temperature and the process was repeated for different coal samples.

# 2.5.2 Evaluation of the volume of the equipment

It is necessary to estimate the void volume in the sample cell very accurately so as to determine the volume of gas adsorbed with limited error. A minor error in the void volume measurement could lead to significant error in the sorbed gas estimation. In the fabricated setup, the void volume consisted of the adsorbent pore volume, inter-particle voids and the free volume that included up to the valve. Helium gas was used to evaluate the void volume by expanding it from FV to SC and the equilibrium pressure measured. Helium has the property of not getting adsorbed on coal, hence it was used. To calculate all the volume, ideal gas equation was used. Before charging gas into the unit, it is necessary to evacuate the gas within the sample and the reference cell using a vacuum pump. The equation used to evaluate the void volume is given as:

$$V_V = \frac{\Delta P V_{FVC}}{\Delta P_{SC}} \tag{11}$$

Where

 $\Delta p$  denotes the change in pressure due to gas expansion after the valve between the sample cell and the reference cell was opened. Each step was repeated thrice so as to minimize any experimental error.

### 2.5.3 Measurement of pure gas

The following procedure was adopted to measure the gas adsorbed in the unit.

Accurately weighed samples was first placed inside the sample cell and sealed tightly. The sample cell was isolated from the reference cell by closing the valve in between. Methane gas was fed into the reference cell by 150 units, and the valve connecting to FV closed. The gas was allowed to attain its equilibrium state and the pressure reading recorded as  $P_1$ . The valve between the sample and reference cell was then opened and the gas from the reference cell flows into the sample cell and gets adsorbed in the sample. When equilibrium state was attained, the pressure reading for both the sample cell ( $P_{s2}$ ) and reference cell ( $P_{r2}$ ) was recorded.

To calculate the moles of gas that was adsorbed by coal in the sample cell, it was necessary to calculate the number of moles of gas that will leave FV, and the number of moles of gas that will appear in the SC as free gas in the void volume. Again a mass balance analysis was carried out.

To obtain isotherm at next higher pressure, the valve between the SC and FV was closed. Now the valve connecting the inlet gas and the FV was opened to inject the gas to next 150 units. The valve was closed and the equilibrium reading in the FV recorded. Same procedure was repeated for the next increment in pressure until isotherm was reached.

To obtain the desorption isotherm, the gas in the FV was first released by 150 unit and the pressure recorded after equilibrium was attained. The valve between the sample cell and the reference cell was opened and the gas allowed to flow from the sample cell (SC) to the reference cell and the pressure reading is noted until equilibrium was achieved. Same procedure was repeated till the minimum pressure was present in the sample cell and the desorption isotherm plotted.

#### 2.5.4 Methane sorption calculations

Gas adsorption data were obtained using a volumetric apparatus. The volumetric apparatus was immersed in the constant temperature water bath. The adsorbed volume was determined at a constant temperature (30°C, 40°C & 50°C) as a function of pressure at equilibrium. Concept of volumetric adsorption is based on the phenomena that adsorption removes the adsorbate gas molecules from the free gas phase, resulting in decrease in free gas pressure within the experimental system [13]. The difference in the total amount of gas  $(n_T)$ introduced into the void volume of sample cell (SC) and the amount of free gas occupying the void volume  $(n_v)$  in the sample cell gives the amount of gas sorbed  $(n_a)$  in number of moles.

$$\boldsymbol{n}_a = \boldsymbol{n}_T + \boldsymbol{n}_v \tag{12}$$

The total amount of gas introduced into the SC during a particular pressure step is estimated using the equation below:

$$\boldsymbol{n}_T = \left[\frac{\boldsymbol{V}}{\boldsymbol{R}T} \times \left(\frac{\boldsymbol{P}_{s1}}{\boldsymbol{Z}_1} - \frac{\boldsymbol{P}_{s2}}{\boldsymbol{Z}_2}\right)\right] \tag{13}$$

# 3. RESULTS AND DISCUSSION

The results obtained from experiments and calculations are discussed and presented in tables and graphs.

