



**CHAPTER- II**  
**GENERAL**  
**EXPERIMENTAL**



## 2.0 GENERAL EXPERIMENTAL

In the present work, the mineralogical characterization coals/lignite of Jharia (Dist. Dhanbad, Jharkhand), Wardha Valley (Maharashtra), and Jammu and Kashmir (J & K) have been studied. This has been done by identifying and determining the various minerals in the raw coal and lignite samples and subsequently in the low temperature ash samples of these coals and lignite. For this, the chemical analysis and many complementary instrumental techniques have been used. For a comprehensive mineralogical characterization of the coals and lignite, the experimental data on X-ray Diffraction, (XRD) Thermal analysis, and Infrared (IR) spectroscopy were supplemented by chemical analysis also. The individual procedures and the details of the experimental conditions set out for these studies, including the method of sample preparation, are described in the following pages.

### 2.1 Selection of Coals

For this work both the bituminous and semi-anthracite coals of different geographical locations, viz., Jharia (Dist. Dhanbad, Jharkhand), Wardha Valley (Nagpur, Maharashtra) and Jammu & Kashmir, were selected. The coal samples were collected from different sampling points of the coal mines. These comprised the following :

- i) Lodna coal (Lodna colliery, Jharia Coalfield) Dali Mine (Kalakot Coalfield)
- ii) Bhowrah Coal (Bhowrah Colliery, East Jharia Coalfield) Metka Mine (Metka Coalfield)
- iii) Sauner Coal (Kamptee and Bandar Coalfields of Wardha Valley Nagpur (Western Coalfields of Coal India Ltd.)
- iv) Tandsi coal (Pench Kanhan Valley Coalfield, Western Coalfields of Coal India Ltd.)
- v) Pathkhera coal (Pathkhera Coalfield, Wardha Valley, (Western Coalfields of Coal India Ltd.)

- vi) Metka coal (Metka Coalfields, Jammu)
- vii) Tata Pani coal from Tata Pani Mine (Kalakot Coalfields, Jammu)
- viii) Chakkar coal from Chakkar Coalfield, Jammu)
- ix) Kashmir Lignite from Nichahom Coalfield (Kashmir Valley Kashnir, J & K)

It may be mentioned here that the coals and lignite of the J&K state are typical and different from other coals of the country in that they were formed under different Geological conditions prevailing during the coalification process. Unlike the bituminous coals of other parts of the country, the Jammu coals are more natured and semi anthracitic type. They are extremely friable, flaky and powdery in nature and are usually mixed with clay matrix near the outcrop. These coals do not show any regular bedding plane and crumble down to powder with little force. It may be of further interest to note that the coals of the lower horizon are somewhat harder than those of the upper horizon, although they are also sheared and crushed. There are however, a few areas in the Kalakot coalfield where the coal seams are less disturbed and some coals are available in lump form. The coals of Jammu region exhibit wide variations in quality varying from very low ash content to as high as 50% or even more within the same area and even within the same seam, and there is no regularity in the nature of the variations. In general, the coals are high in ash and fairly high in sulphur content, the total sulphur being (1- 4%) in general. The coals of Metka coalfields are still higher in sulphur content (2-7%) and at times it may go up to 14% of which 80-90% occurs in pyrite form. These coals are of very high rank, low volatile, semi anthracitic type and non caking in nature. Similarly, Kashmir lignite is also typically different in that unlike other lignite deposits, the lignite of Kashmir valley is very high in ash content (45-55%).

## 2.2 Preparation of Laboratory Coal Samples

Practically all the coal and lignite samples collected from different mines were run-of-mine (ROM) sources. Due to the mechanized mining being practiced those days the coal sample gets mixed up with appreciable quantity of free dirt. Therefore, for true representation, the samples so collected were treated for making them free from the dirt. For this, the samples were crushed to 12-0 mm size and the volume was reduced following the procedure of I.S.I. by coning and quartering<sup>224</sup>. This was then further crushed to 3 – 0 mm size and about 1.5 kg. of the sample was taken by reducing its quantity following the same method. The sample were subsequently deshaled to remove the extraneous dirt by washing them following the method based on densitometric principle float and sink method.) In case of Indian coals having ash content around 30-35% a gravity cut at a specific gravity of 1.80 was reported by Whitaker<sup>223</sup> to be safety employed for removal of extraneous dirt only, without affecting their mineral matter content. As such the medium of 1.80 sp. Gravity was prepared with Bromoform and carbon tetrachloride. The float of the samples thus deshaled was air dried and finally crushed to 211 micron sieve (72 mesh B.S.) and used for all the subsequent mineralogical characterization.

