

AN INTERNSHIP REPORT ON
PROXIMATE ANALYSIS OF COAL AND
ANALYTICAL INSTRUMENTAL TECHNIQUES

Submitted to:

CSIR- NATIONAL METALLURGICAL LABORATORY
MADRAS CENTRE
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CERTIFICATE

This is to certify that **Mr. HARISH J V** (Roll No: 42910007) pursuing his M.Sc., Chemistry at Department of Chemistry, Sathyabama Institute of Science and Technology, Chennai has successfully completed his Internship programme in our organization from 22.09.2023 to 04.10.2023 under the guidance of Dr. Mousumi Gharai, Scientist.

I wish success in all his future endeavours.

Chennai

04.10.2023

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CSIR-National Metallurgical Laboratory (CSIR-NML) is a premier Indian research organisation dedicated to various facets of Minerals, Metals and Materials science, technology, industrial services and human resource development. Since inception, CSIR- NML has diversified its research areas ranging from extractive metallurgy, alloy development and import substitution, refractory material development, corrosion studies, mathematical and physical modeling of metallurgical processes, mineral research, advanced materials and materials tailoring, integrity evaluation of critical industrial components, surface engineering and cleaner and sustainable metals production. The Laboratory has made notable contributions in the areas of mineral beneficiation and agglomeration, ferrous and nonferrous metallurgy, alloy development and processing, materials science & engineering and, resource conservation & environment. CSIR-NML is also carrying out major activities for creating awareness among the common masses on issues relating to health, environment, rural technology and sustainable development. The Laboratory has kept pace with changing research scenarios and needs of the country. In the last few years, greater emphasis is given to industry sponsored research and, alignment with government program; namely, Make in India, Innovate in India, Strategic sector needs, Swatch Bharat, Societal and skill India, etc. The activities of the Laboratory touch upon several major sectors relevant to the growth of India, including iron and steel, power and energy, oil and gas, automotive, railways, strategic, societal and others.

About NML- Madras



CSIR National Metallurgical Laboratory (NML) is a premier laboratory within the ambit of Council of Scientific and Industrial Research (CSIR), New Delhi, a statutory body under Government of India. CSIR National Metallurgical Laboratory Madras Centre (NML-MC) an extension Centre of CSIR NML, over the years has undertaken several R&D projects in the areas of mineral beneficiation, micro-structural characterization and failure analysis of various industrial components. The Centre has developed flow sheets for the beneficiation of ferrous and non-ferrous, industrial minerals which include limestone, feldspar, barites etc. In recent years, the Centre has made significant contributions to the development of column flotation technology for Indian mineral industry. Commercial flotation columns were commissioned for the beneficiation of sillimanite and barites. The principle R& D's of the department includes

- Process development in mineral beneficiation (barites, beach sand minerals, iron ore, limestone, graphite & coal)
- Investigations on failure analysis of metallurgical components, root cause analysis and remedial measures

- Development of value added products from mineral/mines wastes/rejects.

The Centre is actively working towards developing value added products from mineral/mine wastes and also provides services for characterization of tiles, paver blocks and natural building stones. The esteemed notable clients of the department includes Kerala Mines and Minerals Ltd., English India Clay Ltd., RAMCO Cements Ltd., TATA Steel, JSW, Hinduja Foundries, NTPC , Hinustan Zinc Ltd. Indian Rare Earths Ltd and etc.

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COAL

1.1. INTRODUCTION

1.1.1. COAL

One of the most abundant and widely distributed fossil fuel is coal. Coal is a carbon rich material that occurs in the stratified sedimentary deposits. Coal is a heterogeneous material composed of organic and inorganic substances. It is formed from vegetation, that was consolidated between rock strata and converted by the combined effects of pressure and heat over a million of year to form coal. However in recent years, due to the volatility in oil and increase in natural gas cost; coal has gained more interest and is been used as the primary source of raw material for chemical industry.

It is commercially mined in more than 50 countries. On an average the annual world coal consumption is about 5800 million tons of which about 75% is used for electricity production. The significance uses of coal includes steel production, cements manufacture and as a liquid fuel. The Carboniferous period marks the period of formation of coal which is also known as the first coal age. But the major coal deposits were formed 270-350 million years ago (i.e. Upper Carboniferous period). The geochemical process that transformed plant debris to coal is called coalification and is expressed as follows.