#### 3.1 Proximate Analysis Result

Using equation (1);

Sample A  
= 
$$\frac{32.8924 - 32.8805}{32.8924 - 31.8727} * 100 = \frac{0.0119}{1.0199} * 100$$
  
= 1 17%

After heating of the sample and the crucible in the oven for 1 hour 30 minutes at  $110^{\circ}$ C, there were no visible changes in the color of the sample but after calculation, it was found that sample A has the lowest moisture content of 1.17%, followed by sample C with moisture content of 2.49%, then sample B with moisture content of 3.22%. The highest moisture was found in D with moisture content 3.35% (See Table 3).

### 3.2 Result and Calculation of Ash Content

After heating of the sample and the crucible in the muffle furnace first at  $450^{\circ}$ C for 30 minutes, then at  $800^{\circ}$ C for 1 hour there were visible

changes in the color of the samples see Fig. 3. From equation (2);

Sample A  
= 
$$\frac{37.1808 - 35.9303}{37.9349 - 35.9303} * 100 = \frac{1.2505}{2.0046} * 100$$
  
= 62.38%

The results obtained from calculation shows that sample A has the highest ash content of 62.38%, followed by sample C with ash content of 10.22%, then sample B with ash content of 9.1%. The lowest ash was found in D with ash content 5.61%.

### 3.3 Result and Calculation on Volatile Matter

Applying equation (3);

Sample A

$$= \frac{67.7969 - 67.3982}{67.7969 - 65.7969} * 100 - 1.17\%$$
$$= \frac{0.3987}{2} * 100 - 1.17\%$$
$$= 18.76\%$$

After heating the sample with the covered crucible in the muffle furnace at 925°C for 7 minutes, little changes in the color of the samples was observed. It was found from calculations that sample A has the minimum volatile matter of 18.77%, followed by sample C with volatile matter of 30.25%, then sample D with volatile matter of 30.63%. The maximum volatile matter was found in sample B, 33.57% (see Table 5).

# 3.4 Result and Calculation on Fixed Carbon

This was calculated (equation 4) by subtracting the sum of Moisture, ash and volatile matter from 100 (in air dried basis) and is given as:

Sample A = 100 - (1.17 + 62.38 + 18.77) = 17.68%

It was found that sample A has the lowest fixed carbon of 17.68%, followed by sample B with fixed carbon of 54.13%, then sample C with fixed carbon of 57.04%. The highest fixed carbon was found in D with fixed carbon of 60.41%.

#### Table 3. Experimental weights from moisture content analysis

| Sample | Empty crucible (X)<br>(grams) | Empty crucible +Sample<br>before heating (Y) (grams) | Empty crucible+ Sample after<br>heating (Z) (grams) |
|--------|-------------------------------|--|---|
| A      | 31.8725                       | 32.8924  | 32.8805   |
| В      | 52.3250                       | 53.2186  | 53.1898   |
| С      | 42.1637                       | 43.1098  | 43.0862   |
| D      | 41.2210                       | 42.2128  | 42.1796   |

#### Table 4. Ash content results

| Sample | Empty crucible | Empty crucible +Sample     | Empty crucible+ Sample    | % Ash   |
|--------|----------------|----------------------------|---------------------------|---------|
|        | (X) (grams)    | before heating (Y) (grams) | after heating (Z) (grams) | content |
| A      | 35.9303        | 37.9349                    | 37.1808                   | 62.38   |
| В      | 37.8788        | 39.8771                    | 38.0608                   | 9.10    |
| С      | 36.7624        | 38.7613                    | 36.9667                   | 10.22   |
| D      | 31.0953        | 33.0996                    | 31,2077                   | 5.61    |





# 3.5 Results on Estimation of Methane Content

Estimating the volume of methane based on Meisner (1984) who observed that the amount of methane gas ( $V_{CH4}$ ) is related to volatile matter, calculated on dry ash free basis. Using equation (6);

Sample A

 $V_{CH_4} = -325.6 \times \log \frac{18.77}{37.8} = -325.6^* -0.304$ = 98.99 m3/tonnes The values obtained from the adsorption equipment are presented in the Table 6.

