The Proximate Analysis method for the Composition Determination of Different Coal Types

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Abstract— **The global consumption of power is increasing and the shortage in finding cost effective fuel sources kept the coal as one of the biggest contributors of power generation resources or raw materials. This can be regarded as a motivator for this work. This research explains the proximate analysis method to determine the composition of coal in terms of moisture, volatiles, fixed carbon, and ash contents. Hence, the proximate analysis was established as a suitable method for defining the delivery of products obtained by heating coal under a standard set of conditions. In addition, Thermo-gravimetric analyzer TGA 4000 is used to measure the composition of various coal samples from 4 different locations mainly in the UK and Japan: (Pyehill, South Wales, Nigata and South Yorkshire). Furthermore, the thermal properties like calorific value were also determined for each sample by the bomb-calorimeter. The main results that were obtained on air-dried basis for the four different samples are as follows: fixed carbon (55.4%, 78.6%, 62.2% and 60.3%) respectively; volatile matters (28.9%, 34.4%, 29.7% and 10.8%) respectively; and ash content (12.4%, 9.7%, 6.1% and 2.8%). On the other hand, the dry basis analysis reported an increase in the percentages of (volatiles, fixed carbon, and ash) contents such as (30.2%, 63.5%, and 6.3%) respectively. Finally, finding the composition of the coal has countless value to forecast the products and amount of liberated heat that will be produced during the combustion process, as well as establishing a base for the price of coal. Based on the performed measurements, the ranks of the coal samples (Pyehill, south Wales, Nigata and South Yorkshire) were found, on dry basis. These ranks were as follows: (Sub-bituminous, Low volatile bituminous, High volatile B, bituminous and High volatile C, bituminous) respectively.**

Keywords— *Ash content, Calorific value, Coal rank, Proximate Analysis method, Thermo-Gravimetric Analysis.*

I.   INTRODUCTION

According to the International Energy Agency (IEA), Coal is one of the main sources of worldwide fuel supplies, accounting for 27% of all energy used globally and producing up to 38% of total electricity generation. It plays a vital role in many industries such as iron and steel. Nonetheless, uncertainties about air pollution and greenhouse gas emissions cloud the future of coal, [1], minerals, poorly crystalline mineralogist and elements associated with non-mineral inorganics, [2]. Its composition was given in several different ways. Determining the composition of the coal has great importance to predict the products and amount of heat that will be released during the combustion process, as well as establishing the price of coal. There are two main types of analysis commonly used to find the composition of a coal sample: the proximate analysis and the ultimate analysis [3]. It is widely thought that coal is one of our planet's mineral resources, while coal geologically is a sedimentary, organic rock, instead of being mineral. Coal does not meet the conditions of an inorganic material of a definite structure, nor is it a crystal-like solid [4]. A research paper provided a brief summary of the history, development, and variety of applications of electron microscopy techniques to analyze coals, organic-rich shales and carbonaceous materials [5]. Another research work addressed the problem of sparse data by using fuzzy logic and ensemble neural networks to estimate coal ash content from a collection of sparse data [6]. It is probably hardly necessary to state that the analyses of coal are of two kinds. Firstly, ultimate analysis providing information about the percentages of elements such as carbon, hydrogen, sulfur, nitrogen and other metals in any coal. Secondly, proximate analysis, which indicates the proportions of the composition of coal in terms of moisture, volatiles, fixed carbon, and ash content [7]. The variation of sulfur in coal is closely related to the depositional environment of coal seams [8]. The proximate analysis was developed as a convenient method for determining the distribution of products obtained by heating coal under a standard set of conditions. The analysis gives the composition in terms of moisture, ash, volatile and fixed carbon. Ash, can be defined as the residue remaining after complete combustion of the organic portion of coal. Fixed carbon is the solid residue (other than mineral ash) remaining after the removal of all the volatile matters. Therefore, it is a measure of the solid, combustible material in coal [9]. Volatile matter and heat content are employed in the classification of all coals by the American Society of Testing and Materials [10]. Proximate analysis is widely utilized in many trade applications such as buying coal for generating heat for several processes. According to the literature, proximate analyses are completed according to standard methods proposed by ASTM;

nonetheless, other approaches that vary somewhat have been used as well [11].

