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Studies on the Mineralogical Characterization of Coals of Jharia (District-Dhanbad) Coal Field (Lodna & Bhowrah Coal), by X-Ray Differaction (XRD) and IR Spectroscopic Instrumental Techniques

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ABSTRACT

Experimental results of the study of the mineralogical characterization of coals by X-Ray differaction and IR are reported. These results suggest that different minerals present as major and minor phases in the coals could be distinctly characterised by X-ray diffractometry. The IR spectroscopy technique has provided additional supplementry informations for the identification of several other mienral species present in the coals which could not be detected by X-ray differation. X-ray diffraction analysis indicates that a-quartz is the major phase in 'Jharia Coals'. However in Bhowrah Coal, apart from quartz, Kaolinite too is in major phase. The minor phases present in these coals, which escaped detection by X-ray diffracton, are illite and per sulphate. Thus, these studies give a clue as to how these coals could be gainfully and efficiently utilised in coal conversion process.

Key words: Coals, IR Spectroscopy, X-ray Diffraction and Instrumental Techniques.

INTRODUCTION

The changing patterns of energy used in recent years have focused attention of the scientists, technologists and energy planners to the significance of the world coal deposits, which are the mainstay of our fossil fuel supplies. It is now well realized that till the commercially exploitable alternative sources of energy are abundantly available, coal will continue to dominate the world's energy scenario. This is particularly true for India, since it is endowed with huge reserves of this fossil fuel, sufficient to last for 200 years or so. The deposits of coal, ranging from lignite through sub-bituminous and bituminous to anthracite, belonging to different geological periods, are scattered over different geographical locations in the country. The presence of mineral matter and also several toxic and trace elements present in coal[1-4] are responsible for causing environmental pollution and as such are highly undesirable.

Eastern part of the country (Jharia) constitute the major percentage of the total reserves of coal and since the power generation in these States is dependent on the combustion of

such inferior grade high ash content coals and since large number of coal-based industries are coming up in this region, it is rather imperative to have a comprehensive knowledge of their detailed analysis and complete mineralogical characteristics to realize the full utilization potential of these coals in a fuel efficient manner.

Reported in this paper are the results of Investigations on the mineralogical characterization of coals of Jharia (Lodna and Bowrah Coals) by X-ray diffraction and IR spectroscopic technqiues.

MATERIAL AND METHODS

Selection of Coals

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 Coalfiedl)

Preperation of Laboratory Coal Samples

The samples 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. 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 Whitaket 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.

Method of Analysis of Coals

Each of the above mentioned samples of Lodna and Bowrah coal prepared above was subjected to proximate and ultimate analysis following the prescribed I.S.I. procedures[5-10]. The detailed proximate and ultimate analysis including the calorific value of these coals are included in Table I.

Method for the preparation of Low Temp. Ash (L.T. A.) of the Lodna and Bhorawh Coals

The air dried and crushed samples of the coals 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 ± 10°C following the procedure of Hick's and Nagelschmidt[11]. 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[12].

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, 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.

X-ray Diffractometry

In general, the coal mineralogy was studied by X-ray diffractometric analysis method, following the experimental procedure described elaborately by Brown-esp. 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 for which a Rich Seifert diffractometer (German make, model Ms III) was employed. This X-ray diffractometer was equipped with a scientillation 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 -3o (2θ)/min. and the diffraction pattern was recorded on a strip chart, the paper speed being 6 mm/minute. In this manner X-ray diffrectograms of all the individual coals and also of their corresponding L.T. Ashes were recorded. From the intensity and 2θ values of the diffractrograms, the various mineral phase were identified.

Infrared Spectroscopic Analysis

The mineralogical characterization of the low temperature ashes of all the above mentioned coals 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 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.

RESULT AND DISCUSSION

Result and Disccussion of X-ray defraction Analysis

A careful examination of X-ray difraction result gives rise to the following observations.

(i) The X-ray diffraction patterns of all the coals *vis-à-vis* their respective low temperature ashes are essentially similar in nature. However, in case of low temperature ashes, some additional minerals are seen to be present = albeit in minor phases, have escaped detection in original coals because of their being amorphose

(ii) α-quartz is predominantly present as major phase in all the coals, as α-quartz, characterized by the highest peaks in the difractograms. However, in Bhowrah coal, apart from quartz, Kaolinite too is in major phase, the peak of which is slightly less in intensity as compared to other coals.

