Investigation into the Characterization of Bituminous Coal Particles and Surface Structure Study Using Brunauer-Emmett-Teller (BET) and Scanning Electron Microscope (SEM)

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Abstract—The aim of this study is to characterize the Bituminous coal particles and study the surface structure of the particles using BET and SEM. This study aided in suggestions to improve overall handling of coal. BET test result classified bituminous coal as mesoporous material that ranges from 2 to 49.999. The coal were ground dry with a milling machine into powder form and characterized by sieving into seven standard sieve sizes of 75µm, 150µm, 212µm, 300µm, 425µm, 600µm and 850µm with Octagon 2000 sieve shaker. The sieved coal powders were used for BET and SEM experiment respectively. The result of this study showed the adsorptive nature of the coal particles.

Keywords— Brunauer-Emmett-Teller Theory, SEM-Scanning Electron Microscope, nanometer, mesoporous

I. INTRODUCTION

This work will investigate into the characterization of particles and surface structure Study of Bituminous Coal using BET and SEM. It will determine the surface nature of coal particles. A sieve analysis (or gradation test) is a practice or procedure used (commonly used in civil engineering) to assess the particle size distribution (also called gradation) of a granular material. The size distribution is often of critical importance to the way the material performs. A sieve analysis can be performed on any type of non-organic or organic granular materials including sands, crushed rock, clays, granite, feldspars, coal, and soil, a wide range of manufactured powders, grain and seeds, down to a minimum size depending on the exact method. Being such a simple technique of particle sizing, it is probably the most common.

II. EXPERIMENTAL

A. Materials

Bituminous coal will be used in this project and was obtained from Nasarawa State, Nigeria.

B. Methods

a. Determination of Coal Sieve Sizes (Sieve Analysis)

The bituminous coal was ground dry with a milling machine into powder form and sieved with Octagon 2000 sieve shaker comprising size ranges of 75µm, 150, 212µm, 300µm, 425µm and 850µm meshes.
The seven calibrated standard mechanical sieve shaker was used for sieve analysis. The sizes of micrometers are 850µm, 600µm, 425µm, 300µm, 212µm, 150µm and 75µm. Each lower sieve in the column has smaller mesh/openings calibrated respectively than the one above. A representative weighed sample of powdered coal was poured into the top sieve which has the largest screen mesh/openings calibration. At the base is a round pan, called the particle receiver.

The column is typically placed in a mechanical shaker. The shaker shook the column, usually for some fixed amount of time required. At the end of the shaking process, the material particles sizes on each sieve was collected and separated into different micrometers, labeled and weighed, ready to be used for experiments.

The bituminous coal was ground dry with a milling machine into powder form and sieved with Octagon 2000 sieve shaker comprising size ranges of 75µm, 150, 212µm, 300µm, 425µm and 850µm meshes.

One of the precaution measures was to brush the meshes of the sieves to remove the already stacked particles in the meshes.

b. BET Test procedure:

Normally, for any sample to be subjected to BET test or machine it must be weighed within the range of 0.35 –0.40g on a weighing balance. The small tube/beaker that was used for the test was weighed and recorded. About 0.35 – 0.40g of the samples were weighed and put into the tube/beaker. The tube/beaker containing the material for BET test was weighed and recorded along with the both weight of the material/tube, also called weight before treatment. At this point, the materials are ready for heat treatment.

The tube containing the measured material was inserted into the heat treatment machine: Helium was used in the heat treatment for degassing. The process or action of degassing or removing unwanted material from a material is called physiosorption. Heat treatment removes the moisture in the material and as well dries up the moisture in the material. The heat treatment is set at about 400°C for materials that does not decompose on exposure to heat and 10 - 200°C for some materials that decompose on exposure to heat. To every 10°C rise in temperature, the rate of reaction will double (times two) of its original rate of reaction.

At the end of the heat treatment after a set period; say 1hr 30min or about 2hrs, the samples are cooled for about 5min – 10minutes, weighed and the actual dry weight of the samples were recorded. The materials were then fixed on the BET machine: first wear the glass tube a jacket down the bottom, insert a glass rod into the tube, unscrew the BET screw and screw the glass tube to the machine.