Parallel research was conducted on studying the proximate analysis of low and high-quality pure coal and their blends with different compositions in Pakistan. It showed that the tested samples were taken from different locations in the same country but it was representing a significant difference in its properties and its gross calorific values [12]. According to Hilt's law, supposing no magmatic heating has occurred within a given vertical arrangement of coal seams, the rank of the coals is found to rise with depth [13].

This study demonstrates the proximate analysis of the coal by using thermo-gravimetric analysis method TGA to find the composition of four coal samples to evaluate and then rank them. The method that was used depended on the TGA 4000 analyzer to determine the composition of each sample of the 4 coal samples (Pyehill, Nigata, South Wales and South Yorkshire). This research is different from other researches that were done before by analyzing different coal samples from two countries, the UK and Japan. For example, three of the samples were taken from various regions in Britain (Pyehill, South Wales and South Yorkshire) and Nigata coal samples were taken from Japan. Hence, a considerable property difference has been found and presented in the table of results. The amounts of fixed carbon, volatiles, and calorific values are extremely important to classify the coal rank. For this reason, the bomb calorimeter was used to find the calorific value (CV) of each coal sample.

II.   EXPERIMENTAL PROCEDURE

A.   Method and Materials

This work examines the coal by using two analyzing machines: the first one is the Thermo Gravimetric Analysis (TGA) that determines the proximate analysis of the coal and its composition in terms of (moisture, volatile matter, fixed carbon and Ash) by testing four different samples of coal (South Yorkshire, South Wales, Nigata and Pyehill).This method measures the amount of weight lost or gained by a sample as it is heated, cooled or held at a constant (isothermal) temperature. The physical conditions of the samples are powders saved in plastic containers as received (wet) basis at the lab temperature.

B.   Experimental Procedure and Characterization

1)   TGA

The experiment starts by putting the small ceramic crucible in the TGA and making the weight of it equal to zero. After adding 10 mg of the coal sample to the crucible, it is held inside the TGA machine by using suitable clip. Then the lid of the TGA must be closed. At the same time the balance icon on the computer program is pressed to check the weight of the sample. Then by pressing start icon the machine starts to record the changes in the temperature and the weight of the coal sample. The program records 10 readings per each second. The TGA starts working at 35 °C and this continues for 4 minutes under inert gas N_2 at a pressure equal to the atmospheric pressure, then the sample is heated from 35–110 °C. It remains at this state for 6 minutes to evaporate the moisture. Next, the coal is heated from 110 – 900°C at the rate of 80 °C/min also under inert gas condition by N_2 at the constant pressure equal to the atmospheric pressure. This stage indicates the evaporation of volatile matter by determining the loss of weight. The temperature remains constant at 900°C for 5 minutes at the presence of N_2. Then the atmosphere will be changed from N_2 to O_2, the combustion process will appear at 950 °C this stage indicates the loss of fixed carbon. Additionally, to calculate the amount of the ash content of the coal, the overall amounts of (moisture, volatile matter and fixed carbon) are subtracted from 100. Finally, the TGA is left for few minutes to be cooled via cooling water pipes inside the TGA and removed out of the crucible and cleaned. This procedure is applied on the other 3 samples.

