A PROJECT REPORT
ON
"Determining coking properties using differential fractions of coal samples"

Submitted in Fulfillment of Vocational Trainee-May 2024
Training Programme at RDCIS, Ranchi
By
Anubhav Jangde
21021110
Lokendra Singh Chandel
21021125
Dushyant Singh Karel
21021117

सेल SAIL

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Under the supervision of

	P. K. Pankaj
B. Chakraborty	Dr. B. K. Sahoo
S. K. Kushwaha	Dr. Bablu Ghosh
	A.Kumar

ACKNOWLEDGMENTs

Thanking You,

Anubhav Jangde   Lokendra Singh Chandel
Dushyant Singh Karel

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INTRODUCTION

The training at RDCIS comprised of acquaintance with the characterization of coal/coke samples and a technical project on:-

"Coking- property determination by differential fractions of coal samples".

This report is the outcome of the training at RDCIS in the Coal Carbonization Laboratory carried out from 20th May-20th June, 2024.

About RDCIS:-

The Research & Development Centre for Iron & Steel (RDCIS) at Ranchi is the corporate R&D unit of SAIL. Set up in 1972, the Centre has ISO: 9001 certification to its credit. It undertakes R&D projects in diverse realms of Iron & Steel Technology under the categories of Plant Performance Improvement (PPI), Product Development (PD), Scientific Investigation and Development (SID), Basic Research (BR) and Technical Services (TS).

RDCIS has around 236 dedicated and competent scientists and engineers and its laboratory is equipped with around 300 sophisticated diagnostic research equipment and 5 pilot plant facilities.

RDCIS provides customers with prompt, innovative and cost-effective R&D solutions; develop and commercialize improved processes and products; continually enhance the capability of its human resources to emerge as a centre of excellence. The major efforts are directed towards cost reduction, quality improvement and value-addition to products of SAIL plants and providing application engineering support to SAIL's products at customers' end. RDCIS, along with steel plants, takes initiatives to develop special steel products utilizing the modernized production facilities at steel plants.

RDCIS also offers technological services to various organizations in the form of Know-how transfer of technologies developed by RDCIS; Consultancy services; specialized testing services; Contract research; Technology Awareness Programmes.

RDCIS offers technological services in the form of

- Know-how transfer of technologies developed by RDCIS
- Consultancy services / Contract research
- Specialised testing services
- Training

Major Research Facilities

The research facilities comprise of 15 major laboratories encompassing 6 pilot facilities and 350 equipment and advanced diagnostic facilities. The major areas of expertise of coal and coke division are as follows:

- Characterization of coal, coke and coke chemicals.
- Selection of coals and blend formulation/optimization through laboratory characterization and pilot oven carbonization tests.
- Investigation on pre-carbonization technologies such as group wise crushing coals, partial briquetting of coal charge, stamp charging through pilot oven carbonization tests.
- Group wise crushing of coals.
- Improvement in coke quality through control of operational parameters.
- Computerized combustion control system.
- Coke degradation and stabilization.
- Improvement in yields of primary coal chemicals.
- Production of new coal chemicals.

COAL

Coal is an organic rock (as opposed to most other rocks in the earth's crust, such as clays and sandstone, which are inorganic); it contains mostly carbon (C), but it also has hydrogen (H), oxygen (O), sulfur (S) and nitrogen (N), as well as some inorganic constituents (minerals) and water (H_2O). Most of the world's coal exists in the northern hemisphere.

Coal Formation

Coal was formed from prehistoric plants, in marshy environments, some tens or hundreds of millions of years ago. The presence of water restricted the supply of oxygen and allowed thermal and bacterial decomposition of plant material to take place, instead of the completion of the carbon cycle. Under these conditions of anaerobic decay, in the so-called biochemical stage of coal formation, a carbon-rich material called peat was formed. In the subsequent geochemical stage, the different time-temperature histories led to the formation of coals of widely differing properties, as summarized in Table below:-

Carbon content and age of different coals

Coal type	Approximate age (years)	Approximate carbon content, %
Lignites	60,000,000	65-72
Subbituminous coals	100,000,000	72-76
Bituminous coals	300,000,000	76-90
Anthracites	350,000,000	90-95

Properties, types and ranks of Coal

Because of wide variations in the composition and properties of coals, a classification system is needed to describe the different kinds available for use in homes and power plants. One such system, most commonly used is summarized below:-

	<---------- Low Rank -------><---- High Rank ------------>			
Rank:	Lignite	Subbituminous	Bituminous	Anthracite
Age:	------------------------------------ increases ---------------------------------->			
% Carbon:	65-72	72-76	76-90	90-95
% Hydrogen:	~5 ----------------------- decreases ----------------------- ~2			
% Nitrogen:	<----------------------------------- ~1-2 ----------------------------------->			
% Oxygen:	~30 ---------------------- decreases ----------------------- ~1			
% Sulfur:	~0 ------------ increases ------------ ~4 --- decreases --- ~0			
%Water:	70-30	30-10	10-5	~5
Heating value (BTU/lb):	~7000	~10,000	12,000–15,000	~15,000

The chemical approach to characterizing coals is to determine the amounts of the principal chemical elements in them. In the jargon of the coal business, this procedure is called the ultimate analysis of coal. Carbon and hydrogen are the principal combustible elements in coal. On a weight basis, carbon is the predominant one. It constitutes about 60% to about 95% of the total. For most coals of 90% or less carbon, hydrogen content is generally in the range of 5%; it drops to about 2% for coals having 95% carbon. Nitrogen content of almost all coals is in the range of 1-2%. Oxygen content is inversely related to carbon content. For example, coals of 65% carbon may contain 30% oxygen, while coals of 95% carbon may contain only 2-3% oxygen; this is significant because the more oxygen a coal contains, the easier it is to start to burn it, or to achieve its ignition. Sulfur content of coals is seen to be quite variable.

Another way to characterize coals is to determine the thermal energy released when the coal is burned. We can also measure other characteristics of coals that help predict how coals will behave when handled and burned. This series of tests is called the proximate analysis of coal. Four characteristics are measured by the proximate analysis: moisture, volatile matter, fixed carbon, and ash.

Coal Utilization

There are three major pathways for coal utilization. These are illustrated in Figure below:-
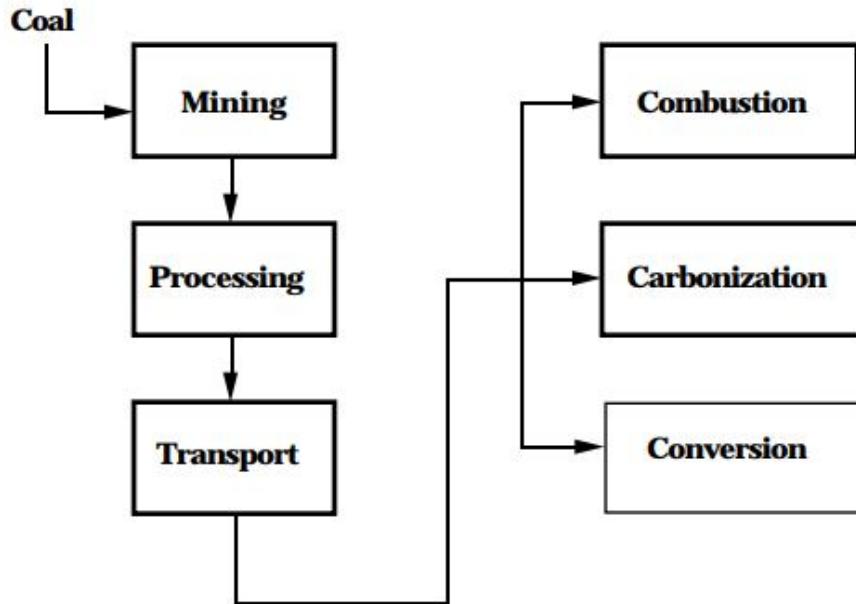
The principal process by which coal is used is combustion; Combustion involves burning the coal in air to liberate thermal energy (heat). The heat is used as such for comfort or to carry out many industrial processes that require high temperatures; it is also used to generate steam for use in electric power plants. Carbonization is the heating of coal to high temperatures in the absence of air; it is used in manufacturing coke for the metallurgical industry.

Distribution of coal utilization pathways is as follows:-
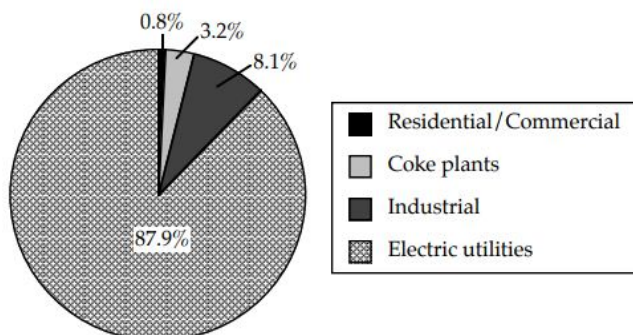
Coal Carbonization (Coking) and coke

The second largest use of coal is the manufacture of coke for the metallurgical industry. Iron and its alloys (particularly the various kinds of steel) are perhaps the most versatile metallic construction materials known to humankind. Unfortunately, iron occurs in nature not as metallic iron but rather in ores containing iron compounds such as oxides or carbonates. The production of metallic iron requires a chemical reaction to remove the oxygen from the iron compounds and liberate the iron as the metal. The conversion of a metal oxide to metal is an example of a general type of chemical reaction known as reduction (as opposed to the inverse process called oxidation). Substances that we add to the reaction to cause the reduction to take place are termed reducing agents. Among the most powerful types of reducing agents, and among the least expensive, are various forms of carbon or substances rich in carbon.

Coals that pass through a plastic stage on heating are called **caking coals**. Some caking coals resolidify on heating to form a hard, very strong, carbon-rich porous mass suitable for use as a reducing agent in the metallurgical industry. This material is called **coke**, and the special class of caking coals that yield a satisfactory coke are known as **coking coals**.

To be useful in the metallurgical industry, the coke needs to meet five criteria:-

- To be a useful reducing agent, the coke must have very high carbon content.
- To keep the iron reasonably pure, the coke must have low contents of sulfur and ash.
- To provide ample heat, the coke must have a high content of fixed carbon and have a high calorific value.
- To let air pass through the fuel bed, but yet keep the fuel bed from being collapsed by the weight of iron ore, the coke must be quite porous and very strong.
- Finally, to help keep the cost of the iron low, the coke must be cheap.

Process

Coal carbonization involves heating of coal in the absence of air in coke oven plant. Coke making process is multistep complex process and variety of solid liquids and gaseous products are produced which contain many valuable products. Various products from coal carbonization in addition to coke are coke oven gases. coal tar, light oil, and aqueous solution of ammonia and ammonia salt. Coke oven gases are about 310-340 cum per tone of dry coal which contains gaseous products, coal tar vapours, light oil and water.

Coke Oven Plant

Due to the development of iron and steel industry coke oven plant has become an integral part of iron and steel industry. Due to increasing demand of iron and steel, there has been a considerable increase in the coke oven capacity which resulted increase output of coal chemicals.

Two types of coke manufacturing technologies use are:-

- Coke making through by product recovery
- Coke making through non-recovery/ heat recovery

In India, building of coke oven batteries was initiated in the beginning of the ninth century; now about 3000 ovens are in operation/ construction in the coke oven plant. By product from coal gasification plant includes coke, coal tar, sulphur, ammonia. Coal tar distillation produces tar, benzol, cresol, phenol, creosote.