## 2.3 Method of Analysis of Coals and Lignite

Each of the above mentioned samples of Jammu coals and Kashmir lignite prepared above was subjected to proximate and ultimate analysis following the prescribed I.S.I. procedures<sup>224-234</sup>. The detailed proximate and ultimate analyses including the calorific value of these coals are included in Table VII.

## 2.4 Method for the preparation of Low Temp. Ash (L.T.A.) of the Coal and Lignite Samples.

The air dried and crushed samples of all the eight Jammu Coals and Nichahom lignite were taken separately for preparing the L.T.A. For this each individual samples was placed in a standard silica dish in thin layers and the carbonaceous matter was destroyed by oxidation in a well ventilated low temperature muffle furnace at a temperature at  $370 \pm 10^{\circ}\text{C}$  following the procedure of Hick's and Nagelschmidt<sup>235</sup>. The process of oxidation was continued till constant weight was obtained and the final residue was the low temperature ash, which was eventually used for the analysis of

minerals. Complete analysis of this low temperature ash was also made, whereby silica, alumina titanium, calcium and magnesium were determined following the prescribed I.S.I. procedure<sup>236</sup>. For this analysis silica / alumina ration were calculated. The ash analysis of the coals and lignite, including the silica / alumina ration are presented in Table No. VIII.

## **2.5 Experimental Procedures for the Instrumental Techniques used for the Identification and Characterization of Different Minerals.**

Several complimentary instrumental techniques, like X-ray diffraction, Infrared Spectroscopy, Thermal Analysis (DTA and TGA) and Scanning Electron Microscopy (SEM) were employed for a detailed mineralogical characterization of the minerals in raw sample and their low temperature ash. Details of the experimental procedures followed for each of these techniques and individually described in the following paragraphs.

### **2.5.1 X-ray Diffraction**

In general, the coal mineralogy was studied by X-ray diffractometric analysis method, following the experimental procedure described elaborately by Brown<sup>237</sup> .especially for identifying and studying the crystal structure of clay minerals. The powder X-ray diffraction method was used for the characterization of various mineral phases in the coals and lignite for which a Rich Seifert diffractometer (German make, model Ms III) was employed. This X-ray diffractometer was equipped with a scintillation counter detector and pulse height discriminator. The radiation used for the analysis and Ni filtered Cu-K radiation operated at 40 K. B. and 30 n. a. The samples were continuously scanned at a speed of 0 -30 (2 $\theta$ )/min. and the diffraction pattern was recorded on a strip chart, the paper speed being 6 mm/minute. In this manner X-ray diffractograms of all the individual coals and lignite samples and also of their corresponding L.T. Ashes were recorded. From the intensity and 2 $\theta$  values of the diffractograms, the various mineral phase were identified.

**Table – VII : Proximate and Ultimate Analysis of JW Coals and Lignite used in the present work**

Sl. No.	Coal (Colliery) Coalfield/	Proximate analysis (air dried basis)				Cal. Value K. cal / Kg.	Ultimate analysis on dmmf basis (dmmf basis)				
		Moist %	Ash %	V.M. %	F.C. %		C %	H%	N%	S%	O%
1.	Lodna (Jharia)	1.20	16.00	24.20	59.60	8700	90.0	4.7	1.9	0.7	2.7
2	Bhowrah (East Jharia)	1.0	21.9	24.9	52.3	8720	80/8	5.0	2.0	0.6	2.6
3	Kamptee (Wardha Valley)	4.2	29.7	27.5	38.6	7585	80.5	4.34	1.78	0.44	13.12
4	Tandsi (Wardha Valley)	1.4	25.2	23.6	48.8	8670	88.27	5.06	1.79	0.28	3.6
5	Pathkhera (Wardha Valley)	1.5	29.1	23.1	46.3	8540	87.7	4.46	1.78	0.30	7.69
6	Metka (Jammu)	0.8	15.3	10.9	73.6	8799	01.8	4.5	1.2	1.0	1.5
7	Tata Pani (Jammu)	0.9	30.3	10.8	58.0	8440	90.1	4.0	1.3	1.7	2.9
8	Chakkar coal	2.1	44.3	9.9	43.7	8650	88.9	4.4	1.1	0.8	4.8
9	Kashmir Lignite	11.9	49.7	33.3	5.1	6125	70.0	4.5	2.0	1.6	21.9