Peat → Lignite → Subbituminous Coal → Bituminous Coal → Anthracite

1.1.2 COMPONENTS OF COAL

The components of coal includes volatile matter is composed of aliphatic carbon atoms or aromatic hydrocarbons and mineral matter. Ash consists of inorganic matter from the earth's crust like limestone, iron, aluminum, clay, silica, and trace elements (concentrations of less than 1000 ppm [$<0.1\%$ of a rock's composition] of zinc, copper, boron, lead, arsenic, cadmium, chromium, selenium)

Pure Coal		Mineral Matter	Total Moisture	
Fixed Carbon	Volatile Matter = Organic + Mineral	Ash	Inherent Moisture	Surface Moisture

Fig.1.1. Composition of coal

1.1.3 TYPES OF COAL

Coal can be classified on basis of geological processes of decomposition of dead biotic material over a period of time as follows-

Lignite: Lignite is a low grade brown coal, which has high moisture content and lowest energy content. Lignite coal is a young coal deposits that were not subjected to extreme heat or pressure. The principal lignite reserves are in Neyveli in Tamil Nadu and are used for generation of electricity.

Subbituminous coal: It has a higher heating value than lignite. Subbituminous coal typically contains 35-45 %carbon, compared to 25-35 % for lignite.

Bituminous Coal: The deep buried coal subjected to increased temperatures is bituminous coal. It is the most popularly utilized coal in commercial use. Metallurgical coal is high grade bituminous coal which has a special value for smelting iron in blast furnaces.

Anthracite: Anthracite is the highest quality hard coal. In India coal occurs in rock series of two main geological ages, namely Gondwana, which ages 200 million years and in tertiary deposits which are only about 55 million years old.

1.1.4 GRADING OF COAL

According to the Indian Ministry of Coal, the gradation of non-coking coal is based on Gross Calorific Value (GCV), the gradation of coking coal is based on ash content and for semi coking /weakly coking coal it is based on ash and moisture content(Source: Ministry Of Coal, Government of India).

A. Grades of Coking Coal

Table 1.1 Grading of coking coal

GRADE	ASH CONTENT
Steel Grade -I	Not exceeding 15%
Steel Grade- II	15% - 18%
Washery Grade-I	18% - 21%
Washery Grade -II	21% - 24%
Washery Grade -III	24% -28%
Washery Grade –IV	28% - 35%
Washery Grade –V	35% - 42%
Washery Grade –VI	42% - 49%

B. Grades of Semi-coking and Weakly Coking Coal

Table 1.2 Grading of semi coking coal

GRADE	ASH+ MOISTURE
Semi coking grade –I	Not exceeding 19%
Semi coking grade -II	19%-24%

C. Grades of Non-coking Coal

Table 1.3 Grading of non-coking coal

GCV BAND (K.Cal./Kg.)	GRADE
Exceeding 7000	G-1
6700-7000	G-2
6400-6700	G-3
6100-6400	G-4
5800-6100	G-5
5500-5800	G-6
5200-5500	G-7
4900-5200	G-8
4600-4900	G-9
4300-4600	G-10
4000-4300	G-11
3700-4000	G-12
3400-3700	G-13
3100-3400	G-14
2800-3100	G-15
2500-3100	G-16
2200-2500	G-17

1.1.5 APPLICATIONS OF COAL

The uses of coal in various sectors are as follows:

❖ Electricity Generation

Power generation is the primary use for coal worldwide. Here, coal is burnt to create steam that drives turbines and generators for the production of electricity.

❖ Metal Production

Metallurgical (coking) coal is a key ingredient in steelmaking. Coal converted to coke is used to produce around 70% of the world's steel. Coal is also widely used in the production of other metals including aluminium and copper.

❖ Cement Production

Coal is used as a key energy source in cement production. By-products of coal combustion such as fly ash also play an important role in cement manufacture and the wider construction industry.

❖ Chemical And Pharmaceutical Industries

Many chemical products are manufactured from the by-products of coal. Refined coal tar is used to make chemicals such as creosote oil, naphthalene, phenol and benzene.