Coal Handling Plant and Coal Preparation

Section Coal needs to be stored at various stages of the preparation process, and conveyed around the coal preparation section. Crushing and screening are the important part of coal handling plant. Crushing reduces the overall size of the coal so that it can be more easily processed and handled. Screens are used to ranges the size of coal. Screens can be static, or mechanically vibrated. Dewatering screens are used to remove water from the product.

Various sections in coke oven plant are:-

Coal Handling Plant and Coal Preparation Section	To prepare coal blend suitable for carbonization. various steps involved are unloading and storage of coal, blending of coal of various grade, coal crushing and transport to coal storage tower
Partial briquetting	To prepare briquette of coal to charge along with coal into the coke oven.
Coke oven Batteries	To convert coal into coke by carbonizing coal in absence of air. The process steps involved are coal charging and coal carbonisation
Coke sorting Plant	Crushing and sorting of coke to suitable size for use in blast furnace. The steps involved are coke pushing, coke quenching, coke crushing/ screening
Coke oven gas recovery	Collection and cleaning of coke oven gas and recovery of by products. . This involves gas cooling, tar recovery, desulphurization of coke oven gas , recovery of ammonia, recovery of light oil
Ammonia recovery and Ammonium Sulphate Production.	Recovery of ammonia and neutralization with sulphuric acid or nitric acid in case of ammonium nitrate/ calcium ammonium nitrate.
Waste water treatment	Treatment of phenolic waste water

Coke oven Batteries:-

Coke oven plant consists of Coke oven batteries containing number of oven (around 65 ovens in each battery). The coal is charged to the coke oven through charging holes. The coal is then carbonized for 17-18 hours, during which volatile matter of coal distills out as coke oven gas and is sent to the recovery section for recovery of valuable chemicals. The ovens are maintained under positive pressure by maintaining high hydraulic main pressure of 7 mm water column in batteries. The coking is complete when the central temperature in the oven is around 950-1000 o C. At this point the oven is isolated from hydraulic mains and after proper venting of residual gases, the doors are opened for coke pushing. At the end of coking period the coke mass has a high volume shrinkage which leads to detachment of mass from the walls ensuring easy pushing. The coke is then quenched and transferred to coke sorting plant.The control of oven pressure is quite important because lower pressure leads to air entry while higher pressure leads to excessive gassing, leakage of doors, stand pipe etc. Proper leveling of coal is important and care is taken so that free board space above (300 mm) is maintained to avoid choking.

By-product from Coke Oven Plant

The high temperature carbonization is used for production of coke for use in blast furnace. Various by-products obtained from coal carbonization are crude tar, crude benzoyl, and ammonia. Typical yield of some important byproduct are: Tar 3.2%, ammonium sulphate 1.1%, crude benzoyl 0.9%. Gas Condensation section: Coke oven gas containing water vapours and chemical products of coking (tar, ammonia, benzoyl etc. at temperature about 750-800o C from the coke oven plant is cooled to temperature of 80-82o C. During gas cooling 65-70% of the tar is condensed. Further cooling of gas, the water vapors and the remaining part of the target condensed along with some ammonia and other chemicals.

Typical Analysis of Coke Oven Gas is:-

Methane	26.0%
Hydrogen	56.5%
Hydrocarbons	2.3%
Carbon monoxide	8.5%
Carbon dioxide	3.0%
Oxygen	0.4%
Nitrogen	3.3%
Density	0.4848 kg/m^3
Calorific value	4300 kcal/m^3

Cleaner Technologies in Coke Oven Plant:-

Coke oven plants are one of the highly polluting industries. Continuous development has been there to reduce the pollution load and energy consumption. Some of the cleaner technology are modified wet quenching, coke dry quenching, coal moisture control,, high pressure ammonia aspiration system, modern leak proof doors, advance technologies for desulphurization of coke oven plant.

Fig. Coke Oven Plant & Batteries

Fig. Coke oven Batteries   **Fig. Dry Quenching of Coke**

Properties of Coke

Ash

Ash in coke is inert & becomes part of the slag produced in the Blast Furnace. Hence, ash in coke not only takes away heat but also reduces the useful volume of the furnace. Hence it is desirable to have lower ash content in the coke. The desired ash content is up to 12.5% (Max.) at NINL.

Volatile Matter (VM)

The VM in coke is an indicator of completion of carbonization & hence the quality of coke produced. It should be as low as possible i.e. < 1%.

Gross Moisture (GM)

It has got no role to play in the furnace. It only takes away heat for evaporation. Hence least moisture content is desirable. A level around 1% (Max.) is desirable.

MICUM Index

Micum index indicates the strength of coke. M10 value indicates the strength of coke against abrasion. Lower the M10 value better is the abrasion strength. A M10 value of <7 indicates good coke strength. M10 of NINL coke is less than 7%. M_{40} value indicates the load bearing strength or strength against impact load. Coke having lower M_{40} value will crumble inside the Furnace which will reduce the permeability of the burden and cause resistance to the gasses formed in the furnace to move upwards. A good coke should have a M_{40} value more than 80.

Coke Reactivity Index / Coke Strength After Reaction (CRI / CSR)

The coke strength after reaction (CSR) test is based on a process developed in the 1970s by Nippon Steel Corporation as an effort to get an indication of coke performance. It refers to the coke's strength (thermal potential) via a simulated reaction emulating what might be expected in an industrial blast furnace. Those original tests over four decades ago have become an accepted standard for testing coke, mainly when blending coking coal to guarantee it performs to the essential specification.

Fig. Coke reactivity test unit   Fig. Coke reactivity test resort

Fig. CSR measurement
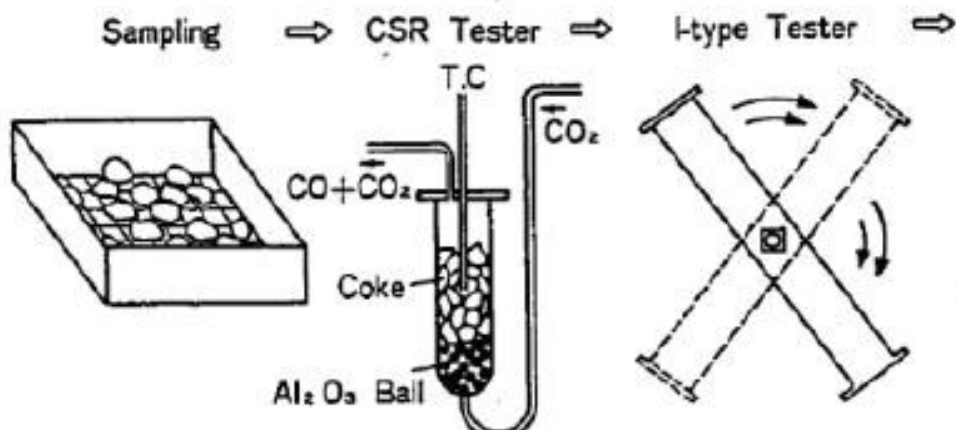
In the test, a 200 g sample of 20 mm coke fragments are put in a carbon dioxide atmosphere at 1 – 2 bar pressure and then heated at 1100 ˚C for two hours. The coke is preheated and cooled under nitrogen and the weight loss during reaction is measured. The percentage weight loss is measured against the Coke Reactivity Index (CRI) for which the universal standard is ISO 18894 (2006).

The reacted coke is put in an I-tester cylinder chamber and subjected to 600 revolutions in 30 minutes. The percent of carbon material eliminated from the drum that is +10 mm after sieving is the CSR.

This recognized test procedure in a laboratory imitates coke lumps descending in an industrial blast furnace where they are subjected to abrasion as they rub together and against the walls of the furnace in a current of CO_2. These two simultaneous processes chemically react with the coke lumps generating an excess of fines that can reduce burden permeability resulting in increased coke requirement rates and lost hot metal production.

A typical coke reactivity furnace (from a manufacturer such as CarboliteGero) has a highest operating temperature of 1100 ˚C with a controller that meets either the ASTM D 5341-14 or ISO 18894 (2006) standard. CarboliteGero's furnace has three distinct heated zones with low thermal mass insulation across a 700 mm length. There is also a gas safety system and audible alarms for over- temperature, flame failure and low gas flow. Most significantly, the interior of the dual walled furnace houses an Inconel retort, which is corrosion and oxidization resistant and which allows incoming gas to be preheated.

It is the capacity of the coke to remain intact by withstanding the reactive atmosphere inside the furnace. Hence less the CRI value, better is the coke. Desirable value should be around 22.

Coke Size

The size of coke is most important to maintain permeability of the burden in the furnace. The required size for Blast Furnace is more than 20mm size & less than 80mm size. If the undersize is more the permeability decreases as smaller coke pieces fill up the voids & increase the resistance to the flow of outgoing gasses. If the oversize is more the surface area of coke for the reactions reduces. Hence the size of the coke is to be maintained between +20mm & -80mm Coke Sorting Plant, where sizing & Cutting takes place. The Coke is taken to an 80mm screen. The +80mm coke fractions are sent to coke cutter to bring down the size. The hard coke of size +20mm to -80 mm size are then segregated to send to Blast Furnace. Coke fraction of +10mm to -20mm, which is called Nut coke, is also segregated & sent to RMHS. The -.10mm fractions, called fine Breeze or Breeze Coke, are also sent to RMHS. (Breeze = +3 to 10mm and Dust – 3mm).

Tests on Coal

1) Proximate Analysis

Proximate analysis indicates the percentage by weight of the Fixed Carbon, Volatiles, Ash, and Moisture Content in coal. The amounts of fixed carbon and volatile combustible matter directly contribute to the heating value of coal. Fixed carbon acts as a main heat generator during burning. High volatile matter content indicates easy ignition of fuel. The ash content is important in the design of the furnace grate, combustion volume, pollution control equipment and ash handling systems of a furnace. A typical proximate analysis of various coal is given in the following Table.

Parameter	Indian Coal	Indonesian Coal
Moisture, %	5.98	9.43
Ash, %	38.63	13.99
Volatile Matter, %	20.70	29.79
Fixed Carbon, %	34.69	46.79

Measurement of Moisture

Determination of moisture is carried out by placing a sample of powdered raw coal of size 200-micron size in an uncovered crucible and it is placed in the oven kept at 108 ± 2°C along with the lid for 1 hour. Then the sample is cooled to room temperature and weighed again. The loss in weight represents moisture.

Measurement of Volatile Matter

Fresh sample of crushed coal is weighed, placed in a covered crucible, and heated in a furnace at 900 ± 15°C for 7 minutes. The sample is cooled and weighed. Loss of weight represents moisture and volatile matter. The remainder is coke (fixed carbon and ash).

Measurement of Carbon and Ash

The cover from the crucible used in the last test is removed and the crucible is heated over the Bunsen burner/ in furnace until all the carbon is burned at 800 ± 10°C. The residue is weighed, which is the incombustible ash. The difference in weight from the previous weighing is the fixed carbon. In actual practice fixed carbon is derived by subtracting from 100 the value of moisture, volatile matter and ash.

Significance of Various Parameters in Proximate Analysis

a) Fixed carbon: Fixed carbon is the solid fuel left in the furnace after volatile matter is distilled off. It consists mostly of carbon but also contains some hydrogen, oxygen, sulphur and nitrogen not driven off with the gases. Fixed carbon gives a rough estimate of heating value of coal

b) Volatile Matter: Volatile matters are the methane, hydrocarbons, hydrogen and carbon monoxide, and incombustible gases like carbon dioxide and nitrogen found in coal. Thus the volatile matter is an index of the gaseous fuels present. Typical range of volatile matter is 20 to 35%.