### 2.5.2 Infrared Spectroscopic Analysis

The mineralogical characterization of the low temperature ashes of all the above mentioned coals and lignite samples were also made by infrared spectroscopy technique, employing a Perkin Elmer 598 spectrometer. The KBr. Pellet method was used for this analysis. KBr pellet of the individual low temperature ashes of coals and lignite were prepared by firstly grinding separately the representative ash sample and A.R. grade KBr to a very fine powder (200 mesh) and then thoroughly mixing the weighed amount of both in a mortar and pestle, keeping the percentage of the ash in the mixture to about 0.5% weight of the total quantity. This mixture was eventually used for making the pellets with the help of hydraulic press at a pressure of 10 Ton. The grating of the spectrometer was fixed at 4000, 2000, 600 and 200  $\text{cm}^{-1}$  and the spectra were recorded at a scanning time of 12 minutes. All the measurements were made at room temperature i.e. at 25°C.

### 2.5.3. Derivatographic (thermal Analysis)

Thermal analysis of the low temperature ashes of the coals and lignite samples were made following the prescribed procedure for mineralogical characterization described in standard reference <sup>238-241</sup>. As the name indicates, the technique of the derivatograph gives three different functions simultaneously.

- i) Differential Thermal Analysis (DTA)
- ii) Thermogravimetric Analysis (TGA) and
- iii) Derivative Thermogravimetry (DTG) with respect to time and temperature. The derivatograph used for the study was of the system of the Paulik and Erdey manufacture by M/s. MCM( Hungarian Optical Works.) The instrument records the different functions photographically and rules chart according to programme of time and temperature. For DTA analysis, ignited alumina was used as the inert material in the holders were in loose stated without being packed and did not come in direct contact with the thermocouples.

0.7 gm of dry low temperature ash was taken for the test and the heating programme was upto 1000°C at the rate of 10°C/min. The experiment was carried out in static air. The water of hydration was

calculated from the derivatogramme. While calculating the water of hydration, correction for the loss due to residual carbon in low temperature ash, which was determined separately were done.

#### 2.5.4 Scanning Electron Microscopic Studies

In addition to the above mentioned instrumental analysis techniques, the electron microscopy –both scanning electron and transmission electron techniques and most frequently combination of both, is finding rapidly increased use as a mineralogical research tool, although it has not found an extensive application in identifying minerals in coals. Scanning electron microscopy with an energy dispersive x-ray system accessory has, nevertheless, been applied to a limited extent to study the minerals obtained from the low temperature ashing of coal<sup>242,243</sup>. However, the major problem facing this technique is the possibility of transforming some of the observed minerals by electron heating, thereby entailing some uncertainty in the characterization of the observed mineral. In any case, with due care this technique can contribute enormously to the study of minerals, especially the trace elements in coals.

In the present work also an attempt has been made to exploit the potentiality of this powerful technique, though to a very limited extent, to identify the minerals present in the low temperature ashes of Jammu coals and Kashmir lignite. The equipment used was a scanning –cum transmission electron microscope (make TBOL JAPAN, model 100-c ii TAMSCA) and was operated at the acceleration voltage of 40 kv.

The samples for electron microscopic examination were prepared in the following:

An ethyl alcohol moistened glass rod gently put on the low temperature ash has of the respective coals in such a manner that a very minute quantity of the ash was adhered on the glass rod which was then immersed in 2-3 ml of ethyl alcohol such that a suspension of ash was formed. This was thoroughly shaken in order to prevent the particles from settling. A drop of such suspension of each of the L.T. ash was carefully put on in previously cleaned and alcohol washed and dried copper stubs and these were left as such for air drying for a few minutes. In this manner, a single particle layer of the ashes was prepared to be successively used as probes. Each stub

containing the single layer film of the ash was separately inserted in the scanning electron microscope and focusing was made at a desired magnification (1500-6000 times ), so that the mineral particles were clearly visible to the naked eye. Then the photograph of magnified particle was taken with the help of the camera fitted inside the equipment and the negatives of the exposed films developed in the usual manner. In this way several photographs at different magnification of different mineral particles in each of the low temperature ashes were taken.