

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

Submitted to:

CSIR- NATIONAL METALLURGICAL LABORATORY
MADRAS CENTRE
(COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH)
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CERTIFICATE

This is to certify that **Mr. Senthil Kumar T** (Roll No: 42910019) 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

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About CSIR



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About NML- Jamshedpur



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 millions of years 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 being 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 significant uses of coal include steel production, cement 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 include volatile matter which 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)

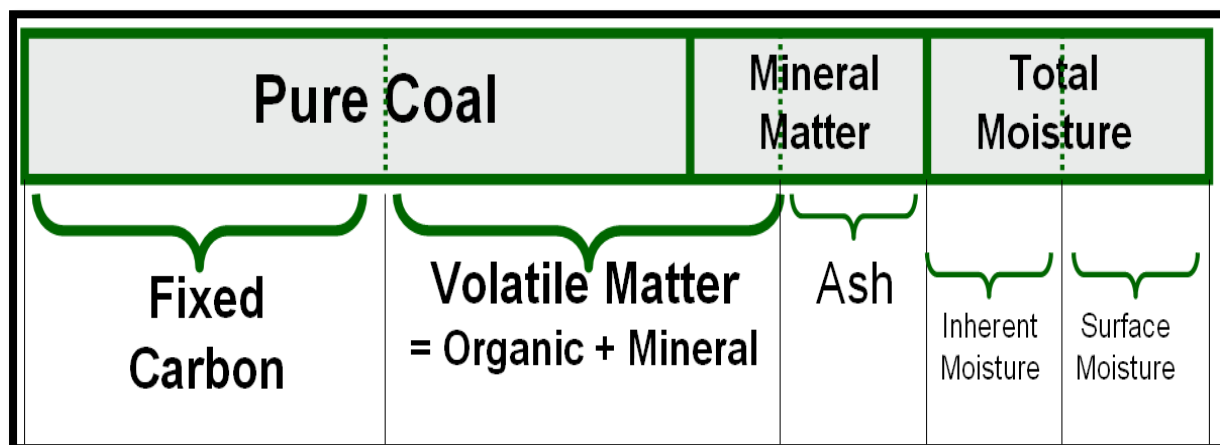


Fig.1.1. Composition of coal

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 1.3Grading 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 (G.E-Non coking coal -HS).
- * Take the empty weight of the crucible and note the value.
- * The given sample (G.E-Non Coking Coal-HS) 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 (G.E-Non Coking Coal-HS).
- * Take the empty weight of the crucible and note the value.
- * The given sample (G.E-Non Coking Coal-HS) 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 (G.E-Non Coking Coal-HS).
- * Take the empty weight of the crucible and note the value.
- * The given sample (G.E-Non Coking Coal-Hs) of 0.5000-0.5050g 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.

1.3 CALCULATIONS

Moisture %

1. $A-B/C \times 100$

$$A = 27.7147$$

$$B = 27.6989$$

$$A-B = 0.0158$$

$$C = 0.5043$$

$$A-B/C = 0.031330$$

$$A-B/C \times 100 = 3.1330$$

2. $A-B/C \times 100$

$$A = 38.6055$$

$$B = 38.5875$$

$$C = 0.5005$$

$$A-B/C = 0.018$$

$$A-B/C = 0.035964$$

$$A-B/C \times 100 = 3.5964$$

Ash %

1. $D-C/A \times 100$

$$D = 20.9890$$

$$D-C = 0.1684$$

$$C = 20.8206$$

$$D-C = 0.0333663$$

$$A = 0.5047$$

$$D-C/A \times 100 = 33.3663$$

2. $D-C/A \times 100$

$$D = 15.8229$$

$$D-C = 0.1683$$

$$C = 15.6546$$

$$D-C/A = 0.3333597$$

$$A = 0.5045$$

$$D-C/A \times 100 = 33.3597$$

VOLATILE MATTER %

$$\begin{aligned}
 &1. \frac{[M2 - M3]}{[M2 - M1]} \times 100 - M \\
 &\qquad\qquad\qquad M1 = 12.6382 \\
 &\qquad\qquad\qquad M2 = 13.6418 \\
 &\frac{[M2 - M3]}{[M2 - M1]} \times 100 = 28.9756 \qquad M3 = 13.3510 \\
 &\qquad\qquad\qquad (M) \% = 3.1330 \\
 &\frac{[M2 - M3]}{[M2 - M1]} \times 100 - M = 25.8426
 \end{aligned}$$

$$\begin{aligned}
 &2. \frac{[M2 - M3]}{[M2 - M1]} \times 100 - M \\
 &\qquad\qquad\qquad M1 = 13.0686 \\
 &\frac{[M2 - M3]}{[M2 - M1]} \times 100 = 29.3177 \qquad M2 = 14.0697 \\
 &\qquad\qquad\qquad M3 = 13.7762 \\
 &\qquad\qquad\qquad (M) \% = 3.5964 \\
 &\frac{[M2 - M3]}{[M2 - M1]} \times 100 - M = 25.7213
 \end{aligned}$$