The estimated methane content from the Meissner's method differs from those determined from the adsorption Isotherm equipment. The estimated volume of methane generated using Meissner's equation varies from 16.78 cc/100g for sample B, followed by sample D with 29.74 cc/100g, then Sample C with 31.50cc/100g. The maximum value was observed in Sample A with 98.99 cc/100g. Whereas the maximum methane adsorbed using the equipment was for



Fig. 4. Methane gas adsorption isotherm at variable pressure for Sample A

#### Table 5. Volatile matter results

| Sample | Empty crucible + | Empty crucible + lid +    | Empty crucible + lid +   | % Volatile |
|--------|------------------|---------------------------|--------------------------|------------|
|        | lid (X) (grams)  | Sample before heating (Y) | Sample after heating (Z) | matter     |
|        |                  | (grams)                   | (grams)                  |            |
| A      | 65.7969          | 67.7969                   | 67.3982                  | 18.77      |
| В      | 54.6735          | 56.6728                   | 55.9377                  | 33.57      |
| С      | 55.2815          | 57.2834                   | 56.6279                  | 30.25      |
| D      | 54.557           | 56.5976                   | 55.9041                  | 30.63      |

| Table | 6. E | Data | of a | dsorp | tion | Isotherm | equipm | ent |
|-------|------|------|------|-------|------|----------|--------|-----|
|-------|------|------|------|-------|------|----------|--------|-----|

| Sample | Temperature<br>(°C) | Reference<br>cell<br>volume<br>(cc) | Sample<br>cell<br>volume<br>(cc) | Sample<br>weight<br>(g) | Sample<br>density<br>(g/cc) | Sample<br>volume<br>(cc) | Dead<br>volume<br>(cc) |
|--------|---------------------|-------------------------------------|----------------------------------|-------------------------|-----------------------------|--------------------------|------------------------|
| А      | 30                  | 635.25                              | 547.75                           | 99.89                   | 0.69                        | 144.77                   | 381.15                 |
| В      | 30                  | 635.25                              | 547.75                           | 100.03                  | 0.73                        | 137.03                   | 353.75                 |
| С      | 30                  | 635.25                              | 547.75                           | 100.59                  | 0.71                        | 141.68                   | 423.50                 |
| D      | 30                  | 635.25                              | 547.75                           | 99.92                   | 0.71                        | 140.73                   | 423.50                 |

Sample C with 50.86 cc/100g, followed by D with 34.15cc/100g, then sample A with 31.44 cc/100g. The minimum value was observed in sample B, 20.61 cc/100g. The volume of the adsorped methane also increases. This trend was

observed throughout the experimental study. Thus, pressure plays a major role in the adsorption process of methane gas from the coal samples.



## Fig. 5. Methane gas Adsorption Isotherm at variable pressure for Sample B

| Initial pressure<br>(Reference cell)(Psia) | Final pressure<br>(system)(Psia) | Reference cell<br>volume | Dead volume of<br>sample cell (cc) | Volume<br>adsorbed (cc) |
|--|----------------------------------|--------------------------|------------------------------------|-------------------------|
| 200  | 124                              | 635.25                   | 381.15                             | 8.20                    |
| 300  | 185                              | 635.25                   | 381.15                             | 13.74                   |
| 400  | 245                              | 635.25                   | 381.15                             | 20.74                   |
| 500  | 305                              | 635.25                   | 381.15                             | 24.99                   |
| 600  | 365                              | 635.25                   | 381.15                             | 27.85                   |
| 700  | 425                              | 635.25                   | 381.15                             | 29.89                   |
| 800  | 485                              | 635.25                   | 381.15                             | 31.44                   |

| Table 7. | Sample A | adsorption | Isotherm data | at variable | pressure |
|----------|----------|------------|---------------|-------------|----------|
|----------|----------|------------|---------------|-------------|----------|

| Гab | le 8 | 3. S | Samp | le | B ac | lsor | ption | lsot | herm | data | at | vari | ab | le | pres | su | re |
|-----|------|------|------|----|------|------|-------|------|------|------|----|------|----|----|------|----|----|
|-----|------|------|------|----|------|------|-------|------|------|------|----|------|----|----|------|----|----|

| Initial pressure<br>(Reference cell)(psia) | Final pressure<br>(system)(Psia) | Reference cell<br>volume | Dead Volume of<br>Sample cell (cc) | Volume Adsorbed<br>(cc) |
|--|----------------------------------|--------------------------|------------------------------------|-------------------------|
| 200  | 116                              | 635.25                   | 453.75                             | 6.26                    |
| 300  | 174                              | 635.25                   | 453.75                             | 6.26                    |
| 400  | 231                              | 635.25                   | 453.75                             | 11.00                   |
| 500  | 288                              | 635.25                   | 453.75                             | 13.86                   |
| 600  | 345                              | 635.25                   | 453.75                             | 15.78                   |
| 700  | 402                              | 635.25                   | 453.75                             | 17.16                   |
| 800  | 458                              | 635.25                   | 453.75                             | 20.61                   |