- Proportionately increases flame length, and helps in easier ignition of coal.
- Sets minimum limit on the furnace height and volume.
- Influences secondary air requirement and distribution aspects.
- Influences secondary oil support

c) Ash Content [Mineral Matter]: The mineral matter in coal is converted into ash during combustion. Ash is an impurity that will not burn. Typical range is 5 to 40%. Mineral matter may be inherent or extraneous. Inherent mineral matter is inorganic material of the original vegetable substance. The extraneous mineral matter of coal is mainly clay or shale consisting of aluminium silicates of different composition.

- Reduces handling and burning capacity.
- Increases handling costs.
- Affects combustion efficiency and boiler efficiency
- Causes clinkering and slagging.

d) Moisture Content: Moisture in coal must be transported, handled and stored. Since it replaces combustible matter, it decreases the heat content per kg of coal. Typical range is 0.5 to 10%.

- Increases heat loss, due to evaporation and superheating of vapour
- Helps, to a limit, in binding fines.
- Aids radiation heat transfer.

e) Sulphur Content: Typical range is 0.5 to 0.8% normally.

- Affects clinkering and slagging tendencies.
- Corrodes chimney and other equipment such as air heaters and economisers
- Limits exit flue gas temperature.
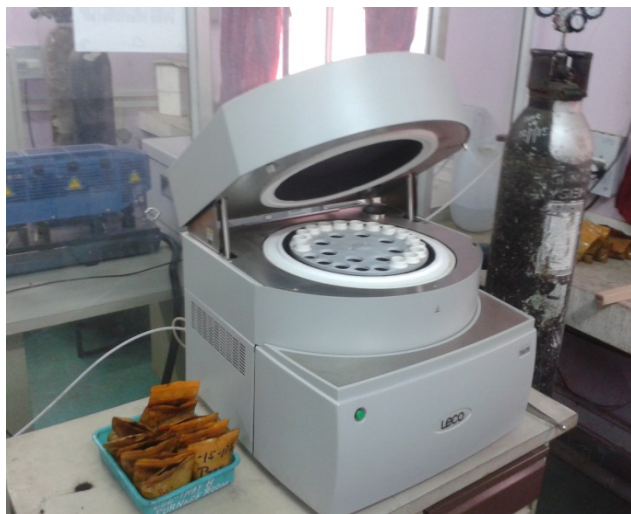
Fig. LECO TGA 701 Thermo Gravimetric Analyser

2) Free Swelling Index / Crucible Swelling Number

Some types of coal during and after release of volatile matter become soft and pasty and form agglomerates. They are called caking coal, Sue to this the coal does not burn completely yielding low combustion efficiency. The coal that does not agglomerate is called free-burning coal. Qualitative evaluation method of determination of extend of caking is called swelling index. Free burning coal has very high value of swelling index. For pulverized coal burners swelling property of coal is of less importance.

Introduction:

The swelling number of coal, as determined by the crucible swelling number test, described below, is intended solely to give some comparative measure of the swelling properties of coals. From a consideration of average error, it has been ascertained that the mean result of four test on the sample is correct to within ±1 unit in 99 out of 100 cases and within ±0.5 unit in 80out of 100 cases, there is thus some assurance that different investigators can closely reproduce results on the same coal. The source of heat used in the test is a gas burner or electrically heated furnace. The international organization standardization (ISO) has recommended both the heating methods.

Procedure:

1. Weigh 1.00 to 1.01g of freshly ground coal into a crucible.
2. Lightly tap the crucible 12 times on the bench to level the surface of the coal.

3. Cover the crucible with the lid and place it upright in the silica triangle supported in the draught shield.
4. Light the gas, heat for such time as is required for the flame of the burning volatile matter to die out, and in case for not less than 2.5 minutes.
5. Allow the crucible to cool and carefully remove the coke button.
6. Repeat the test until four buttons have been obtained.

Observation:

1. Compare the coke button with the standard numbered outlines in fig.3.
2. For the comparison, rotate the button about its axis so that the largest profile is presented to view.
3. A method of viewing which excludes the effect of parallex is shown in fig.4.
4. Place the drawing with which the buttons is to be compared exactly in the centre of the field of vision from the top of the tube.
5. Arrange the button so that the maximum cross section is in line with the drawing when viewed with one eye placed immediately over the top of the tube.

The swelling index of a button is number inscribed in the outline that its largest profile most nearly matches. Report the mean swelling number of the series, expressed to the nearest half number.

*   For non-swollen button, the number'0'is used to describe coals which give a powder residue. The number '1/2'deescribes coals which give a coherent residue that will not bear a 500gm mass. The number '1'describes coals which give a coherent residue that cracks into two or three hard pieces when the 500g mass is applied.

FIG. 3   STANDARD   PROFILES   AND   CORRESPONDING   CRUCIBLE   SWELLING   NUMBERS
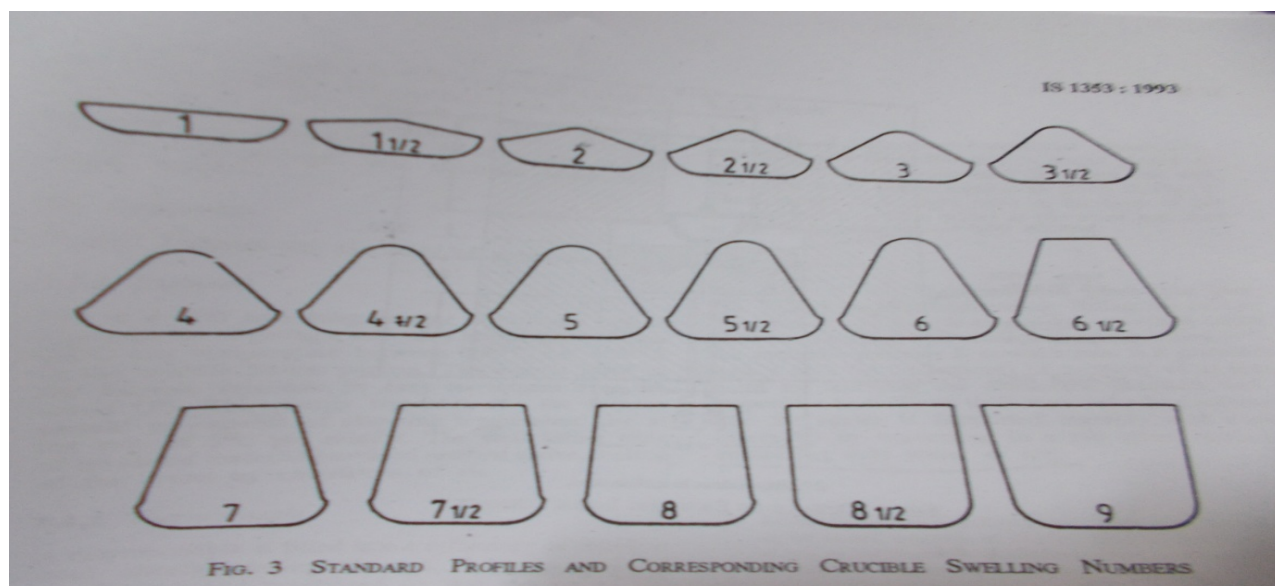
3) Low Temperature Gray-King carbonization (LTGK)

This test requires the heating of 20 gm of coal in a special horizontal retort tube at a rate of 5°C/minute from 300°C to 600°C. The coke pencil produced is classified according to set standards. The residue is visually compared with standard profiles and a letter A to G or G1 to G9 is assigned, representing LTGK Coke Type. A represent non-coking. Letters B to G9 indicates increasing order of caking and swelling capacity. If the coal has >17% ash the sample has to be washed before testing.
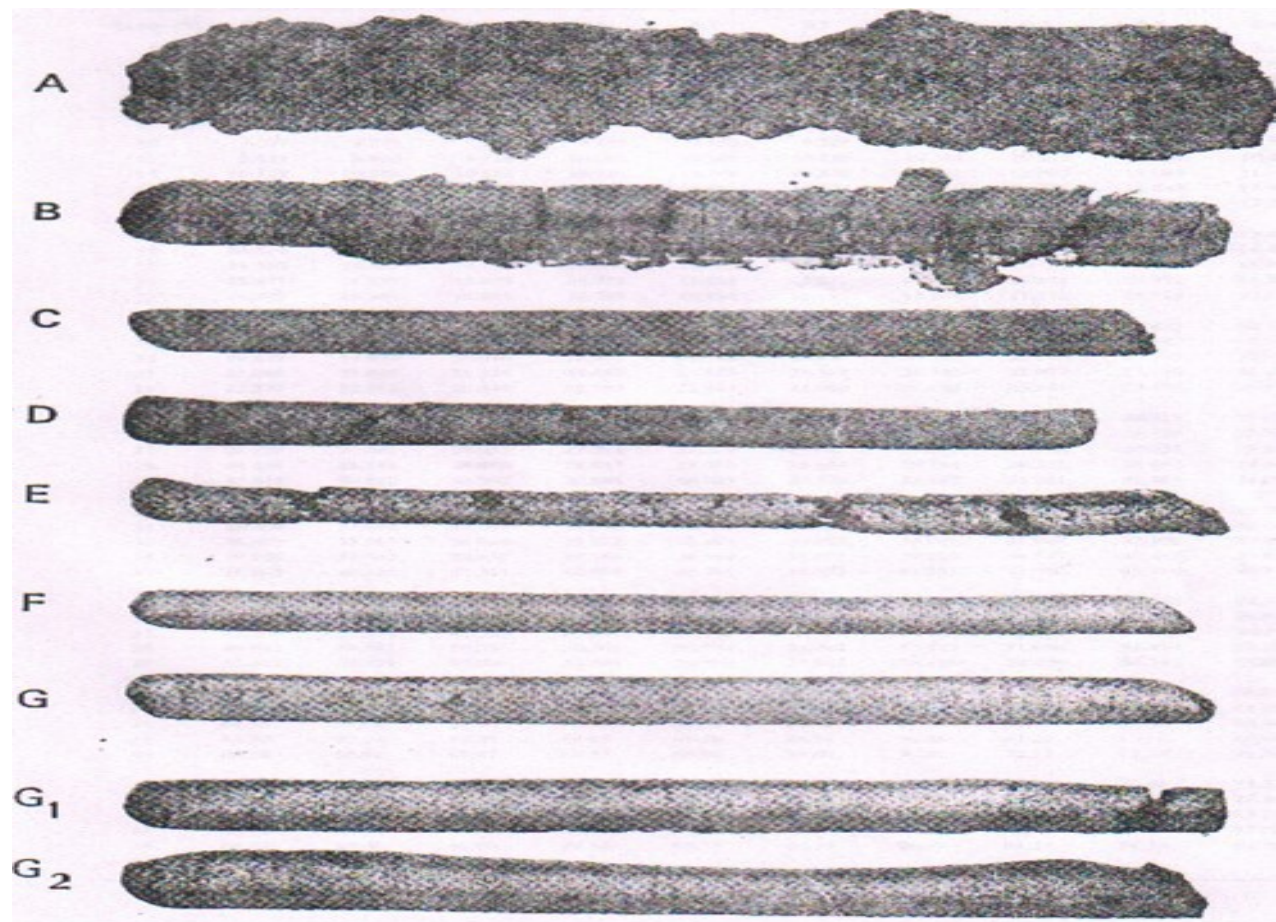
4) Petrography