Fixed carbon %

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

1. $100 - (33.3663 + 3.1330 + 25.8426)$

$$100 - 62.3419 = 37.6581$$

$$\text{Fixed carbon} = 37.6581 \%$$

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

$$100 - (33.3597 + 3.5964 + 25.7213)$$

$$100 - 62.6774 = 37.3226$$

$$\text{Fixed carbon} = 37.3226 \%$$

1.4 RESULTS AND DISCUSSIONS

MOISTURE%

Sample Name: (G.E-Non Coking Coal- Hs)

Empty weight	Sample weight(C)	Total weight before(A)	Total weight after(B)	Moisture% $A-B/C \times 100$
1. 27.2104	0.5043	27.7147	27.6989	3.1330
2. 38.1050	0.5005	38.6055	38.5875	3.5964

ASH %

Empty weight(C)	Sample weight(A)	Total weight before	Total weight after	Ash % $D-C/A \times 100$
1. 20.8206	0.5047	21.3253	20.9890	33.3663
2. 15.6546	0.5045	16.1591	15.8229	33.3597

VOLATILE MATTER %

Empty weight	sample weight	Total weight before(M2)	Total weight after(M3)	VM % $M2-M3 \times 100$ M2-M1
1. 12.6382	1.0036	13.6418	13.3510	25.8426
2. 13.0686	1.0011	14.0697	13.7762	25.7213

FIXED CARBON%

Ash %	Moisture %	Volatile matter %	Fixed carbon % 100-(Ash %+Moisture %+Volatile matter %)
1. 33.3663	3.1330	25.8426	37.6581
2. 33.3597	3.5964	25.7213	37.3226

1.5 CONCLUSIONS

* From the given sample the moisture percentage obtained =(I) 3.1330%
(II) 3.5964%

* From the given sample the ash percentage obtained = (I) 33.3663%
(II) 33.3597%

* From the given sample the volatile matter percentage obtained =(I) 25.8426%
(II) 25.7213%

* From the given sample the fixed carbon obtained =(I) 37.6581%
(II) 37.3226%

CHAPTER - 2

INSTRUMENTAL TECHNIQUES

2.1. X-RAY FLUORESCENCE SPECTROMETER

XRF is an analytical method to determine the chemical composition of all kinds of materials. The materials can be in solid, liquid, powder, filtered or other form. XRF can also be used to determine the thickness and composition of layers and coatings. The method is fast, accurate and the a non-destructive and usually requires only a minimum of sample. Applications are very broad and includes the metal, cement, oil, polymers and plastic and food industries along with a mining, mineralogy, geology and environmental analysis of water and waste materials. XRF is also a very useful technique for research and pharmaceutical analysis.

2.1.1.PRINCIPLE OF XRF SPECTROMETER

In XRF, X-rays produced by a source is irradiated on the sample. In most cases, the sources is an X-ray tube but alternatively it could be a synchrotron or a radioactive material. The analysis of major and trace elements in geological materials by x-ray fluorescence is made possible by the behavior of atoms when

they interact with radiation. When materials are excited with high-energy, short wavelength radiation (e.g., X-rays), they can become ionized. If the energy of the radiation is sufficient to dislodge a tightly-held inner electron, the atom becomes unstable and an outer electron replaces the missing inner electron. When this happens, energy is released due to the decreased binding energy of the inner electron orbital compared with an outer one. The emitted radiation is of lower energy than the primary incident X-rays and is termed fluorescent radiation. Because the energy of the emitted photon is characteristic of a transition between specific electron orbitals in a particular element, the resulting fluorescent X-rays can be used to detect the abundances of elements that are present in the sample. The elements present in the sample will emit fluorescent X-ray radiation with discrete energies (equivalent to colors in optical light) that are characteristic for these elements. By measuring the energies of the emitted radiation it is possible to determine which elements are present and by measuring the intensities of the emitted energies it is possible to determine how much of each elements is present in the sample. Thus, the net intensities are used in the quantitative analysis to calculate the concentrations of the elements present in the sample.

2.1.2. WORKING OF XRF

The basic components of XRF is source, sample and detector. The source irradiates a sample and the detector measures the radiation coming from the sample. Generally, the XRF are of two types

- i) Energy Dispersive systems
- ii) Wavelength Dispersive systems

The difference between the two systems is found in the detection system. EDXRD spectrometers have a detector that is able to measure the different energies of the characteristic radiation coming directly from the sample. The detector can separate the radiation from the sample into the radiation from the

elements in the sample. Thus, this separation is called dispersion. WDXRF spectrometers use an analyzing crystal to disperse the different energies. All the radiations from the sample falls on the crystal. The crystals diffracts the different energies in different directions, similar to a prism.

2.1.3 APPLICATIONS OF XRF SPECTROMETER

X-Ray fluorescence is used in a wide range of applications, including

- research in igneous, sedimentary, and metamorphic petrology
- soil surveys
- mining (e.g., measuring the grade of ore)
- cement production
- ceramic and glass manufacturing
- metallurgy (e.g., quality control)
- environmental studies (e.g., analyses of particulate matter on air filters)
- petroleum industry (e.g., sulfur content of crude oils and petroleum products)
- field analysis in geological and environmental studies (using portable, hand-held XRF spectrometers)
- X-Ray fluorescence is particularly well-suited for investigations that involve bulk chemical analyses of major elements (Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, P) in rock and sediments.



Fig.2.1 XRF Spectrometer

MODEL: XEPOSC-147679

MANUFACTURER: Ametek Company

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