2)   Bomb-Calorimeter

The second device that was used is the Bomb-Calorimeter, to determine the calorific value (the enthalpy of combustion) of the coal samples. The experiment starts by weighing 1 ± 0.01 g coal into ceramic crucible. Stretch 10 cm of a nichrome wire between the electrodes of the bomb cap, about 1 cm per each cab, making sure that the electrodes are clean. Hold the bomb cap on the special stand and place the crucible in its holder. Making sure that the wire is nearly in contact with the coal sample. Collecting the bomb parts and making sure that sealing ring is properly seated in its groove in the bomb cap. Then the bomb cap retaining ring is closed and tightened by hand. Fill the calorimeter's cooling vessel with 2 liters of water for cooling circuit. To start press F1 on the machine's monitor. Connect the O_2 filling tube to the bomb aligning the hole in the filler cap. Make sure the oxygen valve is open, and then press O_2 to fill in the monitor (LED).After around 30- 40 seconds you will hear a sound indicating that O_2 valve is closed automatically after supplying a pressure of around 30 bars (29.61 atm.) O_2 to the bomb. After disconnecting the filling, hold the bomb by the tommy-bar making it ready to place into the calorimeter. Put the calorimeter vessel inside the machine, and then place the bomb into the calorimeter vessel making sure that the three feet of the bomb is seated in its position in the vessel. Next, connect the two black electrodes to the bomb cap. Close the lid of the water jacket making sure that the stirrer is moving freely. To start the test, make sure the standby light is on then press start. Input your sample's identity followed by its weight on the LED monitor. The Calorimeter stabilizes both temperatures (jacket and vessel) automatically, and then the bomb is fired. After around 10 minutes of firing, the LED display records the final temperature rise. Then the bomb is removed from the calorimeter and the pin of the lid is opened carefully to release the pressure from the bomb. After few seconds the sealing ring is opened and checked if complete combustion process has occurred. Incomplete combustion can be noticed by the carbon residue inside the crucible and the sooty deposits on the inner surface of the bomb. This test is applied on the other 3 samples.

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III. EXPERIMENTAL RESULTS

A. TGA Results

The following equations were used to obtain the table of results:

$Ash_{(mg)} = W_{to.mg} - (Y_m + Y_V + Y_{FC})$ mg,

Where: mg= milligrams

$W_{to.mg}$ = original weight of the sample in mg.

Y_m= moisture content taken from the TGA graph in mg.

Y_V = volatile matter content taken from the TGA graph in mg.

Y_{FC} = Fixed carbon content taken from the TGA graph in mg.

delta Y for moisture in pyehill coal cab be calculated as follows:

Delta Y_m = 10.367gm – 9.946gm = 0.341mg

Apply the above equation on the rest of the samples.

TABLE I: THE EXPERIMENTAL RESULTS FROM TGA ON AIR DRIED BASIS FOR FOUR COAL SAMPLES.

Coal Sample	Moisture Content	Volatile Matter	Fixed Carbon	Ash
Pye Hill	0.341mg	2.985mg	5.747mg	1.294mg
South Wales	0.23mg	2.801mg	20.35mg	2.523mg
Nigata	0.529mg	7.585mg	15.89mg	1.55mg
South Yorkshire	0.377mg	5.163mg	9.052mg	0.422mg

B. Bomb-Calorimeter Results, Estimation of Calorific values:

The Calorific Value of the coal samples can be calculated using the formula given as:

CV =((Bomb Factor X Temperature Rise) – Heat Released by wire)/Mass of the dry coal sample X 10^{-3} MJ;kg

The correction factor= 96.3J per 10 cm of the wire or (9.63J/1 cm)
Original wire length = 10 cm
Bomb factor = 9866.4 J/K.

The results from the above table based on calculations and it is taken from the TGA diagrams of the 4 coal samples. In table II the (moisture, volatile matter and fixed carbon) are calculated by dividing the weight loss [Delta Y in (mg)] of each zone by the original weight of the samples. For example, the value of delta Y from (35-110) °C can be considered as the moisture loss zone. At the same time the value of delta Y from (110-900) °C can be considered as the volatile matter zone. Finally, the value of delta Y on 900°C line, which is straight line, can be considered as the fixed carbon loss zone. While the value of ash content in table II are obtained by subtracting the percentages of (moisture, volatile matter and fixed carbon) from 100.As shown in this equation:

%Ash= 100 – (%moisture + %volatile matter + %fixed carbon)
Furthermore, all results in the table achieved by applying this calculation to find (moisture, volatile matter and fixed carbon) of each of the 4 coal samples.
%Moisture= [DeltaY$_{(mg)}$ moisture zone/ Original wt$_{(mg)}$] X 100
In addition, table III explains the annual production of ash from burning Nigata and South Yorkshire coals. The annual consumption of coal is 7.5 million tones. From this ratio the amount of ash production of the coal samples can be found as follows:
Ash production from Nigata coal= (7.5 X 0.061) = 0.4575 million tons /year
Ash production from South Yorkshire coal = (7.5 X 0.028) = 0.21 million tons /year