Petrography is a test used to describe the percentage content of reactive and inert material in a coal. The inert material makes the coal strong while the reactive material gives the coal its chemical properties. When observed under a specially designed microscope that magnifies the image 500 times, the larger granules represent the inert part while the smaller ones denotes the reactive part. The coal in which entire reactive matter has been converted to inert is the finest and is numbered 12
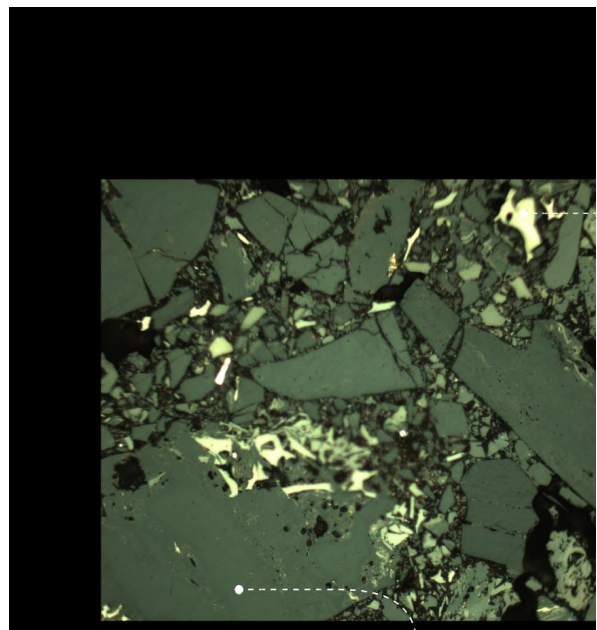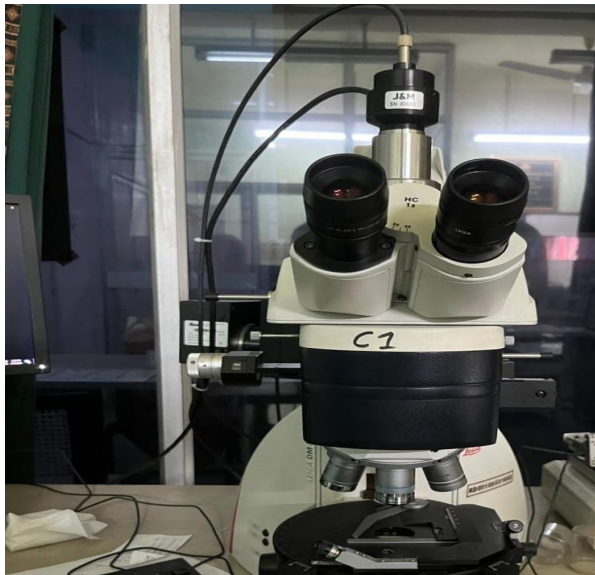
Fig. Petrography Microscope and Results

5) Elemental Analysis (CHNS)

The CHNS(O) Analyzer find utility in determining the percentages of Carbon, Hydrogen, Nitrogen, Sulphur and Oxygen of organic compounds, based on the principle of "Dumas method" which involves the complete and instantaneous oxidation of the sample by "flash combustion". The combustion products are separated by a chromatographic column and detected by the thermal conductivity detector (T.C.D.), which gives an output signal proportional to the concentration of the individual components of the mixture.

There are different techniques for the determination of CHN\CHNS\O. It brings a new level of precision, accuracy, speed of analysis and ease of operation.The built in chromatographic column converts the compound and elutes it in the form of NO_2,CO_2, SO_2,H_2O which are then detected with the help of Thermal Conductivity Detector

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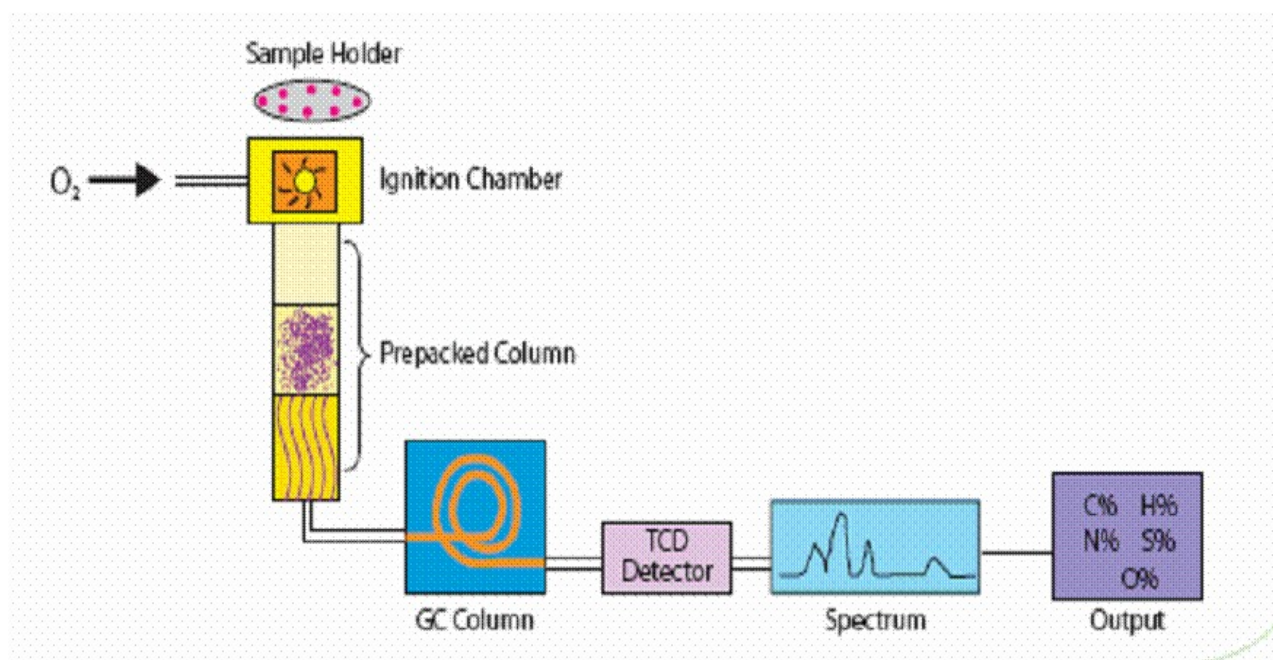
The instrument is calibrated with the analysis of std compounds using the K-factors calculations. Thus the instrument ensures maximum reliability of the results because the combustion gases are not split or diluted but directly carried to build in GC system simultaneous determination of CHNS can be done in less than 10 mins. This method finds greatest utility in finding out percentages of C, H, N, S, (O) in organic compounds which are generally combustible at 1800o C.

Key components of CHNS are auto sampler, combustion reactors ,chromatographic column, and T.C.D. detector.

6) Ash fusion temperature (AFT)

Ash fusion temperatures give an indication of the softening and melting behavior of fuel ash. These temperatures are widely cited in fuel specifications for boilers despite a relatively poor record of correlating with slagging or fouling behavior. Reasons for the poor predictive behavior include:

Fusion temperatures are based on fuel ash whereas deposits commonly are enriched and depleted in several elements relative to the fuel. Fusion temperatures are measured over short time periods while heating ash at a rate of 8 +/- 3 ºC (15 +/- 5 ºF) per minute whereas ash deposits typically accumulate for hours and are formed during cooling relative to the bulk gas temperature. Fusion temperatures do not account for either boiler design or boiler operation, both of which strongly influence slagging and fouling behavior. Fusion behavior changes when samples are allowed to stand at a given temperature. Fusion temperatures generally significantly decrease if the samples equilibrate at a given temperature for an hour or so.

Fusion temperatures at one time were also quite subjectively measured, but this criticism has been addressed by the development of automated techniques for performing the measurements that require no intervention by the operator. Despite the shortcomings, fusion temperatures are valuable guides to the high-temperature behavior of the fuel inorganic material.

Ash fusion temperatures are determined by viewing a molded specimen of the coal ash through an observation window in a high-temperature furnace (ASTM D1857). The ash, in the form of a cone, pyramid, or cube, is heated steadily past 1000 °C (1832°°F) to as high a temperature as possible, preferably 1600°°C (2910°°F).

7) Bomb Calorimeter

The bomb calorimeter is a laboratory instrument used to measure the amount of a sample's combustion heat or heat power when excess oxygen combustion occurs. The modern Bomb calorimeter is a development of the original calorimeter of Berthelot. The modern bomb calorimeter is made of corrosion resisting steel in which the combination Bomb Calorimeter.

The gross calorific value can be used to compute the total calorific content of the quantity of coal or coke represented by the sample for cost effectiveness purposes.

The gross calorific value can be used for computing the calorific value versus sulfur content to determine whether the coal meets regulatory requirements for industrial fuels.

The gross calorific value can be used to evaluate the effectiveness of beneficiation processes

The principle of a bomb calorimeter is to measure the heat released during the combustion of a sample. The sample is placed inside a crucible and ignited in an oxygen atmosphere at high pressure. The heat released during the combustion is absorbed by the water in the water jacket surrounding the bomb, and the temperature change of the water is used to calculate the heat of combustion of the sample.

Bomb calorimeters are commonly used in the field of energy research to measure the heat of combustion of various fuels. This information can be used to calculate the energy content of the fuel, which is important in determining the efficiency of energy production and usage

.

6200 Bomb Calorimeter PARR

8) Dilatometer

Coal Dilatometer is a fully automatic system for determining the swelling properties of hard coal when heated under standard conditions: dilatation and contraction are obtained by inserting a sample of powdered coal, formed under pressure, in a narrow tube topped by a piston and reading the displacement of the piston as a function of the temperature.

Dilatometer test is used to determine the coking properties of hard coal or hard coal blends on the laboratory scale. It is used to indicate the pressures exerted by hard coals on the walls of industrial carbonization ovens.

A pencil made of powered coal formed under pressure is inserted into a precisely calibrated narrow tube and topped by a calibrated steel rod (piston) which slides in the bore of the tube. The whole is heated at a constant and definite rate heat the coal at a rate of 3 'C per minute. By

making regular readings of the displacement of the piston as a function of the temperature and expressing the displacements observed as percentages of the original length of the pencil.
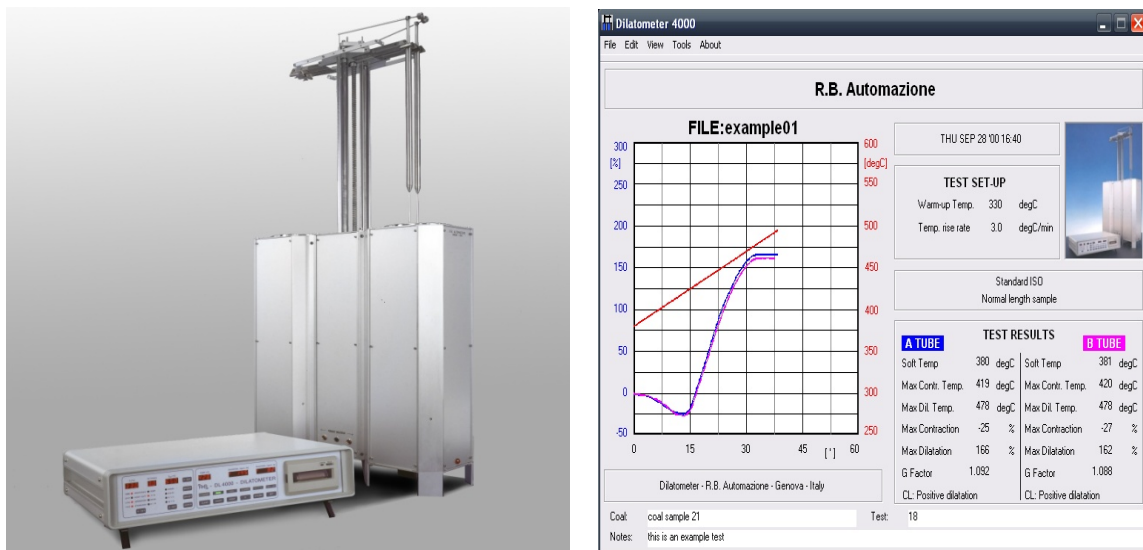
DL 4000 Dilatometer

9) Float and sink Analysis

Float-sink tests are done on samples of coal from beds that will likely need washing (beneficiation, preparation) to remove sulfur or ash (rock and mineral matter) from the coal to meet the desired specifications of the end user. The float-sink analysis determines how much coal can be separated from rock and minerals in fluids of different densities.