(iii) The minerals present in minor phases in both type of coals are seen to be varying, differing from coal to coal. In general, the most common occurance of the minor phases in the coals is that of $FeSO_{4,}$ $7H_2O$, pyrite, illite, gypsum, mica, and anatase, characterized by prominent peaks of lesser heights in the diffractograms.

(iv) The occurrence of minerals in traces or minor phases include those of anhydride, ferrosite, and $FeSO_4H2O$, natase, which are represented by smaller yet conspicuous peaks in the diffractograms.

In this way the different minerals present as major and minor phases in the coals could be distinctly characterized by X-ray diffractometry (shown in Table II & III and in fig. 1 to 4).

Result and Discussion of Infra-red (I,.R.) Spectroscopy

The IR spectroscopy Technique has found its application in coal research as a complimentary technique to X-ray diffraction and other instrumental methods. Although this technique is used primarily for the identification and quantification of various functional groups (e.g. OH, CH_aI, CH_3, COOH), structural parameters (H_{ar}/H, H_{al}/H, C_{ar}/C) of coal, it is also used to deduce information about the different mineral species present in coal. It may be mentioned here that since the broad band of the organic phases masked the bands from the constituent minerals, the mineral characterization used to be severely handicapped earlier. However, with the finer development in the methodology of low temperature ashing, the efficient and near quantitative elimination of the organic fraction of coal became possible, paving way for the clear mineralogical identification by Infra-red spectroscopy. Thus, Karr *et a*t[13] and Estop *et al*[14] successfully demonstrated that a number of minerals occurring in coal could be accurately identified.

The I.R. spectra of low temperature ashes of of the Lodna, Bhowrah (Jharia coalfield) and Sauner, Tandsi, and Pathakhera, (Wardha Valley

Table 1: Proximate and Ultimate Analysis of Lodna and Bhorawh Coals used in the present work

S. No..	Coal (Colliery) Coalfield/	Proximate analysis (air dried basis) Moist %	/ Kg. Ash %	V.M. %	F.C. %	Cal. Value K. cal	Ultimate analysis on dmmf basis (dmmf basis) 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

Table 2: Identification of Minerals Present in Coals of Jharia by X-ray Diffraction

S. No.	Coal Sample from	Minerals Identified Major Phase	Minor Phase
1.	Lodna Coal (Jharia Coalfields)	Quartz, Kaolinite	$FeSO_4.7H_2O$, Calcite anhydride, pyrite and very minute quantity of $FeSO_4H_2O$, mica, gypsum.
2.	Bhowrah Coal (Jharia Coalfields)	Quartz	$FeSO_4$, $7H_2O$, Anhydride, pyrite and traces of calcite and Gypsum

Table 3: Minerals present in Low Temperature Ashses of Jharia Coals by X-ray Diffraction

S. No.	From	Major phases	Miner phases
1	Lodna	Quartz	Kaolinite, Illite, Anatase
2	Bhowrah	Quartz, Kaolinite and Gypsum	Calcite, Illite, Anatase

Table 4: Peak Positions (absorption bands) in the I. R. spectra of Jharia coals and the Different Minerals identified in them

Sl. No.	L.T.A. of Coal from	Absorption band at (cm^{-1}) and intensities	Minerals Identified	Remarks
1	2	3	4	5
1	Jharia Coalfields **(Lodna, and Bhorawh Coals)**	3400 (S.I.) 2640 (V.W.) 1600 (S.I.) 1400 (W.S.), 871 (W) 1380 (W.S.) 1155 (W. Shoulder), 1120 (W. Shoulder) 798, 778 (W. doublet), 687 (V.W.) 537, 1025 (S.I.) (Slightly broadened M.I., 473 (S.I.) 427 (W.Shoulder)	Sodium Montmo rillonite Illite Gypsum Calcite Persulphate Anhydrite Quartz Kaolinite	a) Kaolinite and Quartz are in major phases, kaolinite being dominant b) Small peak near 1230-1240 cm^{-1} is due to silica-oxygen stretching c) The broad envelope between 1200 and 800 cm^{-1} contains the characteristic band of mixed mineral species comprising quartz, Calcite, Illite and anhydrite. d) The bend at 1640 cm^{-1} could also be ascribed to sodium mintmorillonite

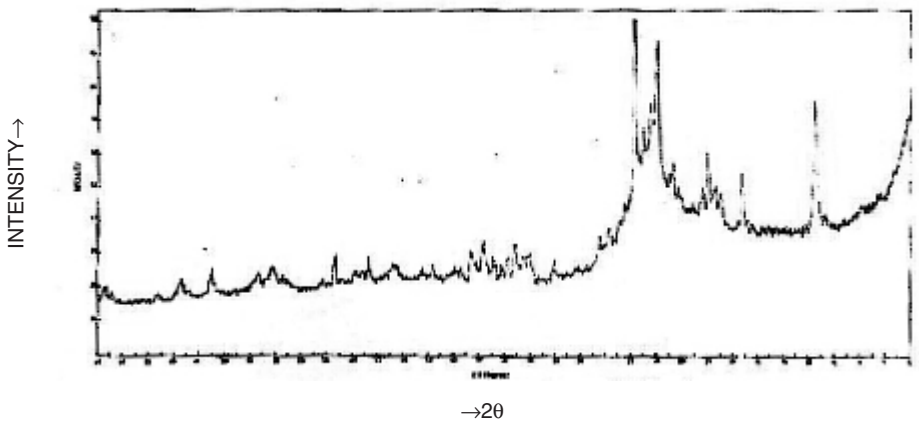

Fig. 1: X-ray diffractogram of Lodna coal (Jharia coalfields)

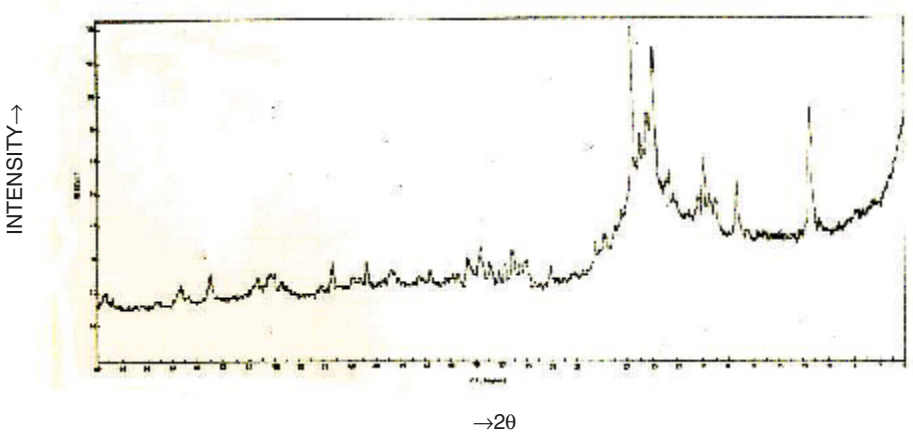

Fig. 2: X-ray diffractogram of low temperature Ash (LTA) of Lodna coal (Jharia coalfields)

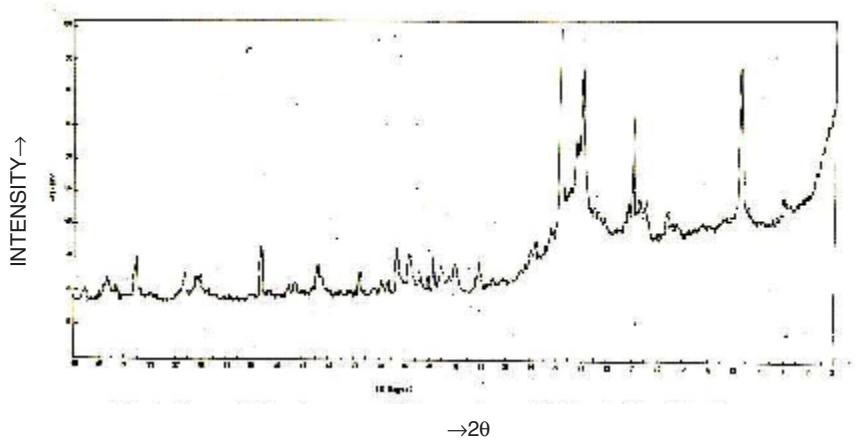

Fig. 3: X-ray defractogram of Bhowrah coal (Jharia coalfields)

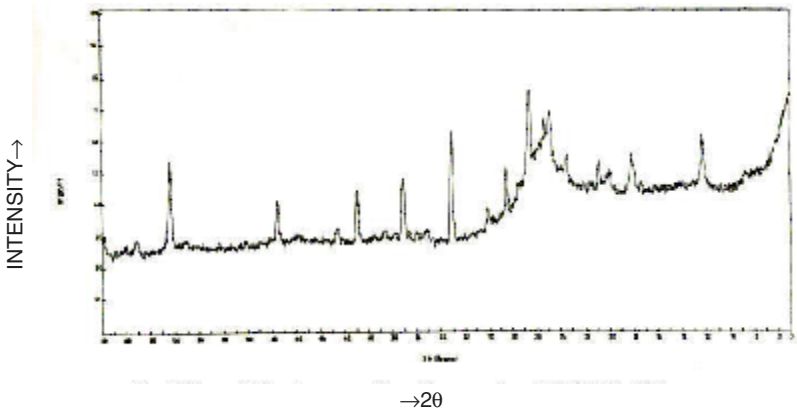

Fig. 4: X-ray diffractogram of Low Temperature Ash (LTA) of Bhowra Coald (Jharia coalfields)

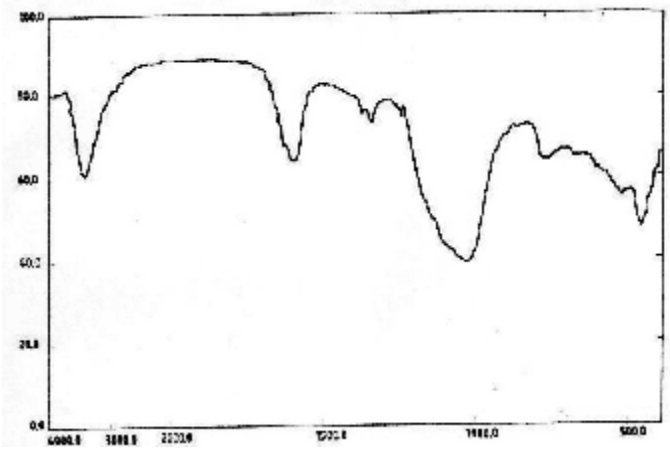

Fig. 5: Infrared (IR) spectrum of LTA of Lodna coal (Jharia coalfields)

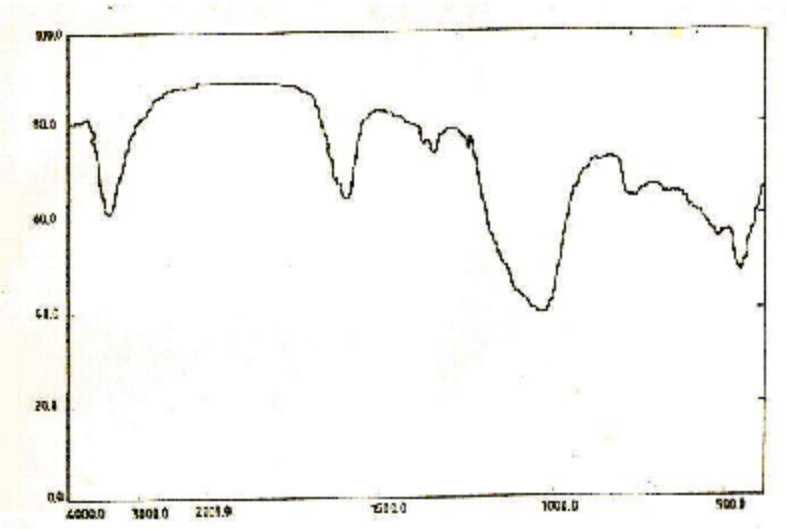

Fig. 5: Infrared (IR) spectrum of LTA of Bhowra coal (Jharia coalfields)

coalfields) are shown in figures 19 to 23 and the corresponding consolidated I.R. data namely absorption bands and the intensity of the peaks together with mineral species identified are summarized in Table IV and in fig. 4 & 6.

The observations made out from these results are mentioned below

(i) Infrared spectroscopy is a good investigative tool to complement and supplement the X-ray diffraction analysis for the characterization of different mineral species found in admixtures in the coal. Some of the minerals that escape detection by X-ray diffraction have been identified by Infra-red spectroscopy.

(ii) In almost all the studied coals of Jharia Kaolinote and Quartz are seen to appear as major phases. The presence of quartz and Kaolinite together is indicated by the presence of bans in the region 680-800 cm^{-1} and between 1200-800 cm^{-1}, characterized typically by overlapping and broadening of peaks. The ubiquitous presence of montmorillonite as a commonly occurring mineral in all the coals together with these major phases is also noticed.

(iii) The other minor phases present in these coals, which escaped detection by X-ray diffraction, are illite and persulphate.

Thus, the Infra-red spectroscopy technique has provided additional supplementary informations for the identification of several other mineral species present in the coals which could not be detected by X-ray diffraction.

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