TABLE II: PROXIMATE ANALYSIS OF SAMPLES

Coal Sample	Coal Rank	Moisture Content%	Volatile Matter %	Fixed Carbon %	Ash %	CV, MJ/kg
1.PyeHill	Sub-bituminous	3.3	28.9	55.4	12.4	25.87
2.South Wales	Low volatile bituminous	0.9	10.8	78.6	9.7	32
3.Nigata	High volatile B, bituminous	2	29.7	62.2	6.1	30.17
4.South Yorkshire	High volatile C, bituminous	2.5	34.4	60.3	2.8	29.34

TABLE III : PROFILE OF COAL - FIRED ELECTRIC POWER PLANT

Annual Coal Consumption	Annual Ash Production burning Coal 3	Annual Ash Production burning Coal 4
7.5 million tons	(7.5 X 0.061) = 0.4575 million tons	(7.5 X 0.028) = 0.21 million tons

C. Dry Analysis Calculations

[(100) / (100 - M)]
Where M = moisture content
The coal rank determined on dry basis according to the American Society for Testing and Materials (ASTM) system is attached in table IV.

TABLE IV: DRY BASIS ANALYSIS TABLE

Coal Sample	Coal Rank	Volatile %	Fixed carbon %	Ash %	CV MJ/kg
Pye Hill	Sub-bituminous	29.9	57.3	12.8	26.75
South Wales	Low volatile bituminous	10.9	79.3	9.8	32.3
Nigata	High volatile B, bituminous	30.3	63.5	6.2	30.77
South Yorkshire	High volatile C, bituminous	35.3	61.8	2.9	30.07