Washability of coal means its amenability to improvement in quality by beneficiation techniques. Float & sink tests are based on difference in specific gravity and shale (other impurities) of coal. Specific gravity of clean coal in its purest form (free from Shale, clay, sandstone etc.) is about 1.22 (1.12 to 1.35) while specific gravity of impurities varies from 1.60 to 2.65 (when no pyrite is present as gangue). When coal containing impurities is immersed in the solution whose specific gravity is intermediate between those of constituents, then the two constituents are separated. Clean coal having lower specific gravity will float while impurities having higher specific gravity will sink. Coal plies and particles have different relative densities, determined by vitrinite content, rank, ash value/mineral content and porosity. Coal is usually washed by passing it over a bath of liquid of known density. This removes high-ash value particles and increases the saleability of the coal as well as its energy content per unit volume. Thus, coals must be subjected to a float-sink test in the laboratory, which will determine the optimum particle size for washing, the density of the wash liquid required to remove the maximum ash value with the minimum work. Float-sink testing is achieved on crushed and pulverized coal in a process similar to metallurgical testing.

Float and Sink

10) Gieseler Fluidity Test

Coking coals can, when heated in the absence of air, to soften, swell, and then re-solidify to form a coherent, porous, hard coke structure. The Gieseler Plastometer test is used to evaluate the plastic properties of a coal or coal blend.
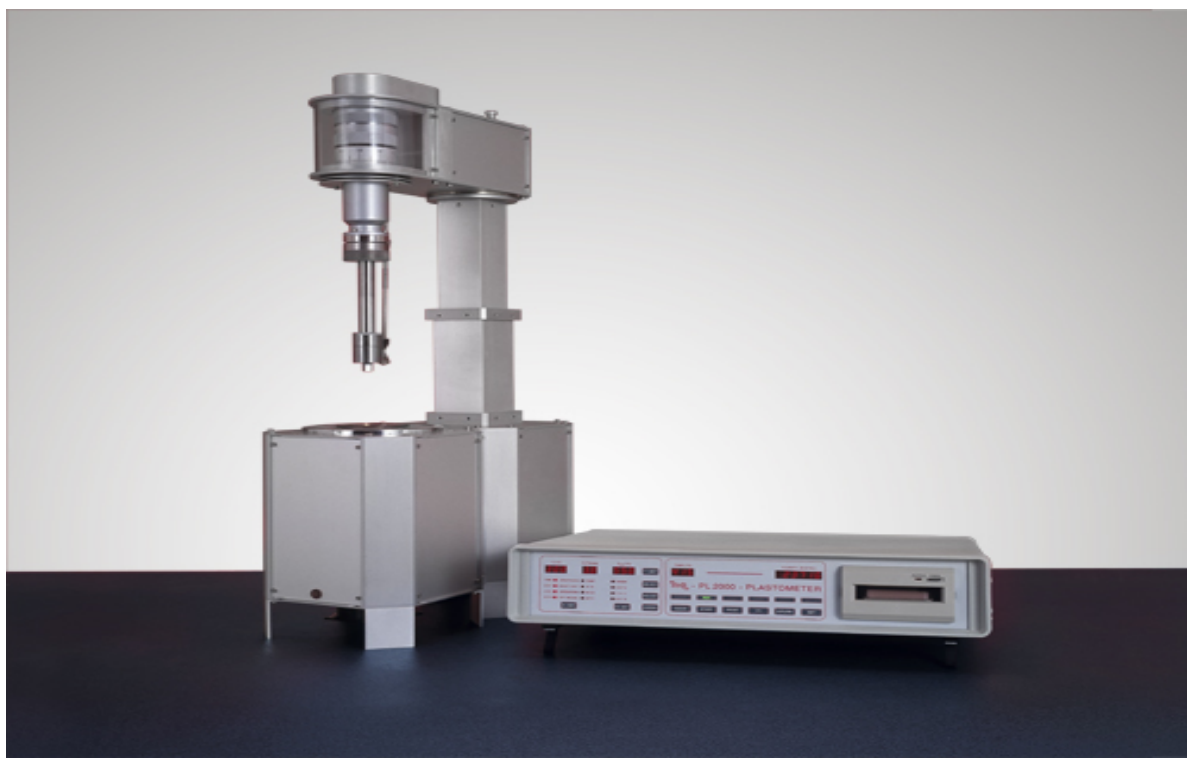
A

Plastometer

Gieseler Plastometric Properties

- Gieseler plastic properties depend on maceral composition, rank, ash content, level of oxidation, heating rate, etc.
- Charge coals having MF in the range of 200 to 1000 ddpm considered most suitable for coke making.
- Larger plastic range improves compatibility & coke strength.
- Oxidation of coal reduces its fluidity.
- Log (MF) is considered very close to additive property.

Do's	Don'ts
1. Always switch on the power first. 2. Press the start button of the control unit in such manner that the stirrer of solder bath should not twirl. 3. Always use dust mask for sample preparation. 4. Always load the sample when 330°C has reached. 5. Always cover the solder bath with the lid while heating and loading the sample. 6. Press the start button once again to start the test. 7. Always use thermal insulating gloves for removal of crucible from the Plastometer after completing the test. 8. Always switch on the exhaust fan while operating Plastometer.	1. Don't touch the crucible unless the test is over. 2. Don't touch the setup for sometime after the automatic lift up of crucible for cooling. 3. Never touch the top of the furnace with naked hand. 4. Never try to smell the exit gas of sample.Never touch the hot solder by naked hand.

Safety Equipment

- Safety glasses, gloves
- Fume hood
- Respiratory mask
- Ear plugs

Precautions

1. Wear safety glasses at all times.
2. When using blaster secure door before operating.
3. Disconnect compressed air if maintenance is to be done.
4. Wear long-sleeved laboratory coat while working the Geiseler Plastometer unit, to protect against splashing with hot solder. Thermocouple lid must cover solder bath at all times.

Notes

- Once the sample has been prepared the test must be carried out.
- It is advised that drying should not be excessive because the plastic properties of coal are altered by oxidation. No sun or oven drying of sample.
- Unsuccessful packing of the sample is signified by immediate rotation of the stirrer. In such cases it is allowable to use 1 to 3 drops of toluene on the shaft of the stirrer near the surface of the coal before the mass is added.
- Controllers must be turned back to reset after test is completed.
- Record details of sample etc in Gieseler Log Book.
- Check Calibration Board at the commencement of each shift to determine whether stirrers/crucibles are due for their fortnightly calibration check.

PROCEDURE

Preparing sample for analysis

- Sieve sample using 1.18 mm screen.
- The plus 1.18 mm material is put through coffee grinder on coarse, medium and fine settings.
- Sieve after each setting.
- Spread on mixing mat and increment divide using around twenty scoops of sample to make up 50 g of sample.
- Bag remaining sample as "RESERVE". Label with Job Reference Number, sample details and date. Place in freezer for two week. Samples are then recorded in drum storage record, placed in appropriate drum and held for total of three months.
- Using the 425 um screen sieve remaining 50 g. Run hand lightly over screen.
- Set coffee grinder to Setting No. 1 (see grinder). Grind +425 um through grinder and sieve.
- Again take +42Sum and grind at Setting No. 2 and sieve.
- Again take +425um and grind at Setting No. 3 and sieve. Repeat this step.
- If any +425um remains after repeating Step 10 grind remaining with mortar and sample. (Refer I&T/MA-TM-01). pestle. Mat mix prepared 425um.

- Put prepared sample of minus 425 micron into an empty, clean bottle. Label Job Reference Number, sample details and date.

Packing of the sample in the crucible

- First mount the stirring rod in the crucible. The stirring rod should be set in the small depression of the crucible bottom.
- Make sure all other thumbscrews are loosened. This applies downward force to the stirring rod to keep it in position.
- Introduce the sample, (a total of 5 grams), into the crucible and rotate the stirring rod to allow the sample to fill the spaces below the stirring arms. DO NOT allow the stirring rod to be lifted out of the small hole in the center of the crucible bottom.
- Continue to add sample until all 5 grams are introduced into the crucible.
- Move the piston of the packer down so that it slightly compresses the sample. Raise the packer hammer and lower the packer weight. Drop the hammer twelve times per the ASTM standard.
- After completely assembling and filling the crucible and stirring rod combination, be particularly careful not to move the rod.
- Insert the rod into the barrel with the chimney attached and crew the barrel onto the crucible. Holding the sample by the barrel, carefully fit the stirring shaft into the drive shaft on the head assembly and tighten the collar.
- Take extra care to insure that the stirring shaft does not move during assembly. (Tightening the threaded connections more than hand-tight should not be necessary if the parts are properly cleaned).

Running A Plasticity Test

- Turn the system power on. Allow the instrument to boot-up the main operating screen. If enabled the furnaces will automatically heat up to the warming set-point temperature.
- Prepare the sample per the sample preparation, loading and packing instructions.
- Enter desired Sample Tag by pressing the shaded box next the Sample Tag indication.
- Once proper furnace temperature is reached, connect the retort assembly (Barrel, Chimney Crucible and Stirring Rod) to the Head Assembly. Press the "START" button on the Plastometer side of the screen. Lower the sample into the molten solder bath and acknowledge by pressing the "OK" button on the screen. The Plastometer side is known in run mode and the screen will automatically change to the Plastometer plot screen. The cooler sample assembly will cause the solder bath to drop in temperature. The controller will heat the furnace up until the "start" temperature is once again reached. At the "start" temperature the controller turns the head assembly on and ramps the furnace temperature at the selected rate of rise.
- Once the "stop" temperature is reached the system automatically goes into an alarm condition letting the operator know that the test is complete. Press the "Ann. Off" button to silence the alarm.

- Raise the sample assembly from the furnace.
- The test is now complete and the system is ready for another test run. The next test cannot be started until the furnace temperature decreases to the "start" temperature.

Removal of apparatus

- Once printer shows no further dial movement turn printer off.
- Turn brake off.
- Switch controller back to reset.
- Lift apparatus out of solder bath.
- Scrape down sides of crucible with spatula.
- Unscrew barrel from plastometer head using gloves and multi-grips.
- Place on tray, brush solder off apparatus.
- Disassemble crucible from barrel. Let cool.

Cleaning of apparatus

- When cool, place guide sleeve and breather tube into acetone.
- Barrel cleaned with circular file, scrape any bulk material off crucible and stirrers. Clean with abrasive blasting unit. Clean off any dust etc. with compressed air. (Do not over clean as this alters dimensions of equipment.)
- Re-assemble.