❖ Other Industries

Other major users of coal include the paper, textile and glass industries. Coal is also used in the manufacture of carbon fibre and specialist ingredients such as silicon metals, which are used to produce ingredients for the household and personal care sectors.

1.1.7 COAL ANALYSIS METHODOLOGIES

The chemical and physical composition of coal can be analyzed according to International and national standards coal analyses guidelines i.e. American Society for Testing and Materials standards for coal analyses (ASTM). The Quality of coal can be determined by two ways

- i) Proximity analysis
- ii) Ultimate analysis

i. Proximity Analysis Of Coal

The proximity analysis gives the amount of gross impurities present in the coal sample . This method is used to determine moisture content, volatile matter, ash content and fixed carbon of coal (J. G. Speight, 2005).

a) Moisture Content

The heating of coal at a temperature slightly above the boiling point of water causes a loss of weight from the sample that is defined as moisture. The moisture content of coal ranges from about 5% to almost 70%. Moisture is an undesirable constituent of coals because it reduces the heating value .The moisture of air-dried, finely powdered coal sample was determined by heating accurately weighed quantity of a sample in a silica crucible at 105⁰ – 110°C for 1 hour in an electric oven. The loss in the weight of sample is found out by the difference in weights. The percentage of moisture was calculated as

$$\text{Percentage of moisture} = \frac{\text{Loss in weight of the sample}}{\text{Weight of air-dried sample}} \times 100$$

b) Volatile Matter

Volatile matter (VM) in coal refers to the thermally decomposed product which liberates upon heating coal at high temperature in the absence of air. The percentage of volatile matter is determined by heating an accurately weighed air-

dried coal sample in a silica crucible covered with lid. The heating is carried out at $925 \pm 5^{\circ}\text{C}$ in a furnace for exactly seven minutes in the absence of air.

$$\text{Percentage of volatile matter} = \frac{\text{Loss in weight of the sample}}{\text{Weight of air-dried sample}} \times 100\% - \text{moisture \%}$$

c) Ash content

Ash is residue obtained after complete combustion of coal, which consists of CaO, MgO, SiO₂, Al₂O₃ and etc. The coal sample whose volatile matter was found was then heated in a furnace at $725 - 750^{\circ}\text{C}$ without lid for 1 hour. Cooled and weighed. Process of ignition, cooling and weighing was repeated till combustion is complete which is indicated by constant weight of the residue.

$$\text{Percentage of Ash} = \frac{\text{Weight of ash(residue)}}{\text{Weight of dried sample}} \times 100$$

d) Fixed Carbon

The Fixed carbon in coal is the amount of carbon that remains in the coal sample after volatile matter is driven off. The fixed carbon in the coal can be calculated as follows

$$\text{Percentage of fixed carbon} = 100 - (\text{moisture\%} + \text{volatile matter\%} + \text{ash\%})$$

Quality of coal depends on Percentage of fixed carbon; it should be as high as possible. Fixed carbon varies inversely with volatile matter. More the percentage of fixed carbon, higher is the calorific value.

1.2 PROCEDURE

1.2.1 DETERMINATION OF MOISTURE

- * Take the given sample Seam VI-(WBC-Head sample).
- * Take the empty weight of the crucible and note the value.
- * The given sample Seam VI-(WBC-Head sample) of 0.5000-0.5050g is taken.
- * Note the value by weighing the sample approximately.
- * And the sample is placed in the oven for 1 hour at 100°C.
- * After heating the sample cool for few minutes and then note the sample weight.

1.2.2 DETERMINATION OF ASH

- *Take the given sample Seam VI- (WBC-Head sample).
- *Take the empty weight of the crucible and note the value.
- * The given sample Seam VI-(WBC-Head sample) of 0.5000-0.5050g is taken.
- *Note the value by weighing the sample approximately.
- *And the sample is placed in the Furnace for 2 1/2 hour at 900°C.
- *After heating the sample cool for few minutes and then note the sample weight.

1.2.3 DETERMINATION OF VOLATILE MATTER

- *Take the given sample Seam VI- (WBC-Head sample).
- *Take the empty weight of the crucible and note the value.
- *The given sample Seam VI- (WBC-Head sample) of 1.000-1.050g is taken.
- *Note the value by weighing the sample approximately.
- *And the sample is placed in the Furnace for 7 minutes at 900°C.
- *After heating the sample cool for few minutes and then note the sample weight.

MOISTURE %

1. $A-B/C \times 100$ $A = 26.0811$
 $A-B = 0.0036$ $B = 26.0775$
 $A-B/C = 0.0071$ $C = 0.5014$
 $A-B/C \times 100 = 0.7179 \%$

2. $A-B/C \times 100$ $A = 25.8513$
 $A-B = 0.0055$ $B = 25.8458$
 $A-B/C = 0.0109$ $C = 0.5012$
 $A-B/C \times 100 = 1.0973 \%$

ASH %

1. $D-C/A \times 100$ $D = 23.4357$
 $D-C = 0.1319$ $C = 23.3038$
 $D-C/A = 0.26123$ $A = 0.5049$
 $D-C/A \times 100 = 26.1239 \%$

2. $D-C/A \times 100$ $D = 25.7352$
 $D-C = 0.133$ $C = 25.6022$
 $D-C/A = 0.2651$ $A = 0.5016$
 $D-C/A \times 100 = 26.5151 \%$

VOLATILE MATTER %

$$1. \frac{[M2 - M3]}{[M2 - M1]} \times 100 - M \quad \begin{array}{l} M1 = 13.4975 \\ M2 = 14.498 \\ M3 = 14.2418 \\ (M) \% = 0.7179 \end{array}$$

$$\frac{[M2 - M3]}{[M2 - M1]} \times 100 - M = 24.8892 \%$$

$$2. \frac{[M2 - M3]}{[M2 - M1]} \times 100 - M \quad \begin{array}{l} M1 = 12.5663 \\ M2 = 13.5683 \\ M3 = 13.3069 \\ (M) \% = 1.0973 \end{array}$$

$$\frac{[M2 - M3]}{[M2 - M1]} \times 100 - M = 24.9905 \%$$

FIXED CARBON %

$$100 - (\text{Ash}\% + \text{Moisture \%} + \text{Volatile matter \%})$$

$$1. 100 - (26.1239 + 0.7179 + 24.8892)$$

$$100 - 51.731 = 48.269$$

$$\text{Fixed carbon} = 48.269\%$$

$$2. 100 - (\text{Ash \%} + \text{Moisture \%} + \text{Volatile matter \%})$$

$$100 - (26.5151 + 1.0973 + 24.9905)$$

$$100 - 52.6029 = 47.3971$$

$$\text{Fixed carbon} = 47.3971\%$$

1.3 RESULTS AND DISCUSSIONS

MOISTURE %

Sample name : Seam VI- (WBC-Head sample)

Empty weight	Sample weight(C)	Total weight before(A)	Total weight after(B)	Moisture % $\frac{A-B}{C} \times 100$
1. 25.5797	0.5014	26.0811	26.0775	0.7179
2. 25.3501	0.5012	25.8513	25.8458	1.0973

ASH %

Empty weight(C)	Sample weight(A)	Total weight before	Total weight after(D)	Ash % $\frac{D-C}{A} \times 100$
1. 23.3038	0.5049	23.8087	23.4357	26.1239
2. 25.6022	0.5016	26.1038	25.7352	26.5151

VOLATILE MATTER %

Empty weight	Sample weight	Total weight before(M2)	Total weight after 7 minutes (M3)	VM % $\frac{[M2-M3]}{[M2-M1]} \times 100$
1. 13.4975	1.0005	14.498	14.2418	24.8892
2. 12.5663	1.0020	13.5683	13.3069	24.9905

FIXED CARBON %

Ash%	Moisture%	Volatile matter%	Fixed carbon% 100-(Ash%+ Moisture%+Volatile matter%)
1. 26.1239	0.7179	24.8892	48.269
2. 26.5151	1.0973	24.9905	47.3971

1.4 CONCLUSIONS

* From the given sample the moisture percentage obtained = (I) 0.7179 %
(II) 1.0973 %

*From the given sample the ash percentage obtained = (I) 26.1239 %
(II) 26.5151 %

* From the given sample the volatile matter obtained = (I) 24.8892 %
(II) 24.9905 %