IV. ANALYSIS AND DISCUSSION

A. TGA result Illustration

The experimental results of the 4 coal samples in table I, the TGA on air dried basis can be represented in the following 4 graphs.
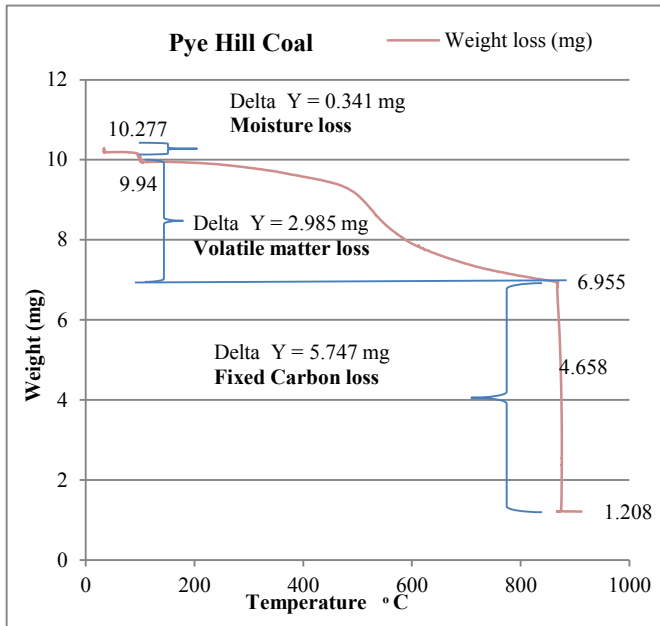
Fig. 1: The weight loss of Pyehill coal sample with the temperature rise
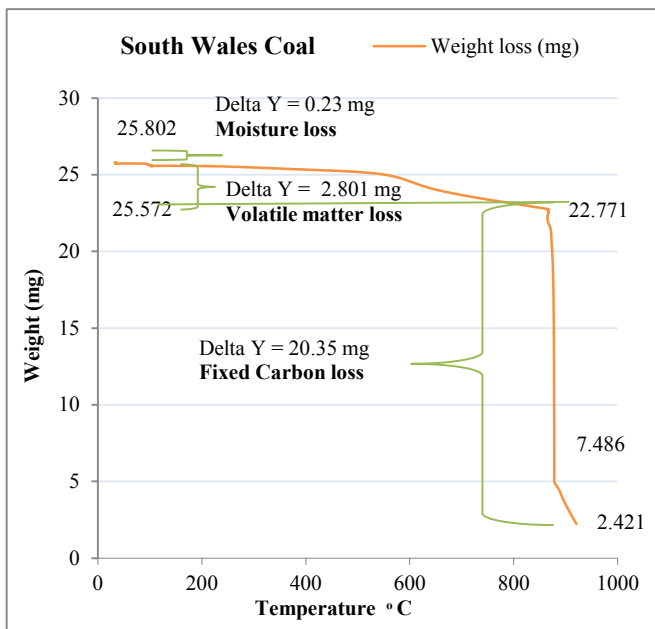
Fig. 2: The weight loss of South Wales's coal sample with the temperature rise.
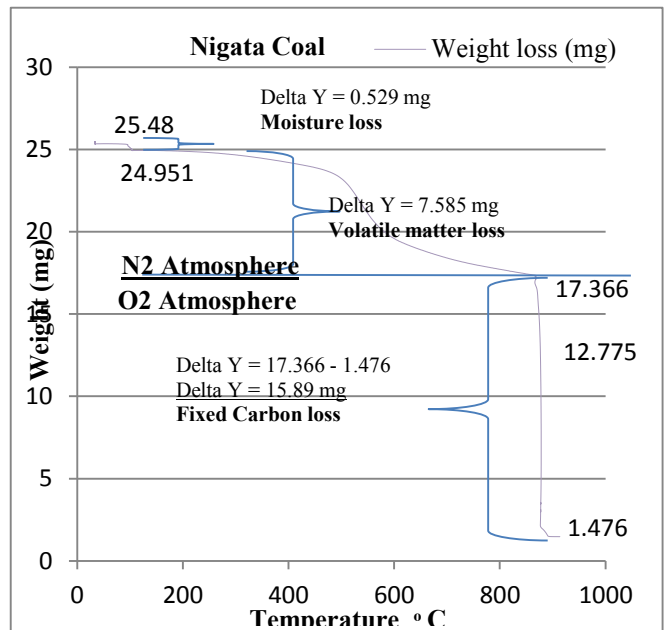
Fig. 3: The weight loss of Nigata coal sample with the temperature rise.
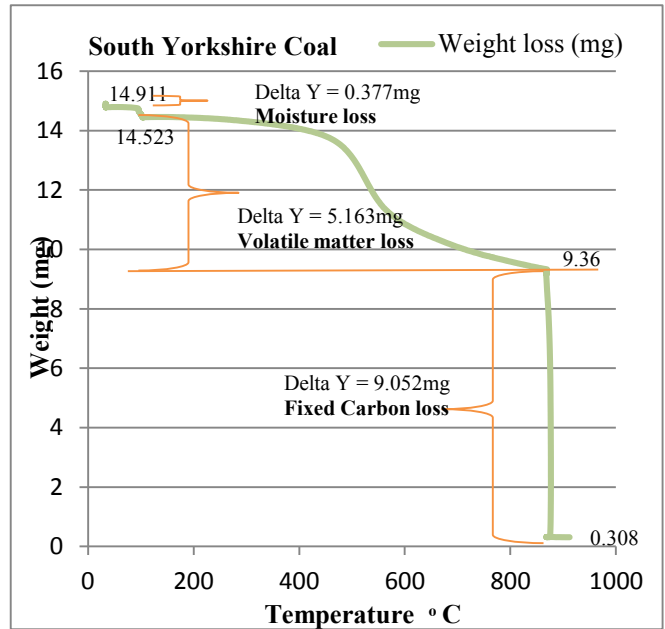
Fig. 4: The weight loss of South Yorkshire coal sample with the temperature rise

B. TGA result Analysis

The above graphs represent the change in the sample weight with increasing the temperature. There is slight difference between the original sample weight and the first weight recorded on the graphs. For example, the original weight of the Pyehill sample was (10.367 mg), while the graph started from (10.281 mg). This difference is because the original weight was measured at the room temperature around (20 °C), while the first point of the graph was recorded at the TGA conditions, which is (35 °C). When the temperature was increasing inside the TGA, the sample was losing some of its weight due to loss of the moisture. From this it can be