Reporting of results on printout

- Initial softening temperature (°C) and Fluidity (Highlighted, at 1 dd/min).
- Maximum fluidity temperature (°C) and Fluidity (Highlighted).
- Solidification temperature (°C) and Fluidity(Highlighted, at 0 dd/min).
- Label with date, operator initials, Job Reference Number, sample details, pot number, crucible/stirrer unit number

Significance

Plastometer test is used to determine the quality of coke. If the dial divisions per minute (DDPM) is high mean the coke is of good quality.

Test Results :-

Coal Sample :-   AUS (HARD)

Gieseler Fluidity	Actual	12-8 mm	8-4 mm	4-0mm
Initial softening temp. C, (1ddpm)	412	417	408	408
Softening Temp at 5 ddpm	428	433	430	425
Maximum fluidity temp., C	456	458	455	456
Resol. Temp at 5 ddpm	482	476	479	483
Final Fluid Temp. C,(1ddpm)	486	480	484	487
Plastic Range, C (1ddpm)	74	63	76	79
Solidification Temperature	494	490	492	492
Plastic Range	82	73	84	84
Maxi. Fluidity, ddpm	395	130	221	588

Table - 1 Test Report of AUSTRALIA (HARD) Coal

Consummation :-

In summary, the data collected throughout this assessment suggests that the fluidity for the sample drawn from AUSTRALIA (HARD) Coal increases with the decrement in the size of the coal particles and increased Plastic range.

```
file: Untitled.pla                    Warm-up Temp.:        330 °C
date:  WED MAY 29 '24 16:27:50        Temp.Rise Rate:      3 °C/min

Coal:  AUS (H) AUCTUAL
Test:  COAL
Notes: 29-05-2024

Soft Temp:       412 °C               Absolute Data:
                                      Max Fluid. Temp:     455 °C
Fin Temp:        487 °C               Max Fluidity:        395 DDPM
                                      Log Max Fluidity:    2.6
Sol Temp:        490 °C               Integrated Data:
                                      Max Fluid. Temp:     457 °C
Sol-Soft T.:      78 °C               Max Fluidity:        370 DDPM
                                      Log Max Fluidity:    2.57
Fluid. Area:    2761 DD
```
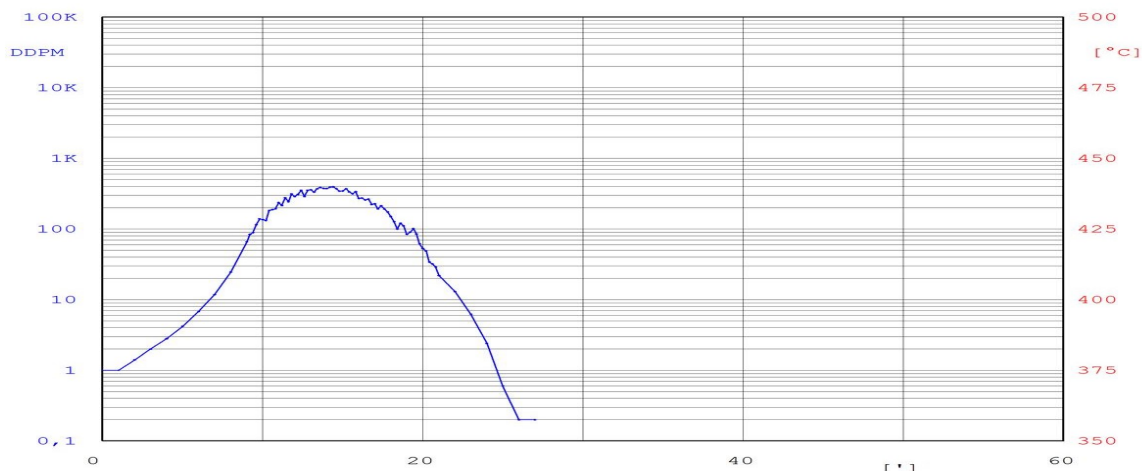

graph 1.1 Characteristics graph of AUS (H) size- Actual

```
file: AUS(H)(8-12mm)31-05.pla         Warm-up Temp.:        330 °C
date:  FRI MAY 31 '24 12:58:00        Temp.Rise Rate:      3 °C/min

Coal:  AUS(H)(8-12mm)
Test:  coal
Notes: 31-05-24

Soft Temp:       417 °C               Absolute Data:
                                      Max Fluid. Temp:     457 °C
Fin Temp:        482 °C               Max Fluidity:        130 DDPM
                                      Log Max Fluidity:    2.11
Sol Temp:        485 °C               Integrated Data:
                                      Max Fluid. Temp:     459 °C
Sol-Soft T.:      68 °C               Max Fluidity:        107 DDPM
                                      Log Max Fluidity:    2.03
Fluid. Area:     732 DD
```


graph 1.2 Characteristics graph of AUS (H) size- 12-8 mm

```
file: AUS(H)(4-8mm)30-05.pla          Warm-up Temp.:        330 °C
date:  THU MAY 30 '24 16:04:46        Temp.Rise Rate:       3 °C/min

Coal:  AUS(H)(4-8)
Test:  COAL
Notes: 30-05-2024

Soft Temp:      408 °C                 Absolute Data:
                                       Max Fluid. Temp:     454 °C
Fin Temp:       486 °C                 Max Fluidity:        221 DDPM
                                       Log Max Fluidity:    2.34
Sol Temp:       489 °C                 Integrated Data:
                                       Max Fluid. Temp:     456 °C
Sol-Soft T.:     81 °C                 Max Fluidity:        187 DDPM
                                       Log Max Fluidity:    2.27
Fluid. Area:    1324 DD
```
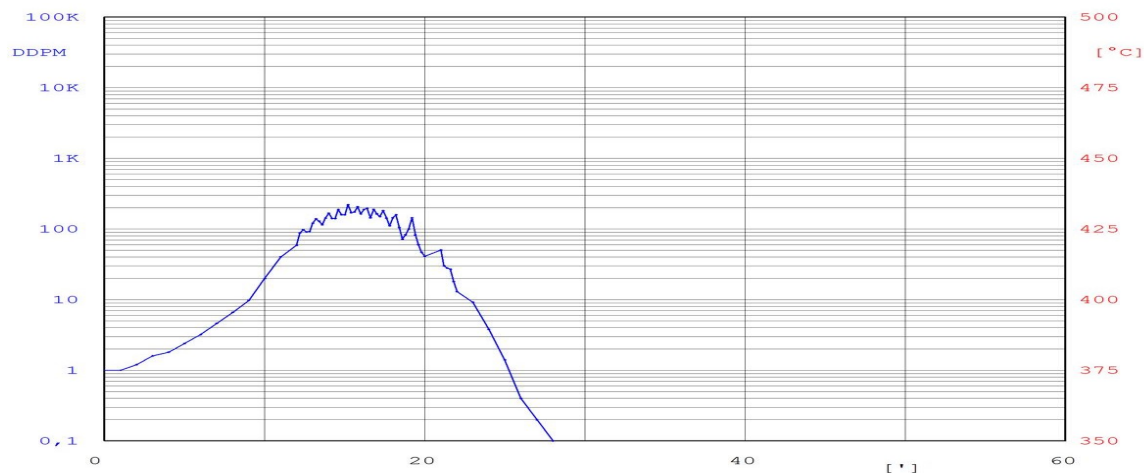

graph 1.3 Characteristics graph of AUS (H) size- 8-4 mm

```
file: Aus(H)(0-4mm)30-05-24.pla       Warm-up Temp.:        330 °C
date:  THU MAY 30 '24 12:11:20        Temp.Rise Rate:       3 °C/min

Coal:  AUS(H)(0-4mm)
Test:  coal
Notes: 30-05-24

Soft Temp:      408 °C                 Absolute Data:
                                       Max Fluid. Temp:     454 °C
Fin Temp:       489 °C                 Max Fluidity:        588 DDPM
                                       Log Max Fluidity:    2.77
Sol Temp:       492 °C                 Integrated Data:
                                       Max Fluid. Temp:     457 °C
Sol-Soft T.:     84 °C                 Max Fluidity:        519 DDPM
                                       Log Max Fluidity:    2.72
Fluid. Area:    4048 DD
```
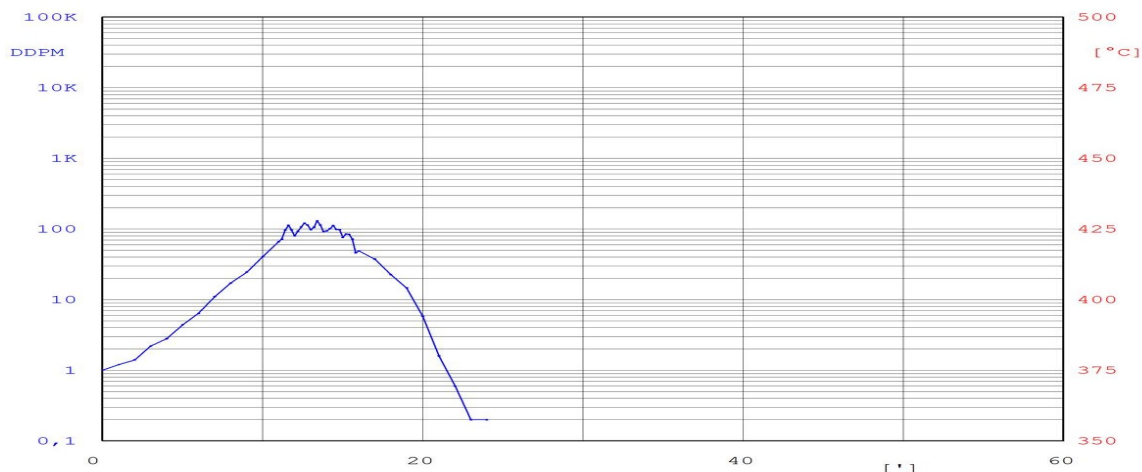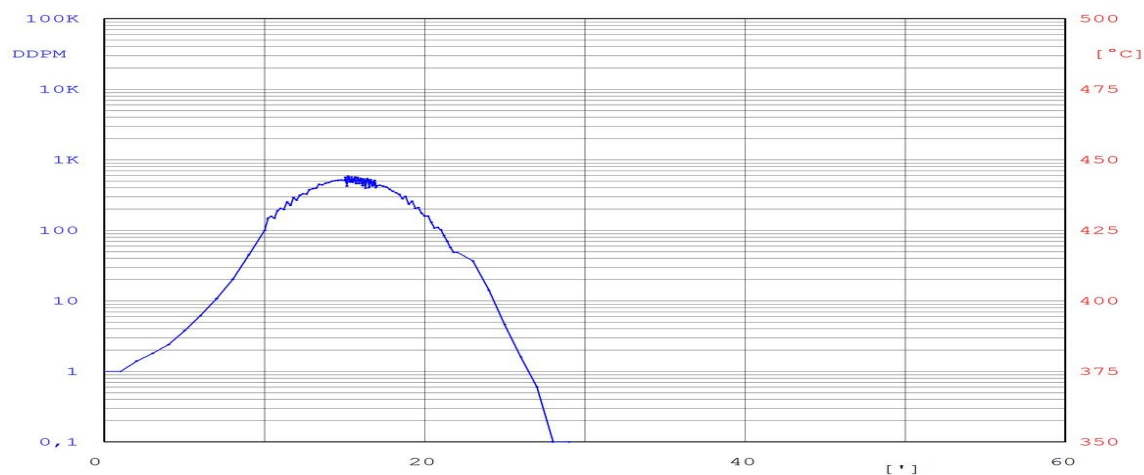

graph 1.4 Characteristics graph of AUS (H) size- 4-0 mm

Coal Sample :-   KESTRAL (SOFT)