*From the given sample the fixed carbon obtained= (I) 48.269 %
(II) 47.3971 %

CHAPTER 2

INSTRUMENTAL TECHNIQUES

2.1 TENSIO METER

Basically, a tensiometer is an instrument used to measure the surface tension of liquids. This can include liquid coatings, liquid adhesives and liquid lacquers. In practical application, they help a scientists predict and develop the behavior of a whole host of products, including cleaning agents industrial parts cleaning, of electroplating processes, measure soil moisture, and the wettability absorption and density of powders. Here, force tensiometry is utilized and it is a powerful and accurate technique to measure static surface and interfacial that tension of liquids. These direct measurements allow determination of a number of material and surface properties, such as dynamic contact angle, surface free energy, critical micelle concentration and wettability. This information can be used to determine adhesion, absorption, adsorption, cleanliness, surface tension surface heterogeneity, density, spreading, porosity, powder or sedimentation of wettability, material solubility, biocompatibility and lubricity of solid surfaces.

2.1.1PRINCIPLE OF TENSIO METER

These devices typically rely on placing a solid object into the liquid of interest, determining the length of the macroscopic solid-liquid contact line (hereafter referred to as the wetted perimeter), and measuring the added force on the object resulting from its contact with the liquid. The interfacial surface tension is then recovered by dividing the measured force by the wetted perimeter. Implicit in these techniques are the assumptions that: i) surface tension does not depend on liquid-surface curvature, ii) the liquid does not apply any force to the submerged portion of the solid beyond hydrostatic pressure, and iii) the angle that the interface makes with the vertical at the contact line is known (usually assumed to be zero for the receding contact angle in the experimental determination of surface tension).

2.1.2.TYPES OF TENSIOMETERS

There are several different types of tensiometer, each measuring the surface tension of a fluid in a distinct way. The best method to use depends on the volume, viscosity, and availability of the fluid.

a) CONTACT ANGLE GONIOMETER/ TENSIOMETER

The Goniometer is most commonly used to measure the surface tension and interfacial tension of expensive liquids, as it only requires a few drops to get a reading. Using either the pendant or sessile drop methods, drops of fluid are captured by the Charge-Coupled Device camera, a device used to capture the movement of electrical charge.

The digital image of the charge is processed through computer software using the Young-Laplace equation, which describes the capillary pressure difference at the interface between two fluids.

b) WILHELMY PLATE TENSIOMETER

The simplest and most accurate form of tensiometer, the Wilhelmy Plate only requires a plate to make contact with a liquid to provide a result. The plate is usually made from filter paper, glass or platinum. In truth, it doesn't matter what the plate is made from as long as the material can be 'wetted' by the fluid. The measurement is of the force exerted on the plate. Because the plate doesn't move, it is generally a highly accurate measurement.

c) DU NOÜY-PADDDAY METHOD TENSIOMETER

The Du Noüy-Padday method works by dipping a rod in and out of a pool of liquid and measuring the precise force used to extract the rod from the fluid. The main advantages of this method are that it is repeatable and accurate even on fluids with high viscosity. The main drawback is that it requires a pool of fluid so it might not be realistic for expensive liquids.

d) DU NOÜY RING TENSIO METER

Similar to the above method, only the Du Noüy Ring tensiometer uses a platinum ring which is submersed and held horizontally in liquid and then pulled out. The ring has to be almost exactly perfect. Any scratches or damage to the ring material can alter the ring significantly.

e) BUBBLE PRESSURE TENSIO METER

By pumping a gas stream into a capillary that is immersed in fluid, this tensiometer is able to measure surface tension through the behaviour expected by bubbles forming at the tip of the capillary.

2.1.3APPLICATIONS OF TENSIO METERS

The key areas of application of the tensiometers are listed below:

- In research and development laboratories to determine the surface tension of liquids like coatings, lacquers or adhesives
- For monitoring industrial production processes like part's cleaning or electroplating
- For monitoring the detergent concentration in cleaning or electroplating processes
- To determine not only surface tension measurements but also powder wettability, absorption, cleanliness, surface heterogeneity and density.



Fig.2.1 Tensiometer

MODEL: Tensiometer K 100

MANUFACTURER: Kruss company

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