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assumed that there were two periods of moisture loss. Firstly, there is limited moisture loss at (20 – 35 °C). Secondly, all the moisture evaporated at (35 - 110°C). Then, the volatile matter loss takes place at (110 - 900°C) at the presence of nitrogen. Finally, the TGA atmospheric condition changed from N_2 to O_2 at 900°C fixed carbon started to burn. The main factors that can cause errors or effects on the samples are: (temperature and method). Furthermore, the scan rate has considerable effects on the experiment for example: fast scan has (quick survey and fast turnaround time). While slow scan rate has (best resolution, good on set determinations and provides the time to kinetics to happen). In addition, sampling methods have considerable role in the thermo-gravimetric analysis of coal. For example, the size and the physical condition of the samples have many effects such as: gaining and losing moisture from the atmosphere.

%Ash= [(mass after combustion) / (original mass)] X 100

%Volatiles= [(Mass of sample after heating) / (Mass of original sample)] X 100

%Fixed Carbon = 100 – (%moisture + %volatile + %ash)

Fixed carbon reported the highest percentage among other compositions of the coal. This percentage varied depending on the coal sample. For example, the highest fixed carbon percentage was reported in south Wales's coal as about 78.6%. While, the lowest fixed carbon percentage was reported in Pyehill's coal as about 55.4%. In addition, the percentages of volatile matters in the coal samples have considerable effects on the coal rank.

The Coal composition on air dried basis can be shown as below
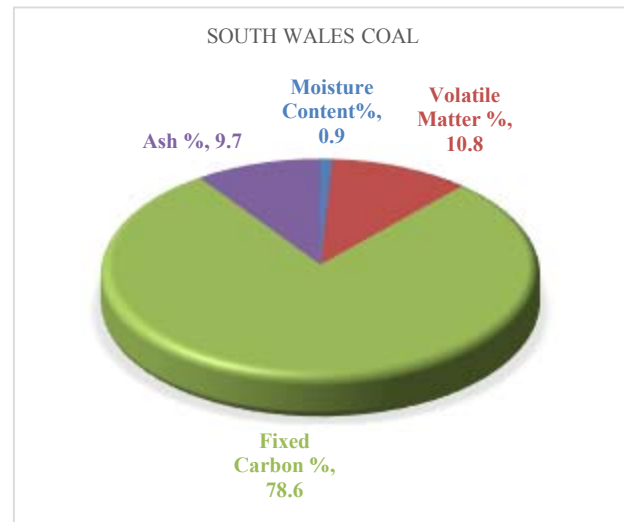
Fig. 6: The composition of South Wales's coal.
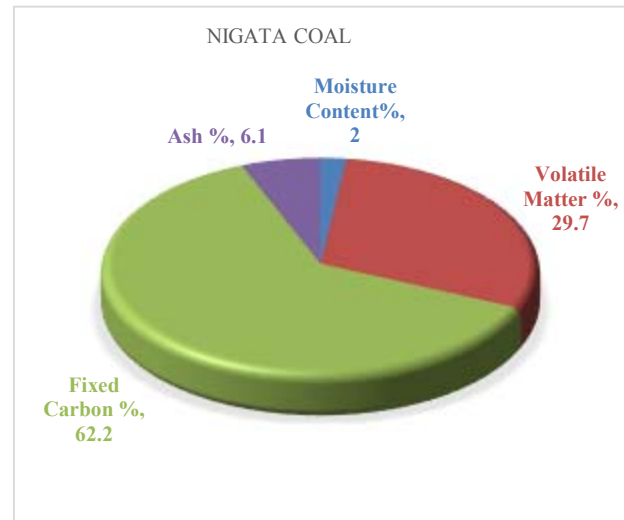
Fig. 7: The composition of Nigata coal.
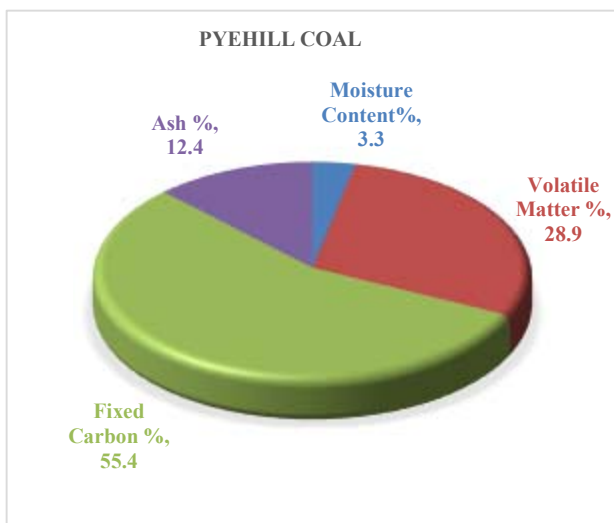
Fig. 5: The composition of Pyehill coal
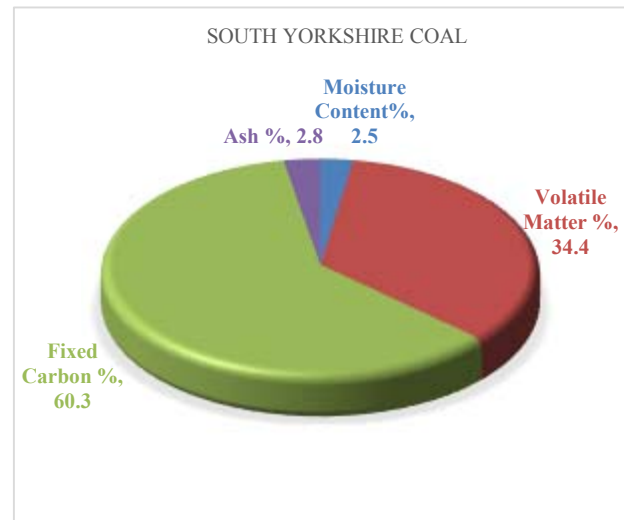
Fig. 8: The composition of South Yorkshire coal.