Gieseler Fluidity	Actual	12-8 mm	8-4 mm	4-0mm
Initial softening temp. C, (1ddpm)	403	397	399	409
Softening Temp at 5 ddpm	417	410	413	425
Maximum fluidity temp., C	437	437	439	441
Resol. Temp at 5 ddpm	458	462	461	456
Final Fluid Temp. C,(1ddpm)	465	468	466	462
Plastic Range, C (1ddpm)	62	71	67	53
Solidification Temperature	470	473	474	469
Plastic Range	67	76	75	60
Maxi. Fluidity, ddpm	235	1452	990	32

Table - 2 Test Report of KESTRAL (SOFT) Coal

Consummation :-

In summary, the data collected throughout this assessment suggests that the fluidity for the sample drawn from KESTRAL (SOFT) Coal Decreases with the decrement in the size of the coal particles and reduction in Plastic range is observed

```
file: KESTRAL(S)(ACTUAL new).pla        Warm-up Temp.:          330 °C
date:   THU JUN 06 '24 14:03:11         Temp.Rise Rate:         3 °C/min

Coal:   Kestral(S)-Actual
Test:   Coal
Notes:  06-06-2024

Soft Temp:       403 °C                  Absolute Data:
                                         Max Fluid. Temp:       437 °C
Fin Temp:        466 °C                  Max Fluidity:          235 DDPM
                                         Log Max Fluidity:      2.37
Sol Temp:        468 °C                  Integrated Data:
                                         Max Fluid. Temp:       439 °C
Sol-Soft T.:      65 °C                  Max Fluidity:          217 DDPM
                                         Log Max Fluidity:      2.34
Fluid. Area:    1247 DD
```
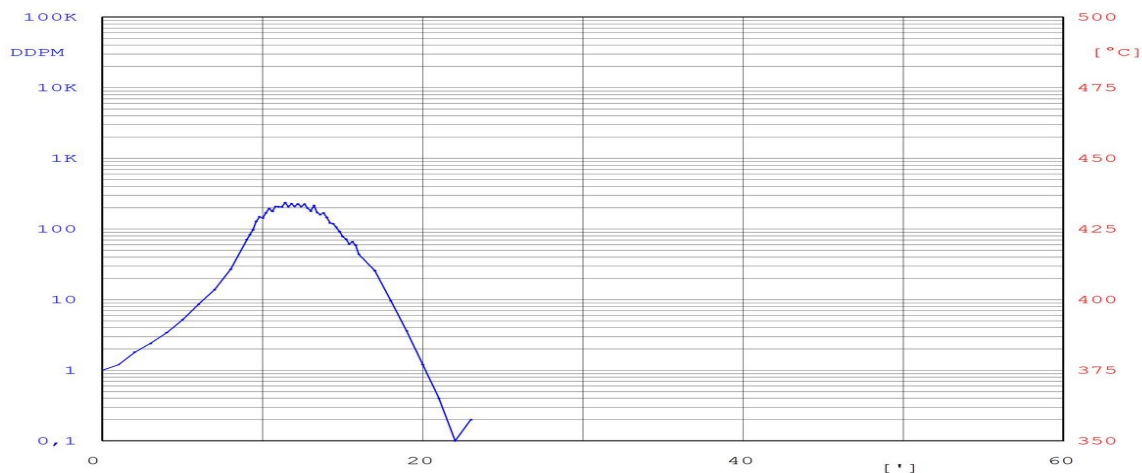

graph 2.1 Characteristics graph of KES (S) size- Actual

```
file: KESTRAL(S)(8-12 NEW).pla          Warm-up Temp.:          330 °C
date:   THU JUN 06 '24 16:20:43         Temp.Rise Rate:         3 °C/min

Coal:   kestral(s)(12-8mm new)
Test:   COAL
Notes:  06-06-24

Soft Temp:       397 °C                  Absolute Data:
                                         Max Fluid. Temp:       437 °C
Fin Temp:        469 °C                  Max Fluidity:          1452 DDPM
                                         Log Max Fluidity:      3.16
Sol Temp:        469 °C                  Integrated Data:
                                         Max Fluid. Temp:       439 °C
Sol-Soft T.:      72 °C                  Max Fluidity:          1285 DDPM
                                         Log Max Fluidity:      3.11
Fluid. Area:    7023 DD
```
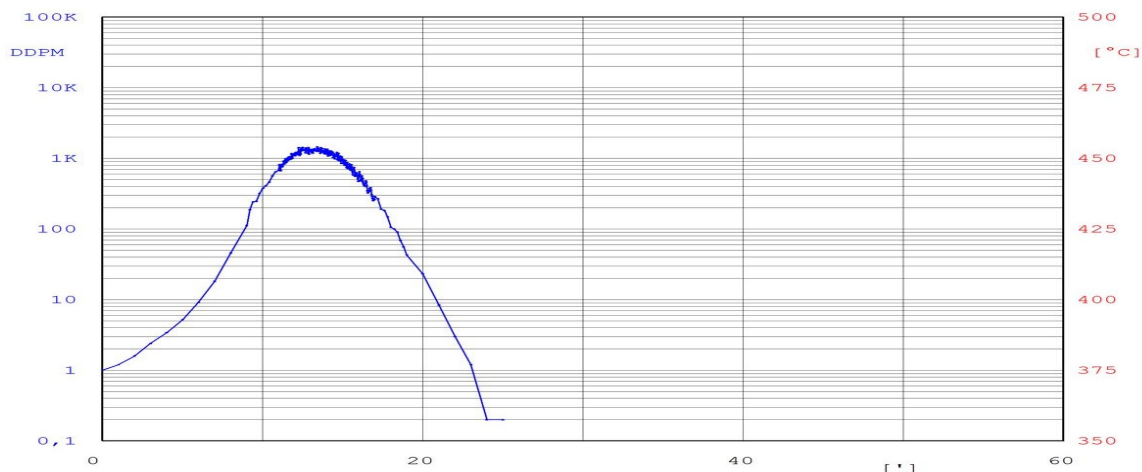

graph 2.2 Characteristics graph of KES (S) size- 12-8 mm

```
file: KESTRAL(S)(4-8 new).pla          Warm-up Temp.:        330 °C
date:  MON JUN 10 '24 13:38:20         Temp.Rise Rate:        3 °C/min

Coal:  kestral(4-8 new)
Test:  COAL
Notes: 10-06-24

Soft Temp:       399 °C                Absolute Data:
                                       Max Fluid. Temp:      439 °C
Fin Temp:        468 °C                Max Fluidity:         990 DDPM
                                       Log Max Fluidity:     3
Sol Temp:        471 °C                Integrated Data:
                                       Max Fluid. Temp:      438 °C
Sol-Soft T.:      72 °C                Max Fluidity:         845 DDPM
                                       Log Max Fluidity:     2.93
Fluid. Area:     4515 DD
```
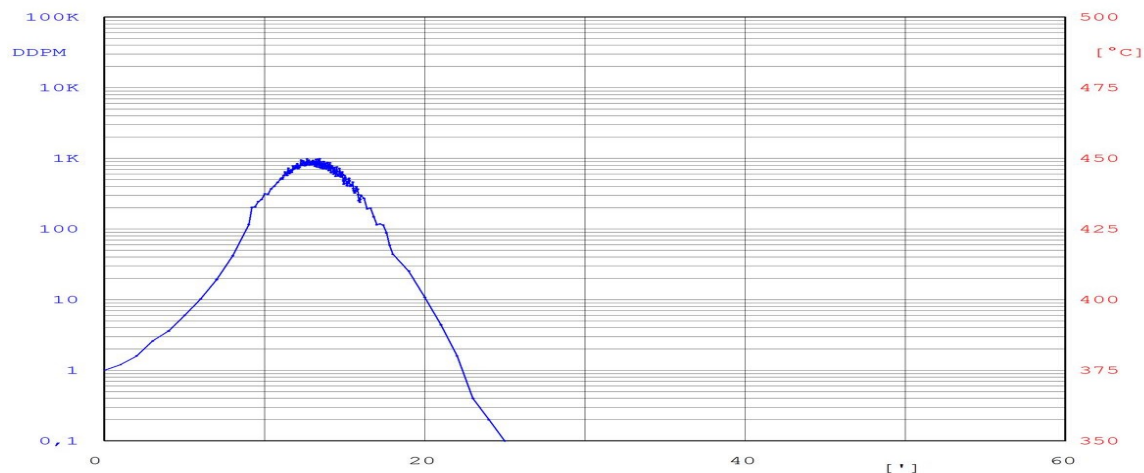

graph 2.3 Characteristics graph of KES (S) size- 8-4 mm

```
file: KESTRAL(S)(0-4NEW).pla           Warm-up Temp.:        330 °C
date:  MON JUN 10 '24 16:03:45         Temp.Rise Rate:        3 °C/min

Coal:  kestral(s)(0-4new)
Test:  COAL
Notes: 10-06-24

Soft Temp:       409 °C                Absolute Data:
                                       Max Fluid. Temp:      441 °C
Fin Temp:        462 °C                Max Fluidity:         32 DDPM
                                       Log Max Fluidity:     1.5
Sol Temp:        465 °C                Integrated Data:
                                       Max Fluid. Temp:      441 °C
Sol-Soft T.:      56 °C                Max Fluidity:         32 DDPM
                                       Log Max Fluidity:     1.5
Fluid. Area:      188 DD
```
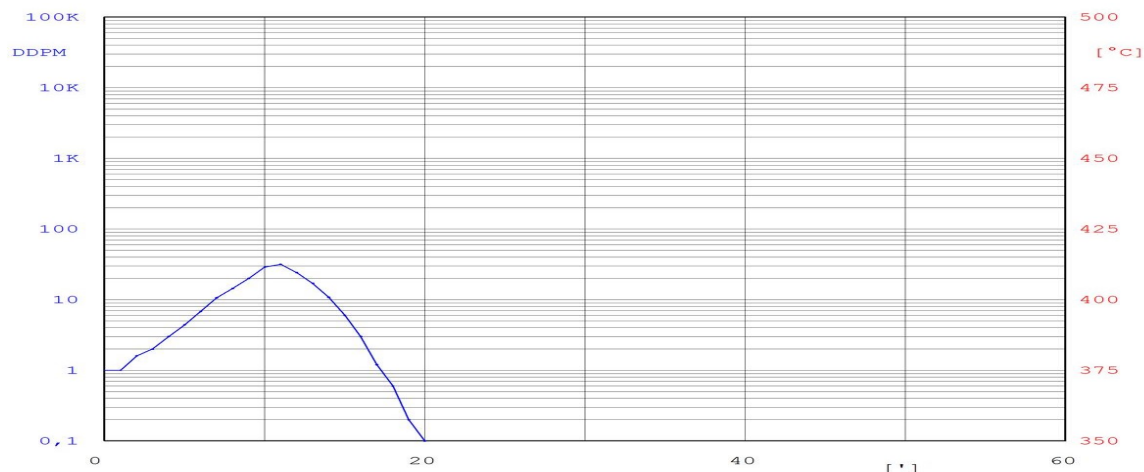

graph 2.4 Characteristics graph of KES (S) size- 4-0 mm

Coal Sample :-   INDIGENOUS (PCC)