Liquid Nitrogen gas was used in the BET. Its temperature is -196°C. The liquid Nitrogen gas was then poured into the liquid Nitrogen dewar-flask and the flask was put back into the BET machine directly under the samples for surface area measurement.

The BET machine glasses are closed and the interfaced computer was configured to the desired/required test output: put the weights and samples name, go to unit 1, select sample analysis, click on brows and align the samples with the machine numbering system and fix any other missing information, then confirm and click ok/start. The BET machine was allowed to read the sample and generate results as set. When the BET machine has finished running the test, the apparatus used for the test set-up were disengaged; glass tube washed and put in the drying machine for other users.
There are two software’s used for BET operation. They are Microactive for result analysis and Tristar for data analysis or for sampling.

To operate Tristar; go to file, click open and go to sample information, click ok and yes on the question dialogue box (do you want to create it?) and run or set up.

### Table 1: BET Experimental data of bituminous Coal (from 06/08/2014)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weight of glass (g)</th>
<th>Weight of sample/glass before heat treatment (g)</th>
<th>Weight of sample/glass after heat treatment (g)</th>
<th>Actual weight of material (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1: (75µm bitu)</td>
<td>18.081</td>
<td>18.463</td>
<td>18.430</td>
<td>0.033</td>
</tr>
<tr>
<td>Sample 2: (150µm bitu)</td>
<td>18.080</td>
<td>18.424</td>
<td>18.391</td>
<td>0.033</td>
</tr>
<tr>
<td>Sample 3: (212µm bitu)</td>
<td>17.823</td>
<td>18.220</td>
<td>18.184</td>
<td>0.036</td>
</tr>
<tr>
<td>Sample 4: (300µm bitu)</td>
<td>17.824</td>
<td>18.169</td>
<td>18.134</td>
<td>0.035</td>
</tr>
<tr>
<td>Sample 5: (425µm bitu)</td>
<td>17.523</td>
<td>17.931</td>
<td>17.892</td>
<td>0.039</td>
</tr>
<tr>
<td>Sample 6: (600µm bitu)</td>
<td>17.824</td>
<td>18.216</td>
<td>18.177</td>
<td>0.039</td>
</tr>
<tr>
<td>Sample 7: (850µm bitu)</td>
<td>17.536</td>
<td>17.916</td>
<td>17.877</td>
<td>0.039</td>
</tr>
</tbody>
</table>

The BET summary result above shows the surface area of different particle size of sub-bituminous coal, the pore width and diameter which indicates the nanometre class of the material; micropores material, mesopores material or macropores material.

In the classification of nanometre materials; micropores is from 1 to 2.999nm, mesopores is from 2 to 49.999 and macropores is from 50 and above, therefore, from the result above, bituminous coal can be classified as mesoporous materials.

c. **Microstructure determination: SEM Test procedure**

Scanning Electron Microscope was used to study the micro structures of particles; 75µm, 150, 212µm, 300µm, 425µm and 850µm (of grain sizes) of the materials.

The particles sample of each grain size is placed on a surface of the electron aluminium stubs with the help of gummy carbon tabs. The stubs are placed on the gold sputter crater, put on for about 15 to 25 minutes as the case may be to vacuum small amount of moisture in the particles and coat the particles with a thin layer of gold to make the sample conducting and examined in a scanning electron microscope (Stereoscan) or first coat the particles with carbon before gold as the case may be.
After the 15 to 25 minutes coating, the machine trips off automatically, then the stubs are removed from the crater one at a time.

The stubs containing the particles/agglomerates are put into the SEM particle chamber and allowed for 3 minutes for electrons drop on the surface of the particles for good magnification. At this time, the image is shown on the display screen consul of the SEM machine and operated to show the image on the interfaced computer/PC.