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C. Discussions

It can be observed from the dry basis analysis results in table IV , that the South Wales coal can be ranked as low volatile bituminous, it has the lowest volatile matter content about 10.9%, while the low-volatile bituminous coal has around 14-22% volatile matter content according to the (ASTM). This sample was ranked according to its carbon content, because it has high fixed carbon content about 79.3% on dry basis. On the other hand, the fixed carbon content in samples 1, 3 and 4 are under 69% then calorific value ranges are used to classify them. Hence, for the above reason the fixed carbon weight percentage has an effect in classifying sample 2. Nigata's coal and south Yorkshire's coal have the same coal rank (high volatile bituminous). They can be ranked according to their fixed carbon content and calorific values about (63.5% and 61.8%) and (30.17MJ/kg and 29.34MJ/kg) respectively on dry basis. Finally, it is found that the amount of ash produced from coal 3 is more than the ash produced from coal 4 that means the inorganic content in coal 3 is more than coal 4 which increases the loss in energy produced from coal 3.

V.   CONCLUSIONS

Based on this work, the following conclusions may be made:

- Proximate analysis of coal has significant role to measure the composition of coal and classify it according to the ASTM.
- The TGA 4000 analyzer contributes directly to, comparatively, measure the amounts of the moisture, volatiles, fixed carbon and ash contents in 4 different coal samples (Pyehill, south Wales, and South Yorkshire) from the UK and Nigata coal from Japan.
- Based on the performed measurements, the ranks of the coal samples (Pyehill, south Wales, Nigata and South Yorkshire) were found, on dry basis. These ranks were as follows: (Sub-bituminous, Low volatile bituminous, High volatile B, bituminous and High volatile C, bituminous) respectively. This ranking is important to determine the most valuable coal resources as best fuels for power and electricity generation.
- The highest percentage of fixed carbon was reported in south Wales's coal being about (78.6 %) with a calorific value of (32 MJ/kg), compared to the lowest percentage of fixed carbon which was reported in Pyehill's coal sample being about 55.4 % with calorific value of 25.87 MJ/kg.

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