Gieseler Fluidity	Actual	12-8 mm	8-4 mm	4-0 mm
Initial softening temp. C, (1ddpm)	434	427	425	428
Softening Temp at 5 ddpm	443	441	449	441
Maximum fluidity temp., C	458	463	452	462
Resol. Temp at 5 ddpm	475	473	460	481
Final Fluid Temp. C,(1ddpm)	482	481	474	487
Plastic Range, C (1ddpm)	48	54	49	59
Solidification Temperature	492	490	486	496
Plastic Range	58	63	61	68
Maxi. Fluidity, ddpm	21	14	6	41

Table - 3 Test Report of INDIGENOUS (PCC) Coal

Consummation :-

In summary, the data collected throughout this assessment suggests that the fluidity for the sample drawn from INDIGENOUS PCC Coal increases with the decrement in the size of the coal particles and increased Plastic range. The analysis conducted in this test also highlights that INDIGINEOUS PCC Coal have critically low amount.

```
file: PCC(actual).pla                    Warm-up Temp.:        330 °C
date:  MON JUN 03 '24 16:02:54           Temp.Rise Rate:       3 °C/min

Coal:  PCC(actual)
Test:  Coal
Notes: 1-06-24

Soft Temp:      434 °C                    Absolute Data:
                                          Max Fluid. Temp:      458 °C
Fin Temp:       485 °C                    Max Fluidity:         21 DDPM
                                          Log Max Fluidity:     1.32
Sol Temp:       488 °C                    Integrated Data:
                                          Max Fluid. Temp:      458 °C
Sol-Soft T.:     54 °C                    Max Fluidity:         21 DDPM
                                          Log Max Fluidity:     1.32
Fluid. Area:    155 DD
```
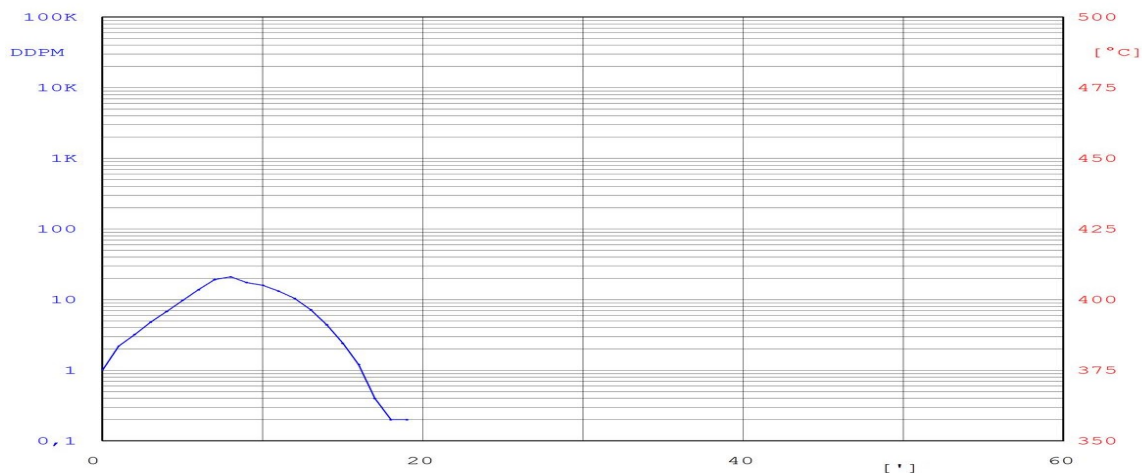

graph 3.1 Characteristics graph of IND (PCC) size- Actual

```
file: PCC(12-8 mm).pla                    Warm-up Temp.:        330 °C
date:  TUE JUN 04 '24 13:28:26           Temp.Rise Rate:       3 °C/min

Coal:  PCC(12-8mm)
Test:  COAL
Notes: 4-06-24

Soft Temp:      427 °C                    Absolute Data:
                                          Max Fluid. Temp:      463 °C
Fin Temp:       481 °C                    Max Fluidity:         14 DDPM
                                          Log Max Fluidity:     1.13
Sol Temp:       486 °C                    Integrated Data:
                                          Max Fluid. Temp:      463 °C
Sol-Soft T.:     59 °C                    Max Fluidity:         14 DDPM
                                          Log Max Fluidity:     1.13
Fluid. Area:    128 DD
```
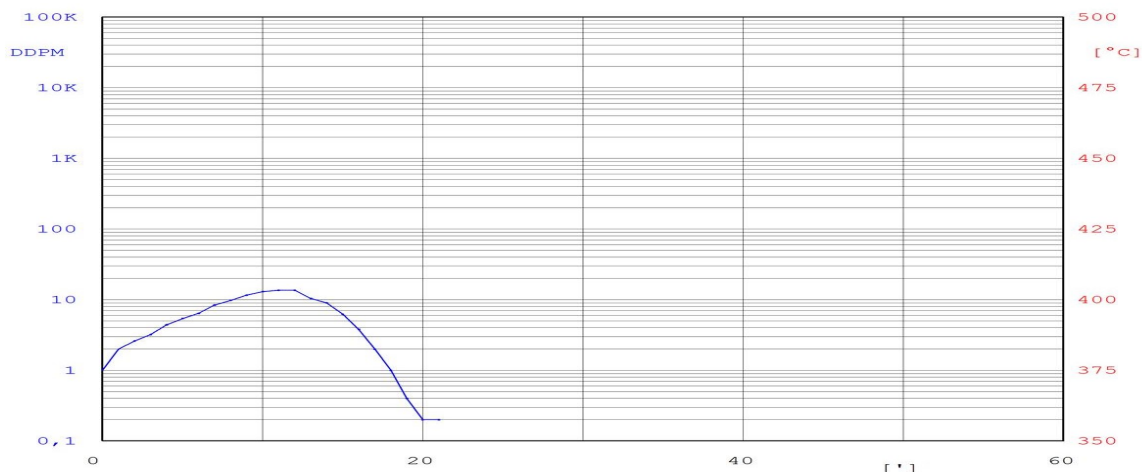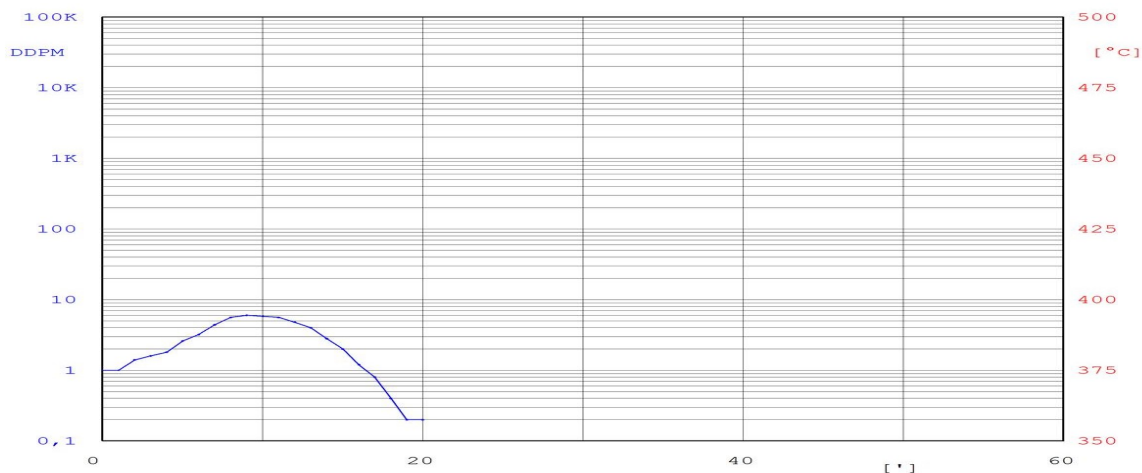

graph 3.2 Characteristics graph of IND (PCC) size- 12-8 mm

```
file:  PCC(4-8 mm).pla              Warm-up Temp.:        330 °C
date:  TUE JUN 04 '24 15:50:10      Temp.Rise Rate:       3 °C/min

Coal:  PCC(4-8mm)
Test:  COAL
Notes: 4-06-24

Soft Temp:       425 °C             Absolute Data:
                                    Max Fluid. Temp:      452 °C
Fin Temp:        477 °C             Max Fluidity:         6 DDPM
                                    Log Max Fluidity:     0.78
Sol Temp:        482 °C             Integrated Data:
                                    Max Fluid. Temp:      452 °C
Sol-Soft T.:      57 °C             Max Fluidity:         6 DDPM
                                    Log Max Fluidity:     0.78
Fluid. Area:      56 DD
```


graph 3.3 Characteristics graph of IND (PCC) size- 8-4 mm

```
file:  PCC(0-4 mm).pla              Warm-up Temp.:        330 °C
date:  WED JUN 05 '24 14:15:50      Temp.Rise Rate:       3 °C/min

Coal:  PCC(0-4mm)
Test:  coal
Notes: 05-06-24

Soft Temp:       428 °C             Absolute Data:
                                    Max Fluid. Temp:      462 °C
Fin Temp:        488 °C             Max Fluidity:         41 DDPM
                                    Log Max Fluidity:     1.61
Sol Temp:        491 °C             Integrated Data:
                                    Max Fluid. Temp:      462 °C
Sol-Soft T.:      63 °C             Max Fluidity:         41 DDPM
                                    Log Max Fluidity:     1.61
Fluid. Area:     319 DD
```
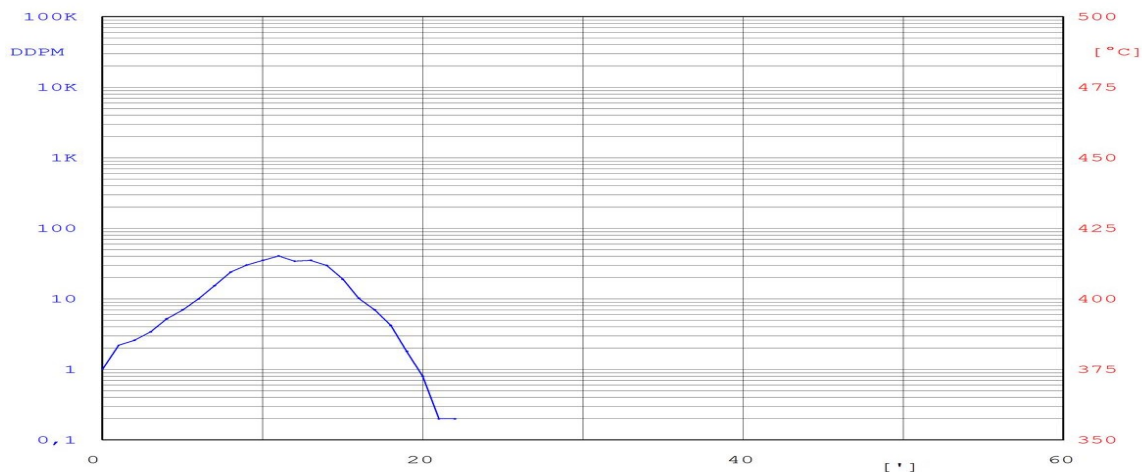

graph 3.4 Characteristics graph of IND (PCC) size- 4-0 mm

Conclusion:-

The vocational training comprising general awareness on the characterization of coking coals, hands on training on different testing of coking coal samples and a project work. The training has provided some basic understanding of coking coal and their characterization. The hands on training have helped to acquire some skill in testing some basic properties of coking coal.

Different physical and chemical properties were determined. The tests included are Proximate Analysis, LTGK coke type, CSN, Fluidity and Petrography.

Especially the study of the plasticity and fluidity property of coal with the variation of size was the major project which was performed and the results of the test are shown above comprising the fluidity and characteristics behavior graph.