![Scanning Electron Microscope SEM (Hitachi Model No: S-520)](image)

**Table 2:** Particle magnification Level of both sub-bituminous coal and bituminous

<table>
<thead>
<tr>
<th>Particle size (µm)</th>
<th>First magnification level (particle lump)</th>
<th>Second magnification level (particle lump surface)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1: (75µm)</td>
<td>X1000</td>
<td>X4000</td>
</tr>
<tr>
<td>Sample 2: (150µm)</td>
<td>X500</td>
<td>X3000</td>
</tr>
<tr>
<td>Sample 3: (212µm)</td>
<td>X500</td>
<td>X5000</td>
</tr>
<tr>
<td>Sample 4: (300µm)</td>
<td>X400</td>
<td>X3000</td>
</tr>
<tr>
<td>Sample 5: (425µm)</td>
<td>X150</td>
<td>X3000</td>
</tr>
<tr>
<td>Sample 6: (600µm)</td>
<td>X120</td>
<td>X4000</td>
</tr>
<tr>
<td>Sample 7: (850µm)</td>
<td>X100</td>
<td>X2000</td>
</tr>
</tbody>
</table>

**III. RESULTS AND DISCUSSIONS**

**A. Table 3: BET Summary Result of bituminous Coal**

<table>
<thead>
<tr>
<th>Particle size</th>
<th>75µm</th>
<th>150µm</th>
<th>212µm</th>
<th>300µm</th>
<th>425µm</th>
<th>600µm</th>
<th>850µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>BET Surface Area m²/g</td>
<td>3.8392</td>
<td>3.7641</td>
<td>3.6355</td>
<td>3.5839</td>
<td>3.4130</td>
<td>2.8275</td>
<td>2.5708</td>
</tr>
<tr>
<td>BJH Adsorption average pore width (4V/A): nm</td>
<td>5.7612</td>
<td>5.5674</td>
<td>5.3900</td>
<td>5.3465</td>
<td>5.2485</td>
<td>5.1608</td>
<td>5.6600</td>
</tr>
<tr>
<td>t-Plot micropore volume: cm³/g</td>
<td>0.000204</td>
<td>0.000112</td>
<td>0.000096</td>
<td>0.003885</td>
<td>0.003553</td>
<td>0.002871</td>
<td>0.002884</td>
</tr>
</tbody>
</table>
The surface area of particle sizes of bituminous coal, the pore width and diameter were achieved from BET test results. These results indicate that bituminous coal particles belong to the nanometre class of mesoporous materials amongst microporous material, mesoporous material and macroporous material. This is because, in the classification of nanometre materials; micropores range from 1 to 2.999nm, mesopores ranges from 2 to 49.999 and macropores range from 50 and above [1]. And form the result, the values fall within the ranges of 2 to 49.999.

B. Scanning Electron Microscope – SEM of Bituminous Coal

Fig. 2: X1000 of 75µm

Fig. 3: X4000 of 75µm
Fig. 4: X500 of 150µm

Fig. 5: X3000 of 150µm
Fig. 6: X500 of 212µm

Fig. 7: X5000 of 212µm
Fig. 8: X400 of 300µm

Fig. 9: X3000 of 300µm
Fig. 10: X150 of 425µm

Fig. 11: X3000 of 425µm
Fig. 12: X120 of 600µm

Fig. 13: X4000 of 600µm
**Fig. 14:** X100 of 850 µm

**Fig. 15:** X2000 of 850 µm
In these electron micrographs, I observed that the structural surfaces of particles show no micropores of any kind. There are rough observable whitish surfaces on SEM scanned structure of the micrographs.

The study and observation of the structural surfaces of particles in micrographs in chapter three shows no micropores of any kind. And this is in conformity with the BET results which indicated that the coal particles are of mesopores ranging from 2 to 49.999.

The build-up of electrons on the non-conductive area or surfaces of particles is as a result of negatively charged electron meeting with a non-positively charged particle surfaces and as such the electron tends to grow or glow white on that particular surface. There are some observable whitish surfaces on SEM scanned structure.

IV. CONCLUSION

The surface area result of particle sizes of bituminous coal from the BET test carried out indicates that bituminous coal particles belong to the nanometre class of mesoporous materials (ranges from 2 to 49.999).

The observation on the structural surfaces of particles in micrographs in chapter three shows no micropores of any kind and which is in agreement with the BET results which indicated the mesopores nature of the coal particles and ranging from 2 to 49.999.

REFERENCES

   http://particle.dk/methods-analytical-laboratory/surface-area-bet/surface-area-bet-theory