Elizabeth et al.; BJAST, 10(2): 1-14, 2015; Article no.BJAST.18445



Fig. 6. Methane gas Adsorption Isotherm at variable pressure for Sample C



| Fia. | 7. Methane c | as Adsorption | Isotherm at variable | pressure for Sample D |
|------|--------------|---------------|----------------------|-----------------------|
|      |              |               |                      | p                     |

| Initial pressure<br>(Reference cell)(psia) | Final pressure<br>(system)(Psia) | Reference cell<br>volume | Reference cell<br>volume | Volume<br>adsorbed (cc) |
|--|----------------------------------|--------------------------|--------------------------|-------------------------|
| 200  | 118                              | 635.25                   | 423.5                    | 17.94                   |
| 300  | 174                              | 635.25                   | 423.5                    | 36.51                   |
| 400  | 231                              | 635.25                   | 423.5                    | 41.25                   |
| 500  | 288                              | 635.25                   | 423.5                    | 44.11                   |
| 600  | 345                              | 635.25                   | 423.5                    | 46.03                   |
| 700  | 402                              | 635.25                   | 423.5                    | 47.41                   |
| 800  | 458                              | 635.25                   | 423.5                    | 50.86                   |

| rable 9. Sample | C adso | orption | Isotherm | data | at variable | pressure |
|-----------------|--------|---------|----------|------|-------------|----------|
|-----------------|--------|---------|----------|------|-------------|----------|

| Initial pressure<br>(Reference cell)(psia) | Final pressure<br>(system)(Psia) | Reference cell<br>volume | Reference cell<br>volume | Volume Adsorbed<br>(cc) |
|--|----------------------------------|--------------------------|--------------------------|-------------------------|
| 200  | 118                              | 635.25                   | 423.5                    | 17.94                   |
| 300  | 176                              | 635.25                   | 423.5                    | 24.06                   |
| 400  | 234                              | 635.25                   | 423.5                    | 27.15                   |
| 500  | 292                              | 635.25                   | 423.5                    | 29.01                   |
| 600  | 350                              | 635.25                   | 423.5                    | 30.25                   |
| 700  | 408                              | 635.25                   | 423.5                    | 31.14                   |
| 800  | 465                              | 635.25                   | 423.5                    | 34.15                   |

 Table 10. Sample D adsorption Isotherm data at variable pressure

### 4. CONCLUSION

Based on the experimental results and calculations obtained from this study, the following conclusions were made.

- 1. The sorption capacity of Nigerian coal is evaluated and the volume of methane that can be trapped and released for use was established.
- 2. High pressure Adsorption Isotherm equipment was fabricated with locally made materials for the purpose of this study.
- 3. Based on the proximate and the ultimate analysis, the fixed carbon is directly related to the carbon compound found in coal and the volatile matter is directly related to the amount of carbon compound available in the coal.
- 4. The amounts of methane generated in the coal samples are higher than the amount calculated and it shows that the Nigeria coal deposit can sustain CBM.
- 5. The differences in the methane content can be attributed to the pressure conditions and hydrological conditions.

### **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

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# **APPENDIX: CALCULATIONS**

Estimating the volume of methane based on Meisner (1984)

$$V_{CH_4} = -325.6 \times \log \frac{V_M}{37.8}$$

Sample A

$$V_{CH_4} = -325.6 \times \log \frac{18.77}{37.8} = 98.99 \text{ m}^3/\text{tonnes}$$

Sample B

$$V_{CH_4} = -325.6 \times \log \frac{33.57}{37.8}$$
 = 16.78 m<sup>3</sup>/tonnes

Sample C

$$V_{CH_4} = -325.6 \times \log \frac{30.25}{37.8}$$
 =31.50 m<sup>3</sup>/tonnes

Sample D

$$V_{CH_4} = -325.6 \times \log \frac{30.63}{37.8} = 29.74 \text{ m}^3/\text{tonnes}$$

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