

# ATMA MANTHAN

## PAST ACHIEVEMENTS AND FUTURE PROSPECTS OF COAL R & D

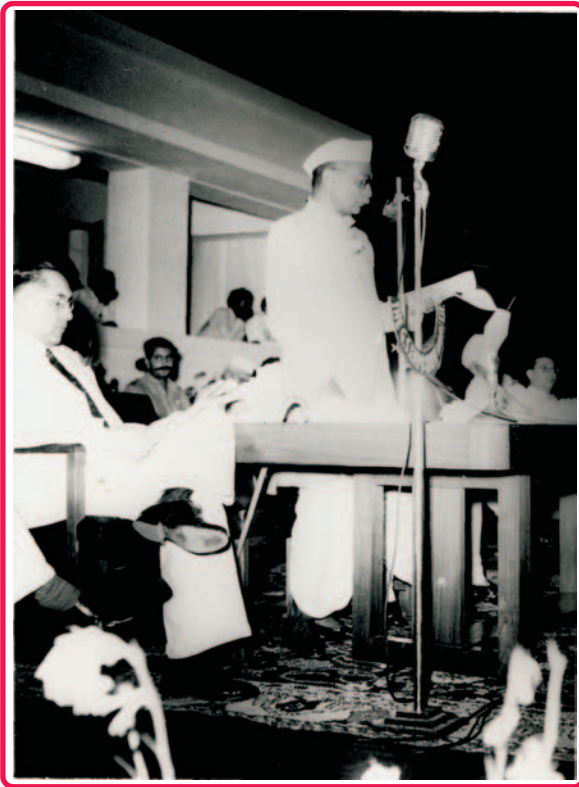


DIAMOND JUBILEE CELEBRATION  
17<sup>TH</sup> NOVEMBER 1946 - 16<sup>TH</sup> NOVEMBER 2006  
CENTRAL FUEL RESEARCH INSTITUTE, DHANBAD





Pandit Jawahar Lal Nehru, Dr. R. L. Bhattnagar & Sir C. V. Raman (from L to R) during the address of Founder Director Dr. J. W. Whitteker



Dr. Rajendra Prasad addressing at CFRI, Dhanbad

Pandit Jawahar Lal Nehru addressing at the present *Adinath Lahiri Hall*







## Mission

*"Enhance the position of the Institute as a premier R & D centre for technology development and transfer by forging strategic alliance with other agencies and continuously strive for excellence in the areas of potential expertise for generation of basic knowledge, innovation and advanced concepts in science and technology for economic, efficient and environmentally safe energy management"*

## Preamble

*Research and Development is essential in the framework of planned development of a country. It is in this perspective, just after independence a chain of National Laboratories was set up under the aegis of Council of Scientific and Industrial Research, New Delhi, the Central Fuel Research Institute, CFRI being one of the first amongst such group of laboratories. It was inaugurated by Dr. Rajendra Prasad, the first President of the Republic of India. The ceremony was also solemnised in the presence of the first Prime Minister and a great national leader, Pandit Jawahar Lal Nehru and top Scientists of the time, namely Sir C. V. Raman, Prof. Meghnath Saha, Sir J. C. Bose and many others. Very soon, the Institute became nationally and internationally acclaimed for its pioneering work on coal science and technology under the able guidance of founder Director Dr. J. W. Whitteker and Dr. Adinath Lahiri who later became the Director. One of the earliest and most valuable contributions of this Institute has been the systematic survey of Indian coals in all the coalfields eventually leading to the compilation and publication of eight monographs on Indian coals. The Institute made*

significant contributions towards the development and expansion of the major coal based industries like Steel, Power, Cement, Fertilizers by providing meaningful scientific and technical inputs in the form of basic information and database on geology, occurrence and quality of Indian coals including the best mode of its utilization for different ranks, grades and types of coals (classifications). More than anything else, the Institute since its inception has been continuously seized with the development of a sound fundamental base for coal science – its Chemistry, Physics and Constitution, for which the Institute's contribution as a whole, have already been acclaimed throughout the world as the Indian School of thought. Keeping pace with the current global trends the Institute has made a significant impact on the industrial scenario of the country with major applied research activities carried out to cope up with the ambitious industrial development programmes based on coal. **Technology awards** in the year 2002 and 2003, and **ISO 9001** accreditation in 1995 received by the Institute conforms the relevance and importance as the premier energy research institute in India. On 16<sup>th</sup> Nov'2006 CFRI is completing its Six decades of glorious existence and this august occasion is being celebrated by "**ATMA MANTHAN**" - "**Self Introspection**" on the subject '**Past Achievements and Future Prospect of Coal R & D**'.

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### *Key Facilities Available*

# Resource Quality Assessment Division



# **Resource Quality Assessment: An Account of Six Decades**

## **Physico-Chemical Evaluation of Coal Resources**

Since inception of CFRI, a wing also came into existence as Jharia Coal Survey Station. Later on other coal surveys were open in different coal fields situated in various part of the country. The wings were situated at Jharia, Jorhat, Jammu, Ranchi, Raniganj, Bilaspur and Nagpur as respective coal survey laboratories.

1. The major aim of these wings was to assess the quality of coal existing in the then virgin area of respective coal fields based on bore hole coal cores drilled by GSI, NCDC etc.  
A thorough characterization study was made for all the coal fields to find out the suitability of coal existing in the area for different industrial purposes. The work of all these laboratories was coordinated at RQA Division.
2. Correlation and identification of coal seams leading to exploration and development of mines in various coal fields.
3. Another important work of physical chemical survey was carried out at Jharia Coal Survey Laboratory and other units. This was specifically meant for detailed study of individual seams occurring in the coal field.  
The study was presented in the form of technical reports.
4. The Jharia coal survey laboratory helped many user industries in providing basic information regarding utilization aspect of coal suitable for their use.  
The major user industries were RAILWAYS, COALBOARDS, CEMENT, STEEL, POWER, FERTILIZER, and COKE OVEN.
5. Apart from above Jharia coal survey took up many projects some of which are
  - a) The washability and carbonization characteristics of B and C Category of coals of JHARIA COAL FIELD.
  - b) Washability and carbonization characteristics on NLW coals.
  - c) Washability and characterization study of Botswana coal.
  - d) Washability and characterization study of Jagganath colliery, Chirimiri colliery. etc.
  - e) Study of slurry at discharge point of four washeries, sponsored by Pierce Lessly.
  - f) Identification of direct feed prime coking coal in jharia coal field
  - g) Compilation of data for publication of Indian Coal volumes.

Jharia Coal Survey Laboratory which was later named as Coal Characterization Section, participated actively in many important projects taken up at CFRI.

- 1) Sampling and analysis of washed coal of washeries situated in Jharia coal field.
- 2) Washability and characterization studies of boreholes coal cores drilled coal cores, sponsored by Videocon.
- 3) Identification and evaluation study of coals in BCCL mines.
- 4) Development of equivalency charts for conversion from UHV to GCV.

### **Present Activities**

- 1) Study of bore hole coal cores of Raniganj CF, Talcher CF, Birbhum CF, Brahmani CF and Rajmahal CF.
- 2) Technical aid to industries by characterization of coal ,coke lignite etc for proper industrial utilization.
- 3) Participation in various ongoing projects taken up at the institute
- 4) Participation in various inhouse, collaborating and taskforce projects.

### **Future Activities**

- 1) Studies of borehole coal cores in virgin as well as working areas of various coal fields of our country.
- 2) Technical aid to industry by characterization of coals for proper industrial utilization.

The work carried out in the laboratory has directly contributed in increasing the ECF of the Institute.

## **Coal Geology & Coal Petrology: A Note on Six Decades' of Research innovation**

The present coal petrology section, earlier known as Geology Section had been actively engaged with the Resource Quality Assessment activity since inception of CFRI. Initially the section was engaged in detailed geological investigation for exploration of new coal measures in the country in association with Geological Survey of India along with characterization of the coal for specific end uses.

Coal quality assessment or characterisation of coal is a major area that is the controlling factor for all coal utilisation activities.

Both before and after nationalisation of Indian coal mines, authentic information has been generated on the quality of Indian coals (coking and noncoking/caking/thermal) by way of physical, geological chemical survey of coals from different collieries as well as from the study of borehole cores drilled by various organisations. The data bank thus developed, helped the coal industry in general and steel and power sector in particular. This prolific work has found its application in the estimation of coal reserve/resources of the country by Geological Survey of India (GSI), standardisation of coal and products by Bureau of Indian Standards (BIS), mine planning by Central Mine Planning & Design Institute Limited (CMPDIL), grading of coal by Coal Controller and all areas of coal utilisation. A network of highly capable survey units, generating extensive and intensive information on resource quality with their utilisation pattern, has been continuously catering to the user needs. Large numbers of data of Indian coals of different coalfields were thus compiled in 'Indian Coal' volumes (eight) and in CD's.

***Following the world trend in coal research, coal petrographic studies were also started in the Geology Section of CFRI during the early fifties of the last century*** where substantial work has been carried out since then. The R & D on coal petrological studies have covered original contributions to the status of knowledge in both fundamental and applied areas of earth science with special reference to coal geology and coal petrology.

Studies on the petrological properties of coal have contributed significantly to the different R&D activities related to basic coal science, coal preparation, coal carbonization, coal liquefaction and coal combustion.

These days the section has taken some initiative in the clean coal technology especially related to Coalbed Methane, CO<sub>2</sub> Sequestration, Underground Coal Gasification etc.11/8/2006.

### ***Broad Areas of Activity***

- Development of basic experimental technique.
- Study in the area of mineral exploration as applicable to coal is in regard to the systematic characterization of a vast majority of coals and lignites under exploration from all the important coalfields through a build-up of their petrographic profile vis-à-vis correlation from the petrographic and chemical composition of coal seams,
- Studies on fluorescence characteristics of coal and lignite.
- One of the most fundamental nature is that pertaining to the studies on petrographic aspects of coal structure with special reference to the correlation of conventional rank parameter i.e. reflectance of vitrinite.
- Innovative supplementation of new lines of maceral identification and application studies, and CFRI was pioneer in introducing the concept of 'Semi-vitrinite' for Indian coals.
- Correlation between petrographic parameters and chemical/technological properties of coal particularly for metallurgical uses. Several graphical correlations have been developed for Indian coals relating the chemical/carbonisation properties of coal with the maceral and reflectance values. The hot and cold strength of the coke were also correlated from the petrographic studies.
- Assessing the quality of coal/blends for coke making from the supplied data.
- Beneficiation of difficult-to-wash coals and optimization of composite washing of mixed coals.
- Microtextural characterization of coke and their importance,
- Special studies on sulphur minerals and their distribution pattern vis-à-vis possibilities of their removal by bacterial means,
- Correlation between reflectance of vitrinite and their grey scale values by image analysis system,

- Studies of combustion characteristics of coal with relation to their petrographic properties
- Petrographic studies of coal and their correlation with the gas content of coal measures for Coalbed Methane (CBM) operations.
- Gainful industrial utilization of unused large quantity reserve of heat affected coals/jhama from Jharia and Raniganj coalfields.

## Major Contributions

### Coal preparation

- Distributions of macerals in different density fractions after size reduction to different sizes have been carried out for difficult to wash coking coals. This knowledge help to identify the density of cut so that the final cleans have sufficient amount of reactive macerals commensurate with the reflectance to ensure production of metallurgical grade coke. The petrographic data can be used in conjunction with the washability data in doing simulation studies for predicting the yield at particular ash and reactive level.
- Detailed investigations have been carried on the maceral concentration of the coal fines. This analysis is a prerequisite for knowing the concentrates of reactive macerals in flotation concentrate at the desired ash level. Furthermore, this is a unique tool, which has been used to identify the degree of oxidation of coal fines, which adversely affect the flotation behaviour.

### Coal carbonization

Extensive work has been done at this institute on the role of petrographic parameters on the formation of metallurgical coke. Some of the major contributions are:

- Development of mathematical model for prediction of cold strength of coke by petrographic parameters.
- Identification of blend components (both imported and indigenous coals) and the optimum mix for production of desired coke quality. Such studies have been carried for most of the steel plants. A SAIL-CSIR Interaction project entitled “Optimisation and control of blend proportion of different coals at steel plant cokerries from the study of their **petrographic characteristics**” was carried out.
- To assess the coke quality from the petrographic results of coal/blends.

### Microscopic studies of Mesophase



Besides the above two areas significant contributions have been made in identification and quantification of mesophase, developed from coal tar pitches through microscopic studies, in a project entitled "Identification of best sources of coal and conditions to obtain tar suitable for Mesophase pitch required for special application like carbon fibers' Sponsored by Vikram Sarabhai Space Centre (VSSC)

These findings from the specialized microscopic study on mesophase pitch provided significant inputs in the preparation of the desired quality of product and helped in optimising the process parameters. The carbon fiber of high strength that has to be produced from specific type of precursor called mesophase pitch, generated by special treatment of original pitch. India meets its total requirement by importing it. In order to achieve self sufficiency to produce suitable precursor pitch from coal tar for carbon fiber.

### **Present Activities**

- To identify the role of petrographic components on the combustion behaviour of coals and their blends.

A project entitled: "The influence of rank and maceral/microlithotype and physico-chemical composition on combustion of pulverised coal" (Sponsored by SSRC) has been completed. The main focus was to study the effect of rank, macerals, and microlithotypes on the burning efficiency of coals of different rank and their washed fractions. Special initiatives were taken to quantify the different types of char found in the unburnt residues from Drop Tube furnace (DTF) and relate them with the petrographic compositions of the parent coals. The maceral distribution and their associations in coal of different rank play an important role in their combustion behaviour. This aspect, though acknowledged, has been rarely studied for Indian coals. To have a better understanding of the burning behaviour of Indian coals systematic, studies have been initiated to have a better insight to the burning behaviour of some Indian coals in Drop Tube Furnace (DTF).

Similar studies are being carried under different ongoing R&D projects including Task Force projects

- An in-house work has been initiated to study the "Combustion behaviour of heat affected coal, natural coke (Jhama) and their blends with non coking coal for power plants." On successful completion of the project the natural coke can be utilized as blends in thermal power plants

- A special study has been started to use Image Analysis system to measure the reflectance distribution of inertinites to identify the content of reactive inertinites. This reactive inertinites in combination with traditionally known reactive macerals may help to explain the combustion behaviour, observed in Thermo Gravimetric Analysis (TGA), DTF and Fuel Evaluation Test Facility (FETF). Special efforts are being made to find the rank dependence of the cut off boundary of the inertinites of Indian coals.
- Petrographic analyses were being carried out on different thermal coals from the major coalfields of the country for the “Development of equivalency chart between UHV and GCV”. Petrographic data thus created in this project is a part of the comprehensive data bank along with the other data set, that is being prepared by the Institute for Indian power coals.
- Gainful Industrial Utilization of Heat Affected Jharia Coals. This work was initiated to aid the coal industry, as Bharat Coking Coal Ltd., (Coal India Ltd.) in its Jharia Coalfield is having ~1250 Million Tonnes of Natural coke (generated due to igneous intrusives) & another ~2000 Million Tonnes blocked under mine fires may be studied for best utilisation in the industries, which has at present no good market. On successful completion of the work huge reserve of heat affected coal & jhama will be utilized in various industries thus making economic benefit to the coal industries. The objective of the above R&D work is petrological and chemical characterization of normal and heat affected coals including natural coke (jhama) from different seams of Jharia coalfield. Characterization of heat altered products of macerals including the micro-textures and microstructures in natural coke generated due to mine fires and igneous intrusive, and their comparison with laboratory carbonized coking coals/anthracites and Calcined Petroleum Coke under different simulated conditions. Laboratory experiments for making artificial graphite and other industrial uses from the coal, coke, natural coke, CPC and anthracite and possible recommendations for utilization of heat-altered products in various industries including the Graphitization.

Washability Study of these natural coke (Jhama) from CV area and Burragarh area, BCCL, and XIV seam Jamadoba colliery of TISCO were also carried out as this heat affected coals available in these areas with meager marketability have become challenge to industry. Due to peculiar physical and chemical characteristics viz. hardness, high abrasive index, loss in caking property, and slow in burning due to loss of volatile matter, this natural product has been discarded as a refuse. Tremendous scope exists to refine the above resource through beneficiation to be used in

several industries. The cleans, at different ash levels may be used in carbon artifact industries.

- There are several running projects of the institute where the jhama/heat affected coals were used to blend with slurry. Petrographic study finds its importance to know the nature of these blends and the quality of coke by 'top charging' as well as 'stamp charging' produced there from.
- The application of the coal petrology study is an integral part of the R&D efforts which are being regularly and essentially given to Technical -aid- to industries to support them and these external organisation are NTPC, ONGC, BCCL, SAIL, DPL, CMRI, etc. and many other organizations. The coal samples done for ONGC are in connection with one of the most important national project i.e Coal Bed Methane. Besides this the petrographic study has been carried out to assess the carbonization behaviours of coal samples obtained from SAIL, BCCL, IISCO etc and other private organisations

#### **Coalbed Methane**

- Physico-chemical and petrographic characteristics of some Gondwana coals of India for Coal Bed Methane prospecting. In connection with the Coal Bed Methane project of Ministry of Petroleum & Natural Gas, GSI has entrusted CFRI to provide a data package on physico-chemical characteristics of some coals of selected boreholes (chosen by GSI) and also on colliery samples. Data package on petrographic characteristics of coal both on borehole and colliery samples were provided as available in the Jharia, Raniganj, East & West Bokaro coalfields of the Damodar valley basin. Considering the lateral variation in micropetrographic characteristics, the four coalfields of Damodar valley basin were divided into several petrographic areas/zones/sector. These zones with stacked histograms showing the variation of maceral content and rank were shown in the respective coal field's map.
- Characterisation of coal core samples for estimation of CBM content from five exploratory boreholes - sponsored by CMRI, Dhanbad
- Capacity building for in-place gas content of the coal measures for coal bed methane exploration is in the process.

#### **Preparation of BIS standards**

Scientist of coal petrology section with the capability as convenor of BIS PCD 7.3 along with the other experts from different working groups of the institute have prepared the draft report on “Classification and Codification of Indian Coals, Lignites and Pseudo/Semi-anthracites (Third Revision of IS 770) which is sent to BIS office at New Delhi for further action.

### **Idea Propagation**

- Imparted training/demonstration on coal petrography and coke microscopic studies to the executives of - ONGC, GSI, TISCO, CMPDI, Vizag Steel Plant etc
- Through invited lectures –ISM, IICM, NTPC, DPL etc.
- Through guided programs and rendering advice on setting up of the R & D facilities.

### **National & International Seminars/ Workshops**

Scientist of coal petrology section with the capability as a **convenor** has conducted the following work shop and seminars of the Institute

- Workshop on HR Coke -2005, 26-28 February, 2005, Org. Secretary Dr A. K. Singh.
- National seminar on - Coal Science & Technology-Vision 2020 (COAL-2003) held on 20-21 April 2003, Org. Sec. Dr A. K. Singh.
- International Seminar on - Coal Science & Technology-Emerging Global Dimensions (GLOBALCOAL 2005) jointly organized by CFRI & CMRI held on 12-13 April 2005 at hotel Le Meridien, New Delhi,. Org. Sec. Dr A. K. Singh.

### **New Initiatives**

- Application of Image analysis system for whole coal reflectogram, particularly for identification of the cut off boundary of inertinite reflectance of different rank of coals for assessment of the combustion behaviour.
- Application of Image Analysis system for Vesicles/pore study of coke microstructure and on Char Morphology
- Application of Image Analysis system in the cleat, slit, micro-fracture and pore space studies of coal for Coal Bed Methane studies
- State-of-art of in-place gas estimation through desorption analyses specific to CBM operations has been initiated

- Capacity building for adsorption study of gases on coal surface for CO<sub>2</sub> sequestration and CBM activities is in the process of formulation

### **New Facilities Created**

Desorption analytical facility had been created for Coal Bed Methane studies and, state-of-art of gas measurement and estimation had been developed through the in-house project.



## ANNEXURE

### 1. List of Publication

Sl. No.	List of Publications, Paper	Name of the Journal	Date of Publication Volume No. of Journal
1	Petrographic Analysis of Raw and Washed Products - A New Technique- Lahiri, K.C	Symp. on 'Coal Washing' Ind. Min. J., 4 (9), 100 (1956)	CFRI, Sept. (1953)
2	Petrographic Studies on Indian Coal and Their Relation to Certain Chemical Properties- Lahiri, K.C. Bhattacharya, R.N.	<i>Proc. Symp. on Nature of Coal</i>	(1959) pp. 75-87
3	Studies on the Reflectance of Vitrain of Different Rank for Vertically Incident Light- Basu, T.N., Bagchi, D.	<i>Proc. Symp. on Nature of Coal</i>	CFRI (1959), pp.137-144
4	Petrographic Studies on Indian Coals (Part I)- Lahiri, K.C., Bhattacharya, R.N.	<i>J. Min. Met. F.</i> ,	Vol.9, issue5, 1961
5	Palynological and Petrographical Study of the Argada and Sirka Coals from the Chordhara and Bhurkunda Blocks- Dutta, A.K.	<i>J. Min. Met. F.</i>	Vol.12, issue3, 1964
6	On the inter-correlation of Gondwana coalfields of India, - <b>Basu, T.N.</b>	Metals and Minerals Rev.	Nov. (1964), 1-31
7	Microscopic Investigations into the Petrographic Nature of Some of the 'Dull' Coals from Indian Coking Coals- Basu T.N. and Bagchi, D.	<i>J. Min. Met. F.</i>	Vol.12, issue7, 1964 pp-213
8	Coals of East Bokaro Coalfield - An Appraisal- Basu, T.N, Gupta, S and Moitra, A.K.	<i>FRI News</i>	Vol.15, issue4, 1965 pp-141
9	Coals of Raniganj Coalfield : An Appraisal- Bhattacharjee, D.K. Basu, T.N, Banerjee, N.G and <b>Moitra, A.K</b>	<i>FRI News</i>	Vol.15, issue 3, 1965 pp-86
10	Some Regional Aspects of Sohagpur and Chirimiri Coalfields- Roychowdhury, K.K. <b>Ghose, S.</b> and Lahiri, K.C.	<i>J. Min. Met. F.</i>	Vol.13, issue 4, 1965 pp-115 & 133
11	Usefulness of Petrographic Study on the Evolution of L.T.C. Products- Bagchi, D and <b>Basu, T.N.</b>	<i>Met. Min. Rev.</i> ,	Vol.4, issue 3, 1965 pp-25 & 33
12	Research of Indian Coals- Bagchi, S, Bagchi, D and Mallik, P.C.	<i>J. Min. Met. F.</i>	Vol.14, issue 8, 1966 pp-239
13	A New Classification and Terminology of Rock Types of Bituminous Coals- Sen, S. (Miss) Mukherjee, M and Bagchi, S	<i>Qly. J. Geo. Min. Met. Soc. of India</i>	Vol.38, issue 8, 1966 pp-139
14	A Study on the Petrography and Its Relation to Properties of Some Coals from Chano-Rikba Area of the North Karanpura Coalfield-(Miss) Mukherjee, M. <b>Sen, S</b> and Gupta, S.	<i>J. Min. Met. F</i>	Vol.14, issue 6, 1966 pp-180
15	Some Aspects of Coal Seams in Chirimiri Coalfield- Roy Chowdhury, K.K and	<i>J. Min. Met. F</i>	Vol.13, issue 6, 1966 pp-175

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| 16 | <b>Ghose, S.</b><br>A Study on the Petrography and its Relation to Properties of Some Coals from the Chano-Rikba Area of the South Karanpura Coalfield- Mukherjee (Miss), M., <b>Sen, S.</b> and Gupta, S. | <i>J. Min. Met. F</i>                              | Vol.14, issue 6,<br>1966 pp-115   |
| 17 | Regional Metamorphism of Dishergarh Horizon, Raniganj Coal field - Part I- <b>Ghose, S.</b> , Mukherjee, A.K., Bagchi, S. and Lahiri, A.   | <i>FRI News</i>                                    | Vol.16, issue 3,<br>1966 pp-77  |
| 18 | Petrography and Its Relation to Certain Carbonization Properties of Some Coals from Pundi Block, West Bokaro Coalfield- Mukherjee, M. (Miss.), <b>Sen, S.</b> , Gupta, S. and Bagchi, S.                   | <i>J. Min. Met. F</i>                              | Vol.15, issue 9,<br>1967 pp-275   |
| 19 | Significance of Petrography in Interpreting Chemical Properties of Coal- Lahiri, K.C. and Mazumdar, B.K.   | <i>Canadian Coal Conference</i>                    | , Ottawa, 29-31<br>Mar. (1967)  |
| 20 | Coals of West Bokaro Coalfield : An Appraisal- Bagchi, D., <b>Mallick, P.C.</b> and Bagchi, S.   | <i>Met. Min. Rev.</i> ,                            | Vol.6, issue 7, 1967<br>pp-9  |
| 21 | Some Studies on the Effect of Epithermal Contact Metamorphism of Coal (Jhama Formation) by Lamporphyric Sill- <b>Ghose, S.</b> and Mukherjee, A.K.   | <i>Quart. J. Min. Met. Soc. of India</i>           | Vol. 40 (Part III)<br>141 (1968)  |
| 22 | The Use of Partial Demineralization in Coal Petrography- Lahiri, K.C., <b>Tumuluri, S.</b> and Moitra, A.  | <i>Proc. Sem. on "Coal-A Petrographic Approach</i> | <i>Geology Dept., Nagpur University,</i><br>26-28 March<br>(1968), p.67 |
| 23 | Petrography as an Aid to Interpretation of Certain Abnormal Chemical Properties of Coal- <b>Ghosh, S</b>   | <i>Proc. Sem. on "Coal-A Petrographic Approach</i> | <i>Geology Dept., Nagpur University,</i><br>26-28 March<br>(1968), p.79 |
| 24 | A Study on the Petrography and Its Relation to Certain Carbonization Properties of Some Indian Caking and Coking Coals- Sen, Meera (Mrs.) and <b>Sen, S.</b>   | <i>Proc. Sem. on "Coal-A Petrographic Approach</i> | <i>Geology Dept., Nagpur University,</i><br>26-28 March<br>(1968), p.78 |
| 25 | Correlation of the Gravity and Size Fractions of a Coal with Its Petrological and Chemical Composition- Lahiri, K.C., Moitra, A., Sarkar, G.G. and Lahiri, A.  | <i>Proc. Sem. on "Coal-A Petrographic Approach</i> | <i>Geology Dept., Nagpur University,</i><br>26-28 March<br>(1968), p.81 |
| 26 | Petrographic Study of the Coal from the Lower Seam at Rajhara Colliery, Daltonganj Coalfield- <b>Sen, S.</b>   | <i>Proc. Sem. on "Coal-A Petrographic Approach</i> | <i>Geology Dept., Nagpur Univ,</i> 26-28<br>March (1968), p.25          |
| 27 | Reflectance of Thermally Metamorphosed Coals in Parts of Jharia Coalfield, Bihar - <b>Chatterjee, C.N.</b> , Barun Majumdar and Bagchi, D.   | <i>Proc. Sem. on "Coal-A Petrographic Approach</i> | <i>Geology Dept., Nagpur University,</i><br>26-28 March<br>(1968), p.16 |
| 28 | Significance of Petrography in Interpreting Chemical Properties of Coal- Lahiri, K.C. and Mazumdar, B.K.   | <i>Proc. Sem. on "Coal-A Petrographic Approach</i> | <i>Geology Dept., Nagpur University,</i><br>26-28 March<br>(1968), p.77 |
| 29 | Some Studies on the Nature of Occurrence and Properties of Coals from Samla Seam (R-IV) in the Eastern Sector  | <i>Met. Min. Rev</i>                               | Vol.8, issue 21,<br>1969 pp-21  |



- of the Raniganj Coalfield- **Mukherjee, Anoop K.**, Mukherjee, Anil K. and Bagchi, S
- 30 Groupwise Classification of the Barakar Measure Seams of the Jharia Coalfield- Banerjee, B. and Alam, M.M. *Proc. MGMII Symp. III on "Coal Mining Industry", FRI News* *CFRI, Dhanbad, 14-15 Feb.(1970), pp.1-8*
- 31 Semi-Coking Coals of India- Ghosh, S *FRI News* Vol.22, issue 3, 1972 pp-94
- 32 Rank Characteristics and Petrography of Upper Gondwana Coal in Guneri, Kutch- Banerjee, B., **Alam, M.M.** and Chowdhuri, S.S *J. Min. Met. F.* Vol.20, issue 11, 1972 pp-335
- 33 Petrography and Reflectance of Coal from Badam Area - North Karanpura Coalfield, Bihar- Chakraborty, S.K., **Chatterjee, C.N.** and Bagchi, D. *Met. Min. Rev* Vol.12, issue 2, 1973 pp-3
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| 100 | Studies on the Combustion Behaviour of Coals in Drop Tube Furnace – S. Biswas, A.K. Mukherjee, <b>N.Choudhury</b> , S.G. Sahu, M. Kumar and J. Roy   | Procd. Int. Sem. On Coal Sc. Technol – Emerging Global Dimensions              | 12-13 April'2005                                     |
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| 109 | The natural fairway and their implication to CBM recovery: A perspective view- <b>S.Kumar, P.Boral, N.Choudhury, G.Ghosh and D.Mohanty.</b>  | Procd. Nat. Sem. On GTFM-2006 | Organised by ISM,Dhanbad,2-4 Nov.2006 |
| 110 | Cr-Spinel composition of the chromite ore from the Kathpal mine , Orissa: indicator of physico-chemical parameters-A.K.Sen and <b>D.Mohanty</b>  | Procd. Nat. Sem. On GTFM-2006 | Organised by ISM,Dhanbad,2-4 Nov.2006 |
| 111 | Studies on exploring new dimensions for utilizing heat affected coal as a supplementary resources in Indian Industries- <b>Ashok K Singh, N.K. Shukla,</b> B. Singh, M. Sharma, T. Gouricharan, D. D. Halder, C. K. Basu and S.K. Srivastava | Procd. Nat. Sem. On GTFM-2006 | Organised by ISM,Dhanbad,2-4 Nov.2006 |

## **2. List of Patents**

1. Patent granted to Dr. A. Mukherjee, Mr. S. Biswas, Mr. M. Kumar, Mr. S. G. Sahu, **Mrs N. Choudhury**, Mr. A. Choudhury and Dr. Kalyan Sen for “ A device for feeding pulverized coal to a furnace” from USA, Europe and India. Patent office application No.03819219.1-2301; Patent No. 468 of the year2003.
2. COALCOMB- An Expert System to Assess the threat of Spontaneous Combustibility in Indian Coals- **Ashok K Singh & Kalyan Sen** (Indian Copyright-2001).

### **3. List of Project Reports**

<b>Sl. No.</b>	<b>Name of the Report</b>	<b>Report No.</b>	<b>Year</b>
1	Report on the investigation on petrographic characterisation of coal feed of TISCO pilot coke oven and their relationship with coke strength	TR/83-1/R	1983
2	Evaluation of the characteristics of Bhutan coals and their utilization potentialities (Phase-1). Spons by M/s Penden Cement Authority, Royal Govt. of Bhutan	R/Proj/4.1.78/CFRI	1984
3	Characterisation of Kedla coals of West Bokaro coalfield with their petrographic studies and related tests. Sponsored by CMPDIL	TR/85/12-R	1985
4	Optimisation and control of blend proportion of different coals at Steel plant cookeries from the study of their petrographic characteristics	R & D. 51.02.674.86	November 86
5	Terminology and classification of Indian coals for steel plant use (SAIL-CSIR interaction project)	R&D 11.02.577.86	April 1986
6	Investigations on beneficiation and carbonisation tests on the feed coals of the proposed Sodepur Washery	TR/CFRI/1.05/88	May 1988
4	Further studies on washing and coking potentialities of the cleans and characterisation of raw coals and middlings from seams VIII, VII top, VII bottom and Vi of Kedla collieries of West Bokaro coalfields.	TR/CFRI/1.02/89	February 1989
7	Report on integrated studies on the characterisation of lignite coals from Gurha and Barsingsar areas, Bikaner district, Rajsthan	TR/CFRI/1.01/91	May 1991
8	Establishment of a computerised coal resource and quality information network in India		March 1994
9	Physico-chemical and petrographic characteristics of some Gondwana coals of India for Coal Bed Methane Prospecting	TR/CFRI/3.03/97	July 1997
11	Influence of rank and maceral/microlithotype and physico-chemical composition on combustion of pulverized fuel	TR/CFRI/1.02/03-04	May 2003
12	Report on blending of coking coal. Spons. By M/s Navnagar Metcoke	TR/CFRI/1.13/2003-04	December 2003
13	Assessing the quality and nature of coal for coke making from the data supplied by Durgapur Projects Ltd Spons by DPL, Durgapur	TR/CFRI/1.01/2005-06	March 2005
14	Characterisation of coal samples for coal bed methane, Spons by CMRI, Dhanbad	TR/CFRI/1.11/2005-06	March 2006



#### **4. List of Key Partnerships with Projects of Other Divisions**

<b>Sl. No.</b>	<b>Name of the Report</b>	<b>Report No.</b>	<b>Year</b>
1	Pilot plant studies on the selection of coking blend for Visakhapatnam steel plant	TR/83/2-R	January, 1983
2	Studies on the binder less briquetting potentialities of Kapurdi lignite	TR/CFRI/1.07/89	September 1989
3	Physico-Chemical properties of coals encountered in boreholes in the eastern part of N. Karanpura and Birbhum coalfields for coal bed methane prospecting	TR/CFRI/1.12/98-99	January 1999
4	Standardisation of auto sampler cum sample preparation unit at Nandan washery	TR/CFRI/1.11/98-99	February, 1999
5	Beneficiation and carbonisation of Muraidih (V/VI/VII) Seam coal of Jharia coalfield	TR/CFRI/1.04A/98-99	May 2000
6	Beneficiation and carbonisation of Bhowrah IV Top Seam coal of Jharia coalfield	TR/CFRI/1.04B/98-99	May 2000
7	Beneficiation of Lodna VII Bottom Seam coal of Jharia coalfield	TR/CFRI/1.01/98-99	May 2000
8	The development of handy method of coal categorization and prediction of spontaneous fire risk in mines		June 2000
19	Identification of best sources of coal and conditions to obtain tar suitable for mesophase pitch required for special application like carbon fibres	TR/CFRI/3.03/2000-01	December 2000
10	Studies on washability characteristics of coals from Dhansar and Kalyani, generation of cleans and blending of washed low volatile coals from pilot plant for their use in steel industry	TR/CFRI/1.04/2002-03	June 2002
11	Assessing the coking potentialities of imported soft/hard coals in blends with different Indian coals in various proportions in pilot scale	TR/CFRI/1.16/2003-04	March 2004

#### **5. Books and Monographs**

1. Monograph on Disergarh Seam western sector, Raniganj Coalfield- Compile by **S. Ghosh** under the guidance of A. Lahiri (1972).
2. Coal carbonisation- CFRI Golden Jubilee Monograph by T.K.Basu, P.K.Mitra, **S.G.Chowdhury**, J.Roy and A. Chowdhury (1996)
3. Coal Preparation- CFRI Golden Jubilee Monograph by B.B.Konar, S.B.banerjee, **S.G. Chaudhri**, A.Choudhury, N.S.Das, kalyan Sen.
4. Coal and Organic Petrology – Editor M.P.Singh.
5. Book Edited- Proceedings-International Seminar on Coal Science & Technology- Emerging Global Dimensions (GLOBALCOAL 2005). (Editors- **Ashok K Singh**, Kalyan Sen, S. K. Hazra & Amalendu Sinha)

## 6. Awards and Honours

1. Golden Jubilee award of CFRI received by **Dr. S. G. Chaudhuri, Mrs.N. Choudhury**, Mr. N.B. Sarkar, Dr.D.M. Bhatt, Mr.S.P. Ghose, Mr. D.S. Chatterjee, & Dr.D.K. Mukherjee.
2. Dr. S. G. Chaudhuri and Mrs.N. Choudhury received Subrata Ghosh memorial awards of MGMI on 1999-2000 and 2003-04 respectively for their outstanding performance in R & D on Coal Petrography
3. Bronze Medal awarded to **Dr. S. G. Chaudhuri** by MGMI in the year 82-83 and 85-86
4. Silver medal received by **Dr. S. G. Chaudhuri, Mrs.N. Choudhury and Dr. C.N. Chatterjee** for the paper "Petrographic approach in studying maceral reactivity with special reference to Indian coal" awarded by MGMI on 2000-01.

Fuel Science Division

## **Sixty Years Achievements of Fuel Science Division**

**Coal systematics: from proximate analysis of coal vis-à-vis moisture of coal**

### **Oxygen groups in coal**

There are four types of oxygen in coal

- (i) Carboxylic
- (ii) Phenolic
- (iii) Ketonic
- (iv) Ether type

### **Aromatic structure of coal**

Decade of fifties and sixties can be termed as decades of aromatics. Extensive work on coal structure carried at CFRI and elsewhere established that two to three ring systems are present in lignitic and sub-bituminous rank of coal, three, four and five ring systems are present in bituminous, caking and coking coals. More condensed system is present in semi-anthracite and anthracites.

### **Systematics of high volatile coal**

#### **Dimension of side chain**

CFRI's work on oxidation revealed that coal molecule carries no bigger than methyl or ethyl groups as side chain as aliphatic group.

### **Non-aromatic structure, hydro-aromatic and its disposition in coal**

CFRI work indicates that hydro-aromatic part of coal structure accounts for 80% of the non-aromatic part in coal and it is in a such a disposition that it allows for a scope for cyclisation thus forming a c-c linkage with the aromatic core structure.

**Dehydrogenation and tar formation:** Tar originates from the hydro-aromatic part of the coal structure. 10% dehydrogenation renders coal in to non-tarforming body. 33% of the total hydrogen in a coal can be dehydrogenated from a coal.

#### **Alicyclicity and smoke emission**

Coal can be converted in a smokeless one, only by direct de-hydrogenation or by oxidation de-hydrogenation.

### **Structure, behavior, relationship in coal**

Coals having more aromatics are not amenable to easy oxidation but amenable to hydrogenation/reduction. Oxygen functional groups are less in high rank coal.

### **Validation of coal model through $^1\text{H}$ - NMR**

Second moment of  $^1\text{H}$ -nmr line for the coal models (CFRI) have been tried and found very much consistent with the experimental values.

### **Combustion behavior vis-à-vis the ease of release of hydrogen from coal**

The coal which can release most of its hydrogen during a thermal shock (at combustion temperature) proves to be better for the use in combustion.

### **Engineering plastics and other strategic materials from coal**

Polymer to be prepared from the aromatic moieties obtained from coal would be suitable for engineering plastics.

## **Future**

### **Preparation of organic conductor from coal derived products**

Aromatic moieties obtained from coal oxidation having nitro group, if reduced to amino group followed by polymerization would have a prospect of being used as a organic conductor.

### **Removal of organic sulphur from NE-coal(using bacteria)**

Removal of sulphur (organic) from N.E. coal is a near impossible task, which can be done only by suitable bacteria. The work is envisaged to be undertaken in future.

### **Shell moisture in coal containing more than 35% Ash**

Coal scientist has a rough idea about the shell moisture having ash content below 35%. But for analytical correction in coal having ash more than 35%, which is very common due to present mining practice, it requires a systematic research of mineral matter on coal.

## ANNEXURE

### 1. List of Publications

1. Pyrolysis-cum-water vapour gasification of coals/lignite Part-II : Effect of cobalt (as nitrate) loading on the yield and quality of gas obtained, S. K. Srivastava and S. Alam, Fuel Science and Technology, 15, 15, **1996**
2. Effect of hydrothermal treatment on caking propensity of coal by D.K.Mukherjee, A.N.Sengupta, D.P.Choudhury, P.K. Sanyal and S.R.Rudra, Fuel, **1996** Vol.75, No.4, 477.
3. Przemiany struktur organicznych zwiaskow siarki wystepujacych w weglu z kopalni"Janina" w procesach pirilizy i hydropirolizy, Krzysztof Stanczyk, Piotr Dyala, Sunil K.Srivastava, Karbo Energochemia Ekologia, Poland,XLII, 152, **1998**.
4. Nickel catalysed water vapour gasification of chars obtained from coal-alkali reaction at 600°C, S. K. Srivastava and R. C. Srivastava, Fuel(London), 82, 93, **2003**
5. Application of Instrumental Techniques for determining the Physico Chemical Properties of Air Borne Particulate Matter, D.P.Choudhury, J. George, S.K.Thakur, R.Sen, K.K.Mishra, A.K.Gangopadhyay, A.K.Bandopadhyay and S.K.Srivastava, Ind. J. Env. Prot., 23, 881-885, **2003**.
6. Recovery of sulphur from very high ash fuel and fine distributed pyritic sulphur containing coal using ferric sulphate, S. K. Srivastava, Fuel Proc.Tech. , The Netherlands, 84, 37, **2003**.
6. Kinetics and Energetics of high sulphur North Eastern Coal Desulphurization Using Acidic Hydrogen Per Oxide, Samit Mukherjee and S.K.Srivastava, Energy and Fuels, USA 18, 1764, **2004**.
7. Role of Instrumental Techniques in Studies on Wet Oxidation of Coal : A Review, Raja Sen, Sunil K. Srivastava and Madan M. Singh, Ind. J. Chem. Tech. 12, **2005**, 719-726
8. Use of Fly ash for the production of Hard Scouring Powder, R.Sen, D.P.Choudhury, U.Bhattacharjee, G.Ghosh and S.K.Srivastava, Published as Abstract Booklet 22<sup>nd</sup> International Pittsburgh coal conference 12<sup>th</sup> – 15<sup>th</sup> September **2005**, Session Coal Energy Environment and Sustainable Development.
9. Spectrophotometric study of molecular complex formation of asphaltene with two isomeric chloranils. A. K. Ghosh, Fuel 84 (**2005**) 153–157.
10. Direct Sourcing of Coal : Part-I – Solubilization of Coal from North Eastern Region of India, Debapriya Choudhury, Raja Sen, Gora Ghosh & Sunil K. Srivastava, presented at Pittsburgh Coal Science Conference, September **2006**.
11. Trace Elements in High-Sulphur Assam Coals from the Makum Coalfield in the Northeastern Region of India, Samit Mukherjee and S.K.Srivastava, Energy & Fuels, USA In press.
12. Role of Nitro group on Coal Solubilization in Aqueous Organic Solvents, D.P.Coudhury. S.S.Choudhury, Raja Sen, Joy Mukherjee, Gora Ghosh and Sunil K. Srivastava, Communicated to Energy and Fuels (ACS), November, **2006**.

### 2. List of Patents

1. Scouring Powder Composition and Process of Production Thereof, **European Patent** 02717011.7-2108-IBO201164, October 29, **2004**, R.Sen, U.Bhattacharjee, D.P.Choudhury, L.N.Nandi and S.Moitra



2. Detergent – free Scouring Composition, **U.S. Patent** 6,969,699, November 29, **2005**, R.Sen, U.Bhattacharjee, D.P.Choudhury, L.N.Nandi and S.Moitra.

### **3. List of Reports**

1. Behaviour and utilisation of coal with respect to their petrographic composition and physico-chemical composition – **2000**.
2. Categorisation of Indian coal with respect to their proneness to auto-oxidation and spontaneous fire risk in mines – **2001**.
3. Combustion behaviour of coal with respect to their physico-chemical make-up and petrographic composition – **2004**.

## Coal Preparation Division

# **Six Decades of Relentless Service to the Nation in the Area of Coal Preparation**

## **1.0 Abstract**

Ever since its inception in 1946, the Central Fuel Research Institute has been engaged in R & D activities in the coal washing area, especially for beneficiation of difficult-to-wash coking coals of India. This has led to application of different washing techniques to save the meager coking coal reserves. As a consequence of CFRI's sustained work not only finding of reasonable solutions to the problems of the coking coal beneficiation was possible but also development of flow scheme for non-coking coal beneficiation for different end users came into being. Newer technologies for fine coal beneficiation, dry deshaling, utilization of low volatile coking coal, quality monitoring of coal have been proposed. This paper deals with all these aspects in detail.

## **2.0 Introduction**

Nature has given us a major handicap in the manner by which the vegetal debris were laid down that gave rise to our coal deposits. Due to the 'drift' origin our coals not only contain excessive mineral matter as contamination but the manner in which it occurs in our coals make them largely inherent, rather than extraneous unlike in coals of other countries. As a result, such coals are very difficult to beneficiate.

Mining of coal in small pockets in India began early in the year 1774. However, with the advent of steam locomotives in the year 1853, the production of coal slowly increased to an annual level of slightly more than 6 million tonnes at the beginning of the twentieth century and stagnated at that level. There was a sudden rise in coal production at the end of the First World War and the production reached a level of about 22.4 million tonnes in 1919. India was producing 33 million tonnes at the beginning of the First Five Year Plan (1951). Initially, the railways, was the main consumer; subsequently, as the steel industry grew, thrust was given to exploitation of the coking coal reserves in the Jharia coalfields. Coal resources are of two distinctly different categories, coking and non-coking (also referred to as thermal/steam coal). Our resources of Coking Coal used in steel and other metallurgical industries are meager and of relatively poor quality. In comparison, high ash, low sulphur and low calorific value non-

coking coal resources, which are best suited for thermal power generation, exist in fairly abundant quantities. Helped by the high growth rate of thermal power generation, the power sector has now emerged as the major consumer of coal with coal for metallurgical sector coming next, followed by other industries. Coal consumption in the railways and domestic sector has become insignificant.

The Central Fuel Research Institute (CFRI), Dhanbad (India), ever since its foundation in 1946 has been campaigning for the establishment of large coal beneficiation plants so that the inferior' (high ash) coking coals could be upgraded for the manufacture of metallurgical coke of acceptable quality. It is largely due to the co-ordinated efforts of the Coal Preparation Division that a number of washability parameters were identified and different coking coal washeries have since then been carefully planned and put into operation for upgrading of inferior coking coal. The ultimate problem of cleaning of coal in India (for coking purposes) will be the problem of fine coal washing. A concomitant problem in fine coal washing is the upgrading and treatment of fine slurry as well as the dewatering of washed smalls and slurry cake. The CFRI is fully alive to these problems and its future programme of work has been oriented to find out economic solutions to these problems. New processes like Oil- Agglomeration, Improved Flotation Process and Oleoflotation has already been developed at CFRI for simultaneous upgrading of coal fines.

The low volatile coking coal (LVCC) constitutes about 50% of the total coking coal reserves in India. These coals are characterized by high raw coal ash content (37 to 40%) and very difficult washability potential. The present production from LVCC is at least 16 to 18 mt per year, and is entirely despatched for non-coking coal use. If these coals are treated in the existing washeries which are generally 2 or 3-product systems, the cost of clean coal is high as the by product sale value is low. The Coal Preparation division carried out extensive R & D studies on these coals it has been proposed to produce a new type of by product, which enjoys high demand, namely, foundry coal having 27-28% ash and good coking properties. As the market value of foundry coal is about 2.5 times of that of middling, the production of clean coal by this concept of 4-product washery may be competitive to the world market. CFRI suggested the concept of 2-stage washeries where the first stage will have 3-product jig to produce rejects (25-28% yield at 60-65% ash), power house coals (18-22% yield at 33-35% ash) and the remaining as pre-cleans (50% yield at

22-24% ash) for the beneficiation in the second stage by Heavy Medium washers & flotation to produce steel plant cleans (25-28% yield at 17-18% ash) and foundry coal (22-25% yield at 27-28% ash). Thus, about 50% of LVCC can be utilized as coking coal. After successful trials at Steel Plant (pilot) coke ovens on clean coal beneficiated at CFRI pilot plant, the MoC has been spear heading a Demonstration Plant of capacity 1 mt per annum to study utilization aspects of LVC coals.

Coal has been recognized as the most important fuel source for thermal power generation in India. About 75% of the total coal consumed in the country and some 80% of the domestic production is used for power generation. In addition, other industries like sponge; cement, fertilizers, chemicals, paper and thousands of medium and small-scale industries are also dependent on coal for their process and energy requirements. One of the major problems in the power sector is the varying and high- ash content in the ROM coals averaging above 45%. The stipulation laid by the Ministry of Environment and Forest (MOEF), Govt. of India to transport coal of ash not exceeding 34% beyond 1000 km away is posing problems to MCL for the despatch of its coal to the power plants situated in the Southern part of India. The only solution to this problem appears to be setting up of a beneficiation plant to reduce the ash content. In spite of many techno- economical benefits that can be accrued from the use of consistent and improved quality fuel and the general recommendation in favour of beneficiation of power coals, not many washery plants have been proposed. Due to the wide heterogeneity of Indian thermal coals there cannot be a single strategy for beneficiation. The Coal Preparation Division carried out detailed washability studies of various non-coking coal sources. Based on the washability data of the coals of different region followed by computer simulation and pilot plant trial, identification of the level of washing and development of proper flowsheet including dry and wet beneficiation scheme for coals of different regions /coalfields were prepared.

### **3.0 Washability**

The purpose of coal preparation is to improve the fuel value of coal and to supply to the consumer a product consistent both in ash content and size. At the beginning of the present century, there were in India fairly large reserves of comparatively superior grade coal (below 15% ash), but the demand by the iron

and steel industry and also indiscriminate mining and use in the past have led to their depletion. Attention was therefore, drawn to the need for the beneficiation of the inferior grade coals primarily for carbonization for the production of metallurgical coke as early as in 1918.

Though some preliminary and spasmodic studies on coal preparation problem had been carried out in India prior to 1941, the detailed study of the washability characteristics of Indian coals was pioneered by the Fuel Research Committee precursor of the Central Fuel Research Institute (in which the committee was later merged) sponsored by the C.S.I.R., and this was carried out by Forres, Majumder and their co-workers during 1944-47. The earlier view that the ash material was too finely disseminated in Indian coals and commercial washing of such coals was not an economic proposition was found to be no longer tenable.

Systematic studies on the washability characteristics of Indian coals were initiated in 1944 at the Indian School of Mines, Dhanbad under the auspices of the Fuel Research Committee set up by the then Board of Scientific and Industrial Research. The Central Fuel Research Institute, Dhanbad took over these activities in 1947 and since then coal preparation has been a major area of thrust in the R & D programme of this Institute for over six decades, CFRI has been tackling the various problems of coal preparation in India and playing a major role in the planning of all the Public Sector washeries which were set up during this period and in advising the Government to formulate the policy of coal preparation in the country.

Washability is amenability of a coal to improvement in quality by cleaning. The normal procedure to assess the washability characteristics of a coal is to carry out sink and float tests in the laboratory on a representative sample of coal after crushing and screening it to proper size limits. Sink and float tests are universally used which represent one of the basic coal cleaning characteristics required in expressing the results of coal cleaning tests. Most of the coal producing countries have adapted the procedure of float and sink analysis as a laboratory method of producing basic data for designing coal-washing plant and subsequently for assessing its performance and efficiency.

The term washability implies the extent to which it is practicable to clean a coal, i.e. to remove the dirt. Raw coal is primarily a mixture of pure coal substance



(sp.gr. 1.25 to 1.29) and dirt (shale, sandstone and other minerals of sp.gr. 2.70 and above). The pattern of distribution of these foreign matters in raw coal determines the washability characteristics. The mineral matter consists of:

Mineral matter giving fixed ash:

1. Inorganic material originally present with coal forming plants giving rise to inherent ash.
2. Mineral matter deposited simultaneously with the vegetable matter the peat bog or drift stage and thus often disseminated throughout the coal mass. This may be termed intergrown mineral matter.

Mineral matter giving free ash:

3. Sedimentary materials deposited just before the vegetable matter constituting more or less clearly defined partings or dirt bands, parallel to the bedding plane.
4. Ferruginous and Calcareous matter, often occurring as nodules, lumps or flaking in cracks and joint planes of the more or less mature coal.

The mineral matter producing fixed ash cannot easily be removed by ordinary cleaning methods. The dirt bands and partings (producing free ash) by comparison can often be partly or wholly removed.

The samples of washability investigations and size analysis fall under two categories -(i) run-of-mine samples and (ii) seam or channel samples. In addition to these there are samples from trial pits, bore hole cores and in case of channel sampling in India dirt bands measuring 75mm or below are included for float and sink analysis

### **3.1 Theoretical Work**

The normal procedure to assess the washability characteristics of a coal is to carry out float and sink tests in the laboratory on a representative sample of coal after crushing and screening it to proper size limits. Float and sink tests are universally used which represent one of the basic coal cleaning characteristics

required in expressing results of coal cleaning tests. This basic data are used for designing coal washing plant and subsequently for assessing its performance and efficiency.

The standard washability curves, viz. (i) the characteristics, (ii) the float/ash and (iii) the sink/ash and (iv) yield gravity curves are well, known for representing the results of float and sink tests, and sets of these curves are required for any coal to cover the washability of each screen- size portion. This Institute has developed an ash-units or units curve by which the data from the sink and float tests and from the standard washability curves can be condensed into a single graph. It may be noted that the ash units developed independently at CFRI is more or less similar to M curve used in Germany.

Another simple middling curve has been developed from available washability data, which gives all information on the middlings produced, making it unnecessary to have any tabulated results. From this curve, drawing a simple curve can easily assess the quality of middling produced in cleaning a coal.

A graphical method for specifying the sharpness of separation of a washing plant has been developed and a new factor, called Sharpness Index expressing the same on a percentage, has been evolved, which is independent of the quality of feed, of the characteristics of coal treated and/or the density of separation. The same condition exists for near gravity ash distribution and the resultant misplacement of ash units during any separation. But so far as the same unit is concerned, the total misplacement bears a definite ratio to the amount of near gravity zone material that is affected by misplacements. This ratio multiplied by 100 directly represents the inefficiency of the plant. When the plant inefficiency value is subtracted from 100, the resultant value is designated as sharpness index.

### **3.2 Washability Index**

At the Fourth International Coal Preparation Congress at Harrogate (1962), a simple method was suggested by CFRI to define the washability characteristics of a coal in terms of an index whose values may range between 0 and 100. This index, proposed for the first time is derived from the float and sink analysis of a coal by plotting graphically the per cent distribution of the raw coal ash (as represented by ash units) at different yield levels of the floats and finally

expressing the area bounded by the curve and the diagonal (through zero yield and 100 per cent ash) as per cent of the total area bounded by the two axes and the diagonal.

On the basis of the values of washability index, a classification was put forward for international use to group different coals as per their washability characteristics.

Washability characteristic for equivalent size grading	Range of washability indices
Very easy	Above 60
Easy	41 to 60
Medium	31 to 40
Difficult	21 to 30
Very difficult	Below 20

### **3.3 Optimum Degree Of Washability (Odw) And Washability Number (Wn)**

For every coal of specified size, there is an optimal cut point, which more or less demarcates the free and fixed dirt in the coal. Technically speaking, one can expect the maximum advantage in washing a coal by separating it at this optimal cut point. CFRI described a method for determining this point and evaluating the Optimum Degree of Washability (ODW) for any coal from normal float and sink data and also the Washability Number (WN), which is useful parameter for defining the ease or difficulty of washing.

The degree of washability at any particular level cut can be calculated by the following expression degree of Washability at any particular level of cut

$$\frac{(\text{Recovery \% of clean coal}) \times (\text{ash\% of raw coal} - \text{ash\% of clean coal})}{\text{Ash\% of raw coal}}$$

According to the above expression, the Degree of washability of any coal at zero recovery level and so at 100 per cent recovery level has to be zero. In between these two recovery levels, there lies a peak point that represents the Optimum Degree of Washability. This expression makes it possible to locate correctly the specific gravity or ash level at which maximum advantage can be derived in dirt separation.

To classify coals in accordance with their washability characteristics, it may be more convenient to express the final value as the ratio of the optimum degree of washability to clean coal ash at this optimum level. Since in coals having extremely interactable cleaning characteristics, the value of this ratio is often less than 1, a magnified scale is used by multiplying the resultant value by 10 and rounding off the decimal to the nearest whole number. Thus, the following expression defines the Washability Number :

$$\text{Washability Number} = \text{ODW} \times 10 / \text{clean coal ash at optimal Cut point}$$

The values of washability Number are generally higher and the optimum clean ash level usually lower for the coal seams formed under quiescent conditions while the opposite is true for coal seams deposited under turbulent conditions. The washability Number and the optimum cleans ash undergo change according to the size of crushing and grading of raw coal.

### **3.4 A New Concept For Coal Washing Efficiency**

Several efficiency expressions like Fraser and Yancey Efficiency, Anderson Efficiency, Ash Error, yield Error, etc., are in use for assessing the efficiency of a washing operation.

However, none of the efficiency expressions has been widely accepted. While two or more expressions are used in conjunction to assess the efficiency of any washing operation, confusion may arise because of some contradictory trends figures. For a rational assessment of the efficiency of a washing process, CFRI proposed an expression which confirm to the universal concept of efficiency based on output to input ratio.

The CFRI efficiency expression is deduced as shown in equation:

$$\text{Efficiency} = \text{Degrees of washing at any effective sp. gr. level} / \text{Degree of washability at the same sp. gr. level} \times 100$$

The proposed expression makes use of the effective density of separation as the basis for comparison of practical and theoretical results. The degree of washing of any raw feed coal processed through a separating vessel can be determined

from a knowledge of the ash content of the raw coal as well as of the actual recovery and ash content of the clean coal by using the following equation .

Degree of washing at any particular gravity of cut = Recovery percent of clean coal x (Ash% of raw coal – Ash% of clean coal)/Ash% raw coal

### **3.5 Effect Of Near Gravity Materials On The Efficiency Of Washing**

An investigation was carried out by CFRI to assess the exact nature of the impairment in the conventional efficiency values particularly in relation to the increasing amount of near gravity ( $\pm 0.10$  sp. gravity) materials in a coal and to ascertain at the same time how the efficiency figures evaluated by different formulae are affected by increasing difficulty of washing. As it is not readily possible to obtain coal samples with widely varying near gravity materials at any fixed specific gravity without any changes in the shape and/or porosity of the particles, experiments were done with different synthetic mixtures of the same coal of a specified size grading (6-3 mm) prepared by proper blending of its various sp. gravity components.

The tests carried out in connection with this study fall under three different series as outlined below:

#### **3.5.1 First Series**

Six sets of tests were performed separately in the cone-separator and the cyclone washer using synthetic mixture of coal in which the near gravity materials ranged between 15.1 and 94.1 percent. The distribution ratio of plus 0.10 and minus 0.10 specific gravity materials ranged between 15.1 and 94.1 percent. The distribution ratio of plus 0.10 and minus 0.10 specific gravity materials in all the samples were kept at 50:50.

#### **3.5.2 Second Series**

Four sets of tests were conducted to study the effect of the variation in the distribution of near gravity materials (lying above and below the separation density) on the efficiency of washing. Four prepared samples were used for this purpose, all containing about 60 percent near gravity material at sp gravity 1.50 but having different distribution ratios of 1.40-1.50 and 1.50 – 1.60 sp. gravity fractions in this order 100 : 0, 66.7 : 33.3, 33.3 : 66.7 and 0:100.

### **3.5.3 Third Series**

This series included three series of tests designed more or less on the same lines as the second series of tests with the difference that the distribution ratios of materials lying above and below the neat gravity zone (1.40 to 1.60 sp gravity range) were varied. Thus, the ratios of specific gravity fractions 1.30 – 1.40 and 1.60 – 1.70 were kept at (i) 00 : 0 (ii) 50:50 and (iii) 0:100, maintaining approximately 60 percent near gravity materials in all the three mixtures.

## **3.6 Washability of Non-Coking Coal**

With increasing energy demands, stress is coming on coal for the enhanced production and utilization in electrical utilities. With the vastly increased trend in coal production and associated deterioration of the quality of coal supplies, it has become a serious problem for all concerned including producers, consumers, researchers and planners, as quality and its variability play decisive role in the design and operation of coal fired' power stations and also on the economics of power generation.

Non-coking coals in India constitute the major portion of coal reserve and practically there is no dearth in quantity of such type of coals for industries. But bulk of the resources is of inferior quality. In the Indian context, observing the steady deterioration of the coal quality actually supplied to the electrical utilities and taking into consideration its wide variability, effectiveness of using beneficiated coal for power generation has been seriously considered. In plants, operators faced genuine difficulties using coals more than 35 per cent ash. This has also been supported by the Study Group of Power Economy Committee which has shown preference for an upper limit of ash level of 32+3 per cent.

While studying the beneficiation characteristics of coking coals, it is aimed at cleaning the coals to such an extent that it will fulfill the purity requirements of metallurgical industries. In case of non-coking coals, the main objective is to meet the demands of Power Industries. It is thought that beneficiation of non-coking coals of the country should be undertaken only to the extent of removal of obvious dirt and stones. All the results of cleaning possibilities corresponding to the desired ash level of 35 per cent (preferred as the maximum ash limit for Indian Power coal) have been derived from washability results. It has been found by numerous tests that the average specific gravity of



separation for most of the non-coking coal seams of India for the recovery of 35 per cent ash clean coal is well around 1.80. The general beneficiation characteristics of some of the major and important sources are presented.

The Coal Preparation Division carried out washability studies of all the Major Coalfields bearing Non-Coking Coals in India. After detailed study of coal qualities of coalfields several results are selected to project the beneficiation characteristics of high ash Indian coals from important seams of promising coalfields.

#### **4.0 Planning of Coal Washeries**

The Central Fuel Research Institute has helped in planning and development of most of the existing coal washeries in India. It has acted as technical consultant to the Government for the planning of all the coal washeries in the public sector. Starting from a detailed and systematic survey of the available resources, it has been responsible in drawing up the plans and specifications of the washeries and conducting the guarantee and/or performance tests after the washeries go into operation. In this regard, the Institute has rendered valuable help to Hindustan Steel, National Coal Development Corporation, Durgapur Projects of West Bengal and Industrial Development Corporation of Orissa. At the request of the Indian Railways, a good amount of exploratory work was carried out and a proposal indicating the scheme and economics of washing for five washeries to be installed in different coalfields was submitted to them. For the washeries in the private sector, all the relevant washability and size analysis data required for the planning of washeries owned by M/s. Tata Iron and Steel Company, M/s. Turner Morrison and Company and M/s. Associated Cement Company were supplied by the Institute.

#### **4.1 Problems of Planning Coal Washeries in India**

Compared to the coal washing practice in other countries, the coal washing problems of India are found to be much more complicated due to the following

- a. The difficult cleaning characteristics of most of the Indian coals and consequent need to crush them to proper sizes prior to washing.
- b. Wide variations in cleaning characteristics of coal seam both laterally and vertically.

- c. Low average output from individual seams or mines with daily and /or seasonal fluctuations.
- d. Appreciable divergence in mining and / or loading practices in individual collieries.
- e. Size degradation due to repeated handling especially where a central washery is involved.

#### **4.2 Development of Coal Washeries in India and Role of CFRI**

The development of coal washeries in India has followed some definite trends. The first three coking coal washeries Jamadoba, West Bokaro and Lodna Washeries were erected in India between 1951 to 1955. The washery at Nowrazabad designed to treat non-coking coal for cement manufacture was commissioned by ACC in 1960. All these washeries have been installed at or near the pit heads. The contribution made by CFRI is detailed washability investigation.

The Kargali Washery was established in 1958 solely with the object of washing Kargali seam. CFRI conducted detailed washability test and gave full assistance in conducting the acceptance tests or efficiency trials of this washery.

The Durgapur Washery (HSL) is a central washery, which forms a part of Durgapur Steel Plant. The construction was completed in 1960. CFRI conducted detailed washability test and gave full assistance in evaluating the efficiency of operation of some of the principal washing units.

The central washery at Dugda (I) started its commercial operation in 1962. CFRI carried out detailed washability investigations of eighty different coal samples and prepared technical specifications for this washery and conducted performance studies.

The Bhojudih central washery was commissioned in 1963. CFRI was intimately associated with the planning of this washery and rendered necessary help in the preliminary investigations. CFRI undertook the performance evaluation of this washery in 1966.

The central washery at Patherdih started its commercial operation in 1965. Exhaustive tests were carried out by CFRI to study the possible sources of raw coal feed and performance evaluation of the washing units.

M/s Indian Iron and Steel Company in their colliery at Chasnalla constructed the Chasnalla washery. A number of washability tests on coals, earmarked for Chasnalla washery were carried out by CFRI using mostly borehole core samples.

The Dugda II coal washery came into existence in the year 1968. Detailed washability investigations were conducted by CFRI on various coal seams and bulk washing was done at CFRI pilot plant. On the basis of the recommended flowsheet of CFRI, the specifications were prepared by HSL, CFRI scrutinized the tenders.

The Kathara washery was installed and commissioned in the year 1969. It is the first washery to incorporate flotation cells. CFRI carried out all the basic tests for planning of this washery and CFRI representatives were deputed abroad for technical discussions with the equipment suppliers.

For installation of the Swang Washery, preliminary data and flowsheet were supplied by CFRI. The washery was commissioned in the year 1970.

In case of Giddi washery, CFRI played a major role in carrying out large scale washability investigations and a number of pilot washing tests before finalizing the flowsheet.

For Bhelatand washery, CFRI studied the washability investigations and prepared the flowsheet.

The task of planning the Sudamdih washery and preparing the feasibility report was allotted to CFRI.

## **5.0 Performance Studies**

With the installation of a large number of washeries in India during the Second and Third Five Year Plan Periods, the Coal Preparation Division activities were related not only to the planning of the washeries but also to other fields

concerning the various operational problems of these washeries. The activities covered performance and efficiency studies and acceptance or guarantee tests for different washeries in both private and public sectors.

By means of performance study of a washery, it is examined how far the guarantee in respect of the functions of the individual processing units and the plants as an integrated unit is realized in practice. Periodical study of the performance of a washer serves as a technical audit for the washery, which is essential for making desired improvements in the operation of the washery.

Efficiency study forms only a part of the performance study of a washery. It relates to evaluation of the efficiencies of various processing units such as screening, crushing, washing and dewatering units over a short period of inspection. Acceptance tests for a washery are carried out at the time of taking delivery of the plant from the contractors after the erection of the plant is completed.

The investigations, which were carried out by the Coal Preparation division relating to performance, efficiency and acceptance tests for different washeries are as follows:

- Performance Study of Lodna Washery – (Feb to May 1955)
- Performance Study of Dugda I Washery – (January 1963 to May' 1964)
- Performance of Bhojudih Washery (January 1964 to December'1995)
- Efficiency Study of Kargali Washery (January to February' 1959)
- Efficiency Study of Durgapur Washery (March to April' 1963)
- Acceptance (Guarantee) Tests for: Patherdih Washery – April to June 1966 and Durgapur Washery - March to April 1963
- Performance Study of Rajrappa Washery – Nov'1990 to Feb'1991
- Performance Study of the Flotation Circuit of Chasnalla Washery
- Performance Study of Bina Washery – Nov'1998 to Jan'1999
- Performance Study of Piparwar Washery – Dec'2001 to Feb'2002
- Efficiency Study of Air-Jig – March'2006

## **6.0 Beneficiation of Difficult-to-Wash Low Volatile Coal of Jharia**

The low volatile coking coal (LVCC) constitutes about 50% of the total coking coal reserves in India. These coals are characterized by high raw coal ash content (37 to 40%) and very difficult washability potential. The present production from LVCC is at least 16 to 18 mt per year, and is entirely despatched for non-coking coal use. If these coals are treated in the existing washeries which are generally 2 or 3-product systems, the cost of clean coal is high as the by product sale value is low.

Generally, the lower seams of any coalfield are likely to be more matured than the upper seams and consequently exhibit lower value of volatile matter, as per Hilt's law. It is no wonder that the lower seams of Jharia or East Bokaro coalfields have 15 to 16% volatile matter, as received basis. These coals show higher maturity ( $R_o = 1.3$ ) and are expected to occur in all coalfields of the globe at a greater depth wherever upper seams are being presently mined and utilized as prime coking coal. Fortunately, India is blessed with a huge reserve of such coals (15,680 mt). If it is possible to develop an economically viable technology for use of such coals, the country may even export the knowledge to others like South Africa, Botswana, etc. where the reserves of such coals are likely to be plenty.

The Coal Preparation Division carried out extensive R&D studies on these coals, some of which are highlighted below:

### **6.1 Beneficiation of Finely Crushed Coal by Heavy Medium Cyclone cum Oil Agglomeration Technique**

The R&D work aimed to study (in bench scale) the suitability of the proposed process i.e. application of Heavy Medium Cyclone cum Oil Agglomeration technique in combination for upgrading some of the inferior type of difficult-to-wash coking coals from lower seams in Jharia coalfield and assessment of the clean coals with necessary characterization studies including pilot plant coking tests.

The process proposes essentially involves:

- Crushing the raw coal to below 75 mm.
- Deshaling of the crushed raw coal (75-6 mm)
- Crushing of the deshaled product (75-6 mm) to below 6 mm and mixing with untreated minus 6 mm fraction.
- Desliming at 0.5 mm to separate fine coal.
- Beneficiation of 6-0.5 mm coal in a Heavy medium Cyclone washer.
- Upgradation of the cyclone underflow i.e. middling fraction combined with minus 0.5 mm fines by oil agglomeration technique.
- Dewatering of total cleans in centrifuge.

Composite sample of Patherdih washery feed coal, Sendra Bansjora IX seam and Lodna VIII seam coals when beneficiated by HMC-OA process produces 78.1 % of total cleans at 18.2% ash. 56.7 % of total cleans having 16.8 % ash can be obtained from composite sample of Bhowrah (north) seam, Jeenagora IX seam, Bastacola V/VI/VII seam and Khas Joyrampur VII seam after beneficiation by modified HMC-OA process.

## **6.2 Beneficiation Studies of Golukdih V/VI/VII seam coals**

### **6.2.1 Existing Scenario**

The GOCP coals, popularly known as NLW (Not Linked Washery grade), are despatched generally to thermal power plants and some selects to beehive coke ovens. Existing washeries, which were designed for upper seam coals, cannot produce techno-economically acceptable product from LVCC. Most of the existing washeries is being operated basically as 2-product washeries producing steel plant cleans (40-45% yield at 19% ash) and remaining middlings at 40-45% ash with little or no rejects. When LVC coals are directly treated in these washeries, the sink will have more than 45% ash

with no market potentiality. Such cases, though technically very difficult, will produce hardly 25-30% of clean at 18% ash and 70-75% will require disposal as waste. The cost of clean coal is likely to be more than 5 times of that the raw coal.

Likewise, a 3-product washery where the sink from the first stage washing has the possibility of being rewashed in the second circuit to yield “sweetened middlings”, cannot produce economically viable cleans from LVCC. This is because the old deshaling jigs had low reject disposal capacity (hardly 5% of the feed coals) whereas the present low volatile coking coals require at least 20% rejection. In addition, the all-cyclone washeries suffer from congenital defects of cyclones circuits and are not possible to be run under very stable condition with the aim of producing 30% overflow and consequently discharging more than 70% of solids through the underflow. If these circuits are theoretically accepted to be used for washing NLW coals, the yield of cleans will not be more than 25-30% at 18 % ash and remaining by products will be 10% rejects with 60% ash and rest middling with 45% ash with no market outlet. Even if it is aimed to produce 20% rejects, the ash content in middling will be about 41% and cannot be sold to distant power plants due to pollution control measures.

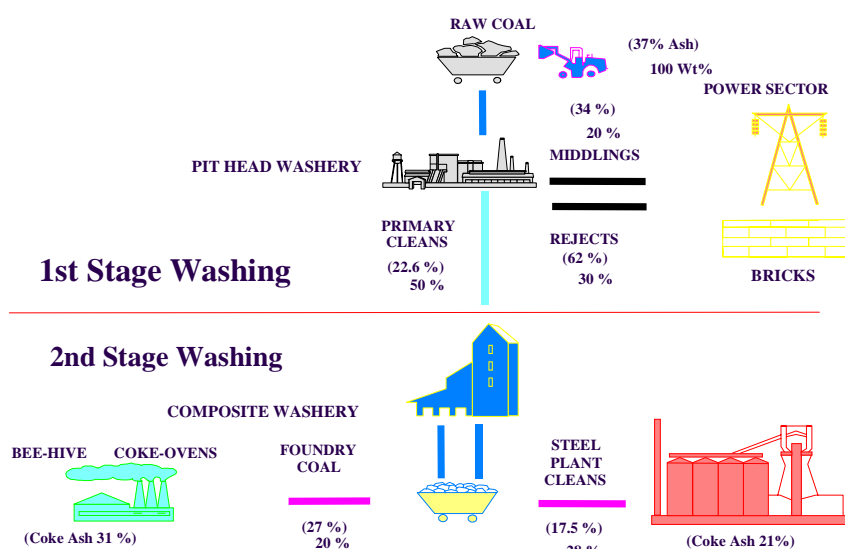
### **6.2.2 Washability Potential**

Unfortunately, due to particular geological formation these lower seam coals in Jharia and East Bokaro coalfields are of very difficult washability potential (see inset). One typical coal from eastern side of Jharia belt was crushed to two different sizes. These were deshaled at 1.8 gravity and the above 0.5 of both the sizes were tested for Float & Sink analysis. Thus, liberation potential of coal/mineral particles from LVCC by progressive size reduction is insignificant.



### 6.2.3 Beneficiation Strategy

The concept of 2-stage washeries where the first stage will have 3-product jig to produce rejects (25-28% yield at 60-65% ash), power house coals (18-22%



**Figure Concept of Multi-stage Beneficiation**

yield at 33-35% ash) and the remaining as pre-cleans (50% yield at 22-24% ash) for the beneficiation in the second stage by Heavy Medium washers & flotation to produce steel plant cleans (25-28% yield at 17-18% ash) and foundry coal (22-25% yield at 27-28% ash). Thus, about 50% of LVCC can be utilized as coking coal.

### 6.2.4 Pilot Scale Study at CFRI

About 1200 t of V-VI-VII-VIII seams coal from eastern side of Jharia was beneficiated in the CFRI pilot plant. The cleans at 17-18% ash content was carbonized at CFRI, RDCIS & subsequently tested at Bokaro Steel Limited test coke oven. The results were highly encouraging and for the first time possibility of blending such washed coal in coke oven charge as the prime coking coal component was established. The Petrographic and Chemical Analysis of cleans and Coke produced there of are presented in Table. It is apparent from the table that CSR & M<sub>40</sub> values are very

high and lower CRI & M<sub>10</sub> values indicating that the washed coal is direct Prime coking type.

<b>Petrographic and Chemical Analysis of LVCC</b>	
<b><u>Washed Coal</u></b>	
<b><u>Petrographic Analysis</u></b>	
Vitrinite %	53.2
Semi Vitrinite %	4.8
Exinite %	0.3
Inertinite %	31.5
Mineral matter %	10.2
Mean R <sub>o</sub> %	1.27
<b><u>Chemical Analysis</u></b>	
Moisture %	0.9
Ash %	18.3
V. M. % (dmf)	21.0
S.I.	5.0
Max. Fl. (ddpm)	150.0
<b><u>Coke Property</u></b>	
Porosity %	48.5
CRI	12.6
CSR	72.9
M <sub>40</sub>	82.6
M <sub>10</sub>	9.8

### **Proposed Washeries**

It is apparent that the circuit for beneficiation of NLW coals must have enough capacity to reject at least 30% of the raw feed. The proposed circuit will, therefore, have one 3-product jig to produce:

- ◆ about 28-30% rejects with 60-65% ash
- ◆ about 20-22% middlings with less than 34% ash and
- ◆ to yield pre-cleans (about 50%) with 22-24% ash.

In fact, these pre-cleans will have washability characteristics similar to upper seam coals produced from underground mines. By second stage washing of such pre-cleans in existing heavy medium and flotation circuits, only two saleable products (no rejects; no middlings; in the 2<sup>nd</sup> stage) will be obtained;

- ◆ 25-30% cleans with 18% Ash
- ◆ 20-25% foundry cleans with 28% ash

The cost of steel plant cleans will be 3 times of the raw coal costs for this case.

After successful trials at Steel Plant (pilot) coke ovens on clean coal beneficiated at CFRI pilot plant, the MoC has been spear heading a Demonstration Plant of capacity 1 mt per annum to study utilization aspects of LVC coals.

## **7.0 R & D Studies on Coal Fines**

Fine coal treatment and disposal of washery tailings are now creating alarming problems to global environment. In India, the Central coal washeries with their present beneficiation circuits are producing about 1.6 Mt of coking coal fines per annum. These slurries are not processed further in most of the washeries because of so many reasons. One of the reasons is that most of the washeries originally designed to treat easy to wash upper seam coking coals did not initially include flotation circuit as the quality of fines produced were enriched in requisite macerals necessary for metallurgical coke. The raw coal fines were simply blended with the washed coarser coals to give steel plant clean. With fast depletion of the good quality coals and deterioration in the quality of coal mined, the existing washeries suffer from severe technical constraints in terms of throughput and discharge capacities of individual washing units while treating the fines of 'difficult to wash' lower seam coals. As a result, a huge quantity of unwashed slurry is left as such and discharged as tailings to nearby ponds or lagoons, thereby polluting the surroundings. The estimated quantity may increase to as high as 4.75 Mt per annum, if the washeries operate with full designed capacity. The coal fines (below 0.5mm) having better coking propensity is a valuable prerequisite in preparing cleans for metallurgical coke. Though enriched in vitrinite content, the fines cannot be mixed directly with clean coal due to their high ash content (more than 25%) and of high percentage of silica content. The slurry needs to be washed through improved coal cleaning technology for enrichment of coking propensity and for utilization of total cleans as high valued component for coke making.

It needs to be stressed that the Indian coking coal fines have distinct characteristics than the European and other foreign coals. Indian coals of drifted origin contain intermixed bands and the mineral matters are intimately associated with the organic matrix. They also contain lesser amount of clay materials in the liberated fine coals. The natural floatability of Indian coal fines below 0.5mm is negligible (less than 5%) compared to that of foreign coals. The above characteristics should be considered while beneficiating the fines of inferior quality coals to produce a low ash (12-15%) concentrate.

## **7.1 Design and development of Flotation Plant**

Most of the existing coking coal washeries in India use simple 'Open-trough' flotation cells comprising of four to five cells in a row, but fail to deliver the recoverable yield from the presently available inferior quality coal due to the limitations in the cell design. The performance may be upgraded with their proper modifications. In the open trough design, even if the serial flotation cells are separated by partitions, the bypassing of positive agitation and re-agitation of the pulp is a common feature. More over, owing to single tailings discharge at the end of the bank of cells, the pulp level between the cells can be varied and controlled.

Research initiatives at CFRI led to the development of improved Flotation Plants for treating the inferior grade slurry. The first commercial plant in small-scale sector was installed at Dhanbad (India) in 1994. The second and third plants were scaled from 5 tph to 10tph in 1995 and in 1997 respectively.

The salient features of improvement of the plants:

1. Individual design of flotation Cell with automatic self-suction of input slurry from conditioner.
2. Belt discharge Rotary drum vacuum filter in place of Rotary disc filter.
3. Use of commercially available synthetic frother instead of pine oil.
4. Percolating bed gravity filtration pond for drying of tailings instead of dewatering system.
5. Use of low powered indigenously designed Emulsifier
6. Completely closed water circuit (zero discharge)
7. No water and air pollution

The two major equipment of the improved design are discussed below:

### **7.1.1 Conditioner**

This equipment is basically a stirred cylindrical vessel with centrally located shaft containing two sets of oppositely pitched angular impellers. One of the impellers is placed inside a cylindrical well at the top and the other one is kept below the well above the bottom of the tank. Both are rotated on a common shaft with the

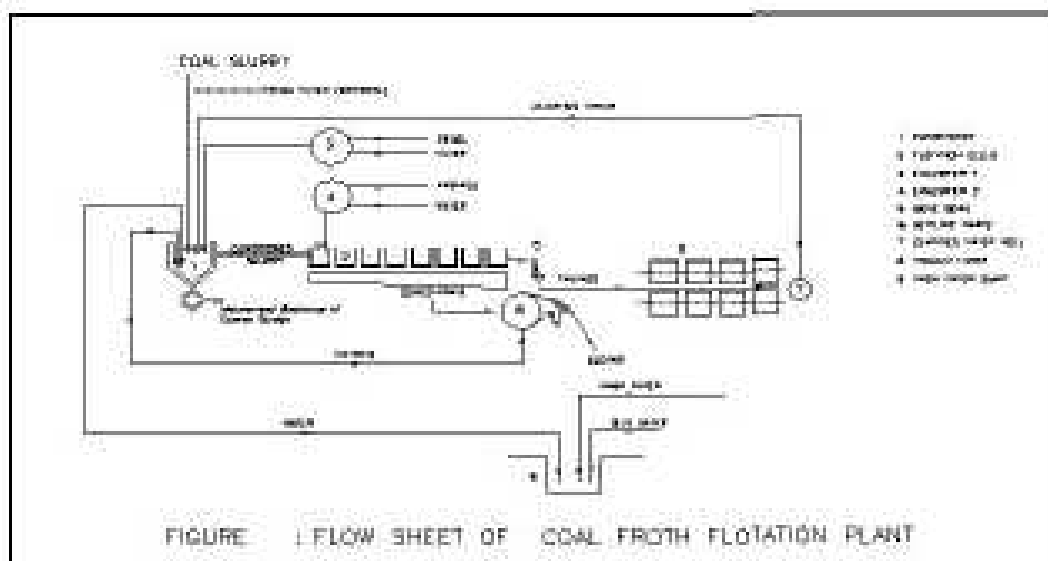
same axis of rotation. The design of the impeller is such that while the upper impeller pushes the incoming slurry downwards, the bottom impeller receives the slurry and forces them upwards outside the well and this creates a continuous re-circulating hydrodynamics which helps in thorough mixing with the added reagent collector for proper conditioning.

Routine cleaning of the deposited hard lump solids through opening bottom discharge spout are required for smooth operation. A new mechanical sludge discharge system has been provided at the bottom of the conditioner of the flotation plant that efficiently removes the bottom rejects. The speed of the agitator is maintained at 240 rpm. A suitable dose of collector is added to the conditioner. The conditioned pulp is sucked by the flotation cell through the stand pipe.

### 7.1.2 Flotation Cell

In the flotation process the successful particle bubble collisions i.e. attachment of bubbles with the hydrophobic particles determine the kinetics of the process<sup>3</sup>. The suspension of the particles, the dispersion of air as bubbles and the effects of reagents are necessary requirements in which the actual separation is realized by the rising of loaded bubbles and subsequent removal of froth. The process takes place in a very highly turbulent flow (Reynolds No.  $> 10^6$  -  $10^7$ ) to achieve good separation.

It may be observed that there are influences of solid particles on turbulence parameters. The fine solid particles dampen the turbulence considerably than coarser particles and rising bubbles incite the turbulence.



In general, the flotation cell designed by the Institute has the following characteristics.

1. Sufficient airflow required for floating the hydrophobic materials has been arranged with improved design of diffuser/impeller combination.
2. Depending on the nature of aeration and impeller speed the size of air bubbles has been restricted within the desired range.
3. Air bubble/ coal particle interaction has been increased through positive re-agitation of the pulp through successful design of individual cell, which is not possible in open trough design, especially for inferior grade slurries. Multiple dosing of reagents as required for optimum yield are being kept for difficult-to-float slurries.
4. Transfer of optimum concentrate has been made possible by allowing mixing of pulp at sufficient depth of the cell.
5. Provision has been made for final cleaning of the froth with spray of water over the froth-laden surface during removal of concentrate from the cell in order to reduce the final ash content of the concentrate.

Though the presently produced Indian coking coal fines are of high ash inferior quality, the physico-chemical separation using improved froth flotation process have been successful with CFRI designed cells. With this process it has been possible to wash slurries containing 30-35% ash down to 12-15% ash level with yields of 50-60% clean coal.

The performance of the plant is quite high compared to the conventional cells used in the washeries. Computer aided design along with R&D studies on Indian fine coals have helped in better planning and design of the process. So far the plant of capacity 10 tph is efficiently producing clean with enriched reactive contents at about 80%. These cleans are being used presently in Steel Plants for making quality coke of type G6-G7.

Successful operation of the slurry flotation plants and the study of the performance characteristics of the plants testify that this improved technology can be suitably scaled-up to a higher capacity. Installation of a Flotation Plant of 25 tph capacity in an existing washery is being envisaged and the feasibility report for setting up a 25 tph plant at Sudamdih and Patherdih Washerries was completed.

## **7.2 Integrated Process (Oleo-Flotation) for Upgrading and Dewatering of Small Coal**

This new process has been developed at CFRI for upgrading of coking coal slurry (below 0.5 mm) and simultaneous dewatering of the concentrates in combination with small coal cleans (over 0.5 mm) recovered by conventional washing system.

The important steps of this process are :

- i) Preparation of pulp of 36-40 percent solid content by thickening of coal slurry of size below 0.5 mm.
- ii) Mixing the pulp with a relatively high proportion of 1 to 1.5 percent diesel oil or paraffinic oil and 0.05 – 0.2 per cent of the tat oil of a specific character (obtained from low or medium temperature carbonization) by weight of dry coal.
- iii) Diluting the oiled pulp with water to 20 percent solid consistency and floating up the oiled flocs by controlled aeration in a cell.
- iv) Removing the oiled flocs and partially dewatering them on a curved wedge-wire screen.
- v) Mixing the product with cleaned oversize (X 0.5 mm) in a suitable mixer and dewatering the resultant product in a continuous basket centrifuge.

The Oleo-flotation circuit has been incorporate in Sudamdih washer

The outstanding feature of the process is that completely eliminates cost vacuum filtration for dewatering and also thermal drying units usual needed for reducing the surface moisture of the product to an acceptable level. By the application of this process, it is possible to reduce the moisture content of the composite coal \*13 mm – 0) to below 7 percent surface moisture even when its content fines goes up to the level of 25 to 30 percent.

For commercial development of this process CFRI collaborated with M/s McNally Bharat Engineering (India) Ltd. and accordingly, their principlas M/s McNally



Pittsburg Manuf. Corp., U.S.A. conducted some investigations in one of their pilot plants where dewatering characteristics of 37-0.5 mm coarse coal in combination with –0.5 mm upgraded (by Oleo flotation process) slurry in a centrifuge was studied. It was reported that the surface moisture of the combined product varied within a limit of 2 to 7 percent depending upon the oil dosage and the nature of oil dosage and the nature of oil used.

To establish the commercial viability of the process, the Oleo-flotation circuit has been introduced in the Sudamdih washery in the Jharia coalfield (India), which has been recently commissioned. M/s McNally Bharat Engineering Company constructed the plant. In this plant provision has been made to beneficiate a part of – 0.6 mm fine coal (20 t/hr) from the slurry circuit by Oleo flotation process and to subsequently mix the concentrates with 37-0.5 mm washed fraction so that the composite product (37 mm – 0) can be centrifuged for final dewatering at the rate of 60 to 70 t/hr. Trial runs for this circuit have been initiated.

Based on the findings of CFRI's Oleo flotation process reported at the 6<sup>th</sup> International Coal Preparation Congress held in France, 1973, West German Scientists from Stbr. Essen, working on the same line and with a view to upgrade and optimize moisture level in sizes below 10 mm, have developed their Oil-Floc process. The process aims at upgrading of super fines below 0.1 mm after getting it classified from slurry, conditioning the same with oils and subsequently dewatering the oiled coal in the centrifuge with or without mixing it with coarse slurry. At the 7<sup>th</sup> International Coal Preparation Congress (Australia 1976) they reported the findings of some pilot plant investigations by the application of some special methods for upgrading and dewatering of small and fine coals. For the beneficiation of coking coals using both pneumatic and desliming methods for fines separation, comparative investigations were made by them to evaluate the performance of five special circuits including CFRI's Oleo flotation process along with conventional filter centrifuge combination for dewatering studies. The findings covered both technical and economic aspects, using thermal drying of filter cake for economic comparison. While the different methods showed more or less similar effect on the yield and ash content of the concentrates, the reduction in product moisture varied from process to process but the best performance could be recorded by the application of CFRI Oleo flotation process.

### **7.3 Oil Agglomeration**

The scarcity of good quality low ash coal has led to a worldwide search for efficient methods of preparation of coal even by grinding the coal to very fine size. In recent years, great attention is paid to the application of the new technique called oil agglomeration technique for effective beneficiation of fine coals and increased recovery of low ash cleans with acceptable moisture content. Moreover, difficult washing coals, which are required to be ground to very fine size for a better release of the associated mineral matter and which do not respond satisfactorily to conventional beneficiation techniques like froth flotation, can be effectively upgraded by oil agglomeration technique.

For more than a decade, National Research Council of Canada has been conducting pioneering work on agglomeration, which in one form or other is presently being pursued with modification in several countries either on bench scale or pilot scale. Bureau of Mines, USA, Shell Research B-V Holland, Broken Hill (P) Ltd., Australia are doing excellent work in this line. In Japan the oil-agglomeration process has been developed to a commercial scale (unit capacity of cell being 10 t/hr). In India, an improved technique has been developed at the Central Fuel Research Institute to suit the characteristics of Indian coals.

Typical coal samples (both coking and non-coking) from almost all the major coalfields of India as well as representative samples of slurry and middling obtained from some of the operating washeries in India have responded favorably to this improved technique. Some of the results of agglomeration tests carried out in the laboratory and bench scale units are reproduced in Table 4.

Extensive R & D studies on oil agglomeration were being carried out at CFRI since early seventies for the beneficiation of coking coal fines and finely ground high ash inferior coals and middling, which don't respond satisfactorily to the conventional processes in the laboratory and bench scale. The findings of the studies were fruitfully utilized by installing a 2-tph pilot plant at Lodna washery of BCCL in 1977. While conducting the tests for longer duration, some constraints were observed in conditioner and agglomeration cell. To remove these difficulties, systematic R & D studies were carried out in 100-kg/hr-bench scale unit at CFRI and solutions of the problems were found out. The Lodna plant was renovated accordingly and it was successfully operated after removing some

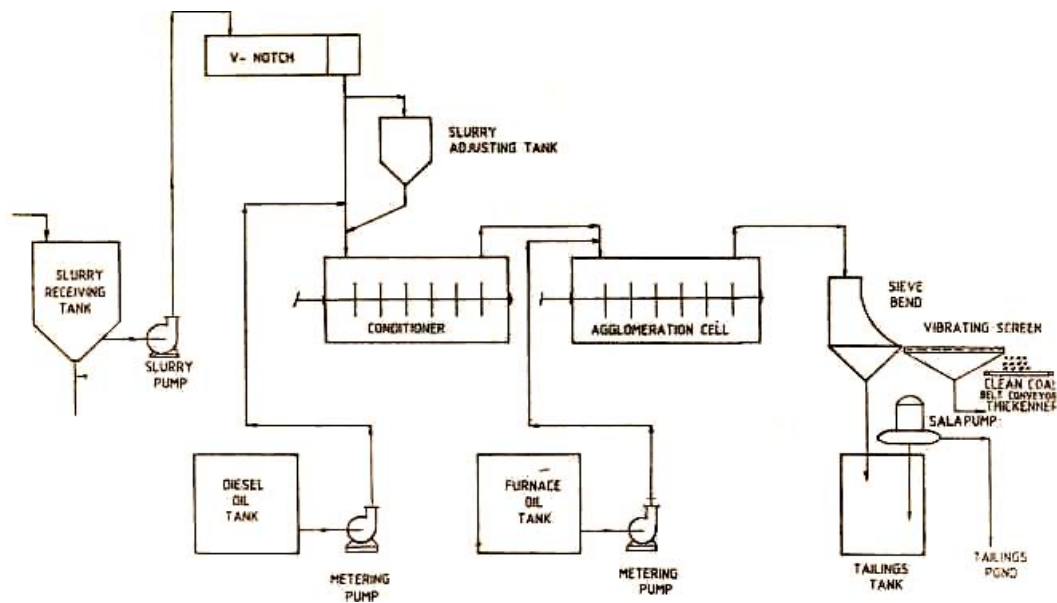
major constraints observed during its long duration trial tests. The plant could be operated successfully for 500 hrs covering 2 shifts a day in 1986-87.

It was decided to install a 10-tph module by the concerned authorities for Dept. of Coal, CIL etc to study the following aspects of oil agglomeration:

- Performance capabilities of the process in a bigger scale.
- To commission and standardize a 10 tph module for commercial application or further scale-up.

The process equipment design of two principal units: Agglomeration cell and conditioner was supplied by CFRI to MBE for fabrication and the plant was installed at Patherdih Washery.

The process flow diagram of the 10-tph oil agglomeration plant installed at Patherdih Washery is shown in Fig. The coal slurry is received at the tank and fed to the conditioner through V-notch and slurry adjusting device. The volumetric flow rate of the slurry is noted and slurry concentration is maintained to estimate the throughput of the dry coal feed and to adjust the reagent dosages. The conditioned pulp is continuously fed to the cell. The diesel and furnace oils are fed to conditioners and agglomeration cell respectively through the metering pumps. The discharge from the cell is transferred to sieve bend followed by vibrating screen. The cleans go the washery clean coal belt and the tailings are thrown out through the sala pump.



**Figure: 10 tph Oil Agglomeration Plant at Patherdih Washery**

The plant was commissioned in 1993 and it was operated continuously for 30 hours successfully. The agglomerates produced during commissioning of the 10-tph module were of high grade in terms of quality and quantity. It has been convincingly proved beyond doubt that 10 tph oil agglomeration plant at Patherdih washery is capable to produce cleans of desired quality and quantity from washery effluent/slurry which may be conventionally used for coke making directly or in blends to be used in metallurgical industries.

## **8.0 Dry Deshaling of Non Coking Coal (Selective Drop Breakage and Rotary Breaker)**

About 80% of Indian Coal reserves are of inferior quality with ash content between 24 – 45% as high as 55% in some areas. The ash content in coal as delivered to the power plants is averaging around 42%  $\pm$  2%, which leads to various problems both at the supplier and user ends. The general problems associated with high ash content of coal are: increase in erosion of heat exchange surfaces of the boiler, increased maintenance, higher transport cost, increase of particulate emissions, requirement of large area for fly ash disposal etc.,

Majority of the coal seams are inter-banded and the bands within the same seam or between the seams contain stone and hard shales. Since, such bands cannot be separated during the mechanized open cast mining operations, they get mixed with the coal thereby increasing the ash percent of the R.O.M coal.

Most of the power plants in India are not operating efficiently due to supply of high ash feed coal then the desired level. Due to increased mechanized mining, free hard stones get admixture with the coal and deteriorate its overall quality. Further, these free hard stones are transported to the CHP wherein it is crushed along with the coal and thereby reducing the overall efficiency of the crushers. Also we burn lot of fuel to transport these hard stones to longer distances.

It is very much desirable to reduce the content of hard stones to a suitable level before feeding to the power plants. The washing by conventional wet method is not economical due to high cost involved and increases in moisture level. Scope exists to develop suitable dry techniques, which can reduce the amount of stone being fed to the power plant.

A cost effective technique for minimizing the percentage of stones/hard shales from these non-coking coal is very much needed. With a view to study the possibilities of removal of stones & hard shales from R.O.M coals of various collieries/mines, CFRI developed a simple laboratory technique known as Selective Drop Breakage test, which is very helpful in understanding the breakage characteristics of coal/shale/stone from various major coalfields.

Extensive Selective Drop Breakage tests of Talcher, IB Valley, N.Karanpura and Korba Coalfields were carried out which indicated positive results for developing the rotary breaker technique.

Further based on the experience gained and supported by selective drop breakage tests, a prototype batch experimental rotary breaker was installed at CFRI. This type of installation may be helpful in the generation of data for commercial installation of industrial rotary breakers, suiting to a particular coal/rock combination.

## **9.0 Computer Application in Coal Preparation**

Computer applications in coal preparation have had an increasingly important role in recent years. The Coal Preparation Division had developed some programs mainly for prediction of practical yields at various ash levels using the

washability data. Broadly the applications can be classified in the following groups:

### **9.1 Data bank**

The area of data handling covers the storage, systematic retrieval and simple mathematical analysis of the large volume of screening and washability data generated from different collieries/seams. This allows data from drill cores, adits, channel samples and run-of-mine coal to be combined so that the characteristics of a raw coal from a given mining scenario may be calculated. Furthermore, the washery personnel can use data of individual coals to generate the washability characteristics of the blended coal that goes as feed to the washery. In recent years, microcomputer has found wide application for data handling, display of operating and accounting data in a coal preparation plant.

### **9.2 Mathematical Analysis**

#### **9.2.1 Raw washability data of plant feed**

The sets of data generated through washability studies are often subjected to error arising due to sampling and analytical methods. Before utilizing these data for generating the washability curves and subsequent prediction of practical yield, considering the performance efficiency of relevant washers, it is essential to identify the errors in the data and apply the suitable numerical techniques to correct the data. For the generation of the fractional yield and ash at any density interval other than the intervals for which the float & sink data are available, several mathematical methods such as cubic spline interpolation, mathematical programming based on the classical optimization principle etc. are being used. These smoothen data sets are extremely useful for plant simulation studies and can easily be done with the help of a computer.

#### **9.2.2 Plant Data**

Data generated in a coal preparation plant/washery include the size/density data of the feed and product streams from individual process units along with the measured values of different assays such as ash %, solids, etc. To obtain the performance efficiency of the individual units, it is necessary to calculate the mass flow in the input and output streams from the units. This is being normally done with the help of balancing the suitable assays, such as ash, size, % solid, etc. in the respective streams. In the case of washers working on the principle of

gravity separation, the mass flow values of the products are used in conjunction with the float & sink data of the product streams to determine the reconstituted feed. The use of reconstituted feed for generating the partition data are based on the assumption that the mass yields and mass distribution in the individual density fractions of the products are accurate which in most cases may not be strictly true. Hence, a separate method for generating the partition data using the mass distributions of the feed and the products should be preferred. This can be achieved with the help of a computer, utilizing a suitable data adjustment algorithm based on the principle of weighted least squares to obtain the best estimates of the mass yields that satisfy the mass balance constraints. Computer programmes developed at CFRI on the above principle has been routinely used for pilot plant and other washery data.

Another important area where computerized software's are of extreme importance is the material balance of the washery circuit. In order to obtain a material balance measurements of assays on each stream can be obtained. However due to errors present in the rates which satisfy all the mass balance constraints at the individual process units. Two distinct approaches are being used to get the total mass flows and the component mass flows in all the streams in the plant. The first approach use all the available plant data to produce the best estimate of the material balance and in the second one only that assay is chosen to obtain the material balance which has minimal sensitivity to experimental to error. For routine plant auditing purpose where minimum sampling and sample preparation is required the second approach is preferred. Considering that the chosen assay is well separated in the product streams and has the minimum relative error should make the choice of the suitable assay. In case where there are more than one assay which can be used to find the material balance it is more appropriate to use all the relevant assays, subject them to minimum adjustment so as to produce a consistent set of mass and component flow rates. The method that is being used to handle such redundant data is based on the principle of weighted least square minimization technique. The weighting/confidence factor is being used on the principle of assigning high weightage factor to the assay data, which has minimum error, and vice versa. Several robust material balance software's are available which utilize either the method of Lagrange multipliers, 'direct search' method or the method of 'independent path'.



A computer programme has "been developed at CFRI based on the 'independent path' method. This programme can accommodate the gap in the data arising out of missing values of assays in certain streams which are not possible to sample. These missing values are assigned an arbitrary value with a confidence factor of near zero. As these types of material balance programme are used with excess data the problem normally is over defined and the gap in the data would not act as any constraint.

### **9.3 Plant Simulation**

Computer simulation technique is a powerful tool in the hands of plant engineers, and designers to achieve a wide range of objectives. Besides providing a tool for quality control, these objectives. Besides providing a tool for quality control, this helps in the (a) development of flowsheets, (b) retrofitting of existing washery circuits, (c) choice of alternate circuit configurations to produce the desired products, (d) plant optimization. In the Indian context, the application of this tool has become extremely necessary, due to the wide variation of the raw coal characteristics that are being fed to the washeries, arising due to gradual depletion of easy to wash coals and also due to mechanized mining which consequently has led to complications in the operations of the existing washeries to the optimum level. It is imperative to use such techniques to identify the suitable operational parameters of individual process units, their inter-connections, to produce the products of desired quality. This will also help to suggest the suitable modifications for retrofitting the existing washeries to accommodate the abundant resource of 'difficult-to-wash' coals of the lower seams of Jharia coalfield.

For simulation studies the following procedure are to be followed:

- a) Identify all the process units and their interconnections.
- b) Identify the feed streams and the final products and refuse streams.
- c) Generate the washability data of individual size fractions of the feed and obtain a balanced raw density and size data using suitable error minimization techniques.
- (d) Develop suitable models for individual process units such as crushers, grinding mills, screens, Jigs, gravity separators etc. Several mathematical models have been developed for the respective units, which can be used after proper validation of the models with the existing plant data. Empirical

models utilizing plant data can also be used for simulation and predictive purposes of the existing plants.

- e) In case of gravity separators the model can be represented by a set of partition curves or generalized curves for individual size fractions. The methodology involved in the generation of partition data from the feed and product streams of individual washers and subsequent plotting of the partition curves are outlined in the ISO standard. The partition data represents the fraction of the feed in a density interval which goes to the cleans and  $d_{50}$  is the density at which the 50% of the feed in that density that goes to the cleans. The parameters derived from the partition/Tromp curves are the a) Relative density at the cut point ' $d_{50}$ '; b) Ecarte Probability Moyen ' $Ep$ '; c) Imperfection T d) Error area. In general the partition curves are specific to the separation units for which they are established and are independent of the density distributions of the feed and depend on size composition, feed rate and other operating conditions. Strictly speaking this is not true. The  $Ep$  value derived from the linear midportion region is independent of feed characteristics but the error area, which embraces the 'tails', is influenced by the nature of the feed. The  $Ep$  value also increases with the density of cut and also increases with the decreasing size of the feed.

As relative density is a controlling variable in a density separation process, variation in the relative density of cuts will yield different set of curves. The product distribution at any other density can be obtained by suitably shifting the curve at the desired density. However, a more accurate method of obtaining the same is to generate a generalized distribution curve for a washer using the data obtained from the set of partition curves, which is independent of density. This is obtained by plotting the partition data against the reduced density, a dimensionless variable defined as  $d/d_{50}$ . From this generalized curve the partition data at any density of cut can be obtained and used with washability data to predict the product yield and quality.

In simulation studies of a gravity Washer treating a particular size range of the feed, the input data of the separating density can predict the product distribution. But this itself is not sufficient since the  $d_{50}$  of the entire size range will not give the density of cut of the different size fractions within the size range which is necessary to know the distributions of different size fractions in the products. To

obtain this, besides the generalized curve of the composite feed, generalized curves for individual size fractions need to be generated from the same plant data. Furthermore, the following parameters would also be required.

- (i) Z50- is the ratio of d<sub>50</sub> of a size interval to the d<sub>50</sub> of the entire size.
- (ii) ZEp- is the ratio of probable error of a size interval to that of the entire size.
- (iii) S- is the ratio of the mean size interval to that of the entire size.

A functional relationship between these parameters can be derived from the existing data, which subsequently will be used to get the separation data of individual sizes from those given for the entire feed.

The data generated from the simulation studies are

- (i) The flow rate, size distribution and ash contents of the individual streams.
- (ii) The float and sink analysis and ash contents of the final and intermediate products, on a size-by-size basis.
- (iii) For gravity washers the performance criteria, the organic efficiency and the percentage of misplaced materials on a size by size basis.

The data generated from the simulation can be linked with suitable optimization criteria to optimize the separation densities for gravity separation systems so as to maximize the yield of clean at the desired quality.

#### **9.4 Plant Optimization**

The ultimate objective of any simulation studies is to select the process variables of individual process units to satisfy some optimum criteria set by the plant manager. The optimum criteria may be of achieving maximum yield of the products of desired quality or producing a product mix of different grade/quality to fetch the maximum returns. In a coal preparation plant which consists of different units ranging from crushers, screens, jig, dense medium washers, secondary crushers for precleans, middling etc. flotation, optimal setting of the process variables can be achieved to produce the desired products from individual units. However, as practiced in most of the existing washeries, gravity separation plays the dominant role and produces the maximum amount of the desired products alongwith some flotation cleans. It is thus necessary to evolve a suitable strategy to optimise the gravity separation processes in particular. As the density is the most important controlling variable in a dense medium washer, besides the other

operational variables such as feed rate, medium/coal ratio and vortex diameter in case of cyclones, it is judicious to select the optimum cut points of different gravity separators so that the combined cleans from the different washers produce the desired products. •

*Principle of composite washing—Optimization strategy:* In a washery having feed from multiple sources with different washability characteristics, the coals are mixed and crushed to the desired size and the crushed products are screened and different size fractions are fed to different gravity separators. To get the desired quality, e.g. the clean coal ash at 17% level there may be many different combinations of the individual clean that can produce the desired quality. However there remains an uncertainty as to which combinations produce the maximum yield. It has been established at CFRI and elsewhere, that to achieve the maximum yield at the desired quality the density of cuts of individual washers should be fixed so that the characteristic ash at the density of cuts of all the washers should be equal. This condition of optimization has a sound mathematical basis and the problem of setting the optimum density of cuts can be achieved through numerical techniques using the principle of constrained non-linear optimization.

The expertise to develop software's for optimizing washing of composite coals by numerical techniques (Ref. International Journal of Mineral Processing, 42, 147, 1994). It is heartening to note that no other country in the world could develop such software's for efficient washing of 'difficult-to-wash' coals. Now it is time to incorporate such software and hardware in all the existing and future coal washeries for best utilization of our coal resources.

## **9.5 Characteristic Ash**

In the M curve, tangents to the point corresponding to the cumulative ash value of any specific cleans at a separation density, say  $d$ , when normalized through zero point on the ordinate, intersects the abscissa. The point of intersection gives the highest ash content in the cleans or the lowest ash content in the rejects. This ash value is a characteristic property of a coal, in the sense that any fraction of the coal having the density lower than the density  $d$ , does not contain any particle with higher ash value.

This implies the boundary ash content between the incremental floats and sinks at the separating density and is numerically the same as the determined-ash content of the fractional floats in the relative density range  $d \pm \Delta d$ . A plot of these fractional ash values against the mid-density values would give a straight line (Fig. 2.4) thus depicting the maximum ash of the particles ( $A_{max}$ ) upto the relative density  $d$  with a relation

$$A_{max} = k_1 d - k_2$$

$$\text{or } A_{max} = k_1 (d - d_0),$$

where  $d_0 (=k_2/k_1)$  i.e., 'Zero-ash density' is the theoretical density of pure coal free from any mineral matter. The value of  $d_0$  is primarily dependent on the rank and maturity of the coal and the value of slope is generally dependent upon the association of mineral matter of different densities with coal.

The concept may be utilized as an indirect means of estimating the maturity of coal, particularly in conjunction with the reflectance data. As the value of  $d_0$  is supposed to be higher for mature coking coal in comparison to the non-coking ones and slope value remains constant for a given-coal seam, a second check to the determined ash values in the washability data can be done prior to the simulation method.

The characteristic ash value in the cumulative ash range of 17-18% for Indian coals (equivalent cumulative ash in case of European/American coals will be 8-10%) falls in the vicinity of 25-30% and may be taken as the maximum allowable ash value corresponding to the desired reactivities content that would give a coke of specific quality (vide Chapter 3).

Similarly, a limiting value of 35-45% characteristic ash for a non-coking coal that might show good combustion property may be worked out from this and the reactivities curves. It is further interesting to note that the cumulative ash content at the cleans can be as high as 28-30% for Indian power coals, whereas for European/American coals it is as low as 12-15%.

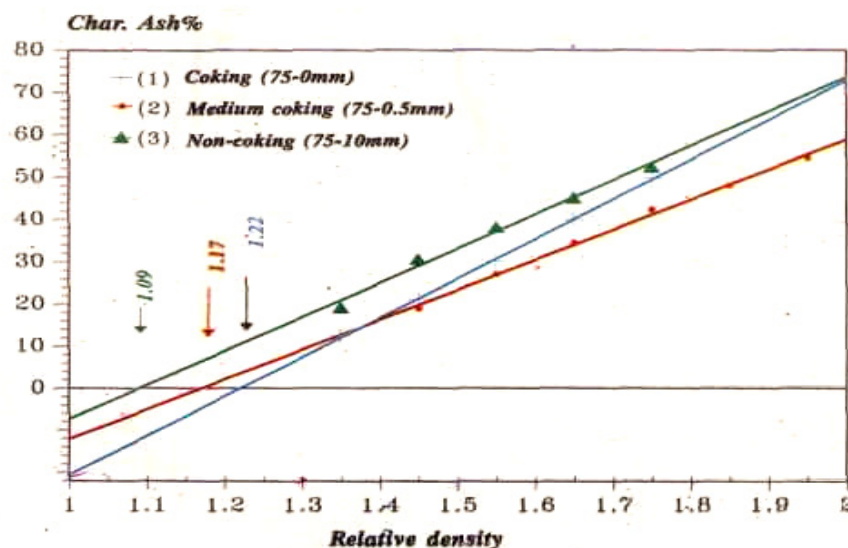


Figure - Characteristic Ash Vs Relative Density Curve

The concept of this characteristic ash may further be utilized in predicting the optimal cut points of different washers treating different coals or different screened fractions of a single or blended coals. For example, if two washers are treating two screened fractions of a feed with the objective of producing the total cleans at a desired ash level; there can be several number of different combinations of the separation densities for achieving the same. But, to get the maximum yield of desired cleans, the density of cuts are so chosen that the characteristic ash value at the respective density of cuts remain the same.

Similarly, this concept of equalization of characteristic ash content among coals of different origins/sources may be adopted for global optimization.

## 10.0 Quality Monitoring of Coal

Quality of coal plays an important role in all its utilization activities. Depending on the ultimate objective, be it steel making or power generation, the feed coal must satisfy same desired quality parameters. Coals being a highly heterogeneous substance in terms of inorganic or organic constituents, exhibits wide variability with respect to size and chemical compositions of the particles. Growing quality consciousness on the part of both suppliers and customers has made quality monitoring an integral part of the industrial activities. Proper quality maintaining of coal involves implementation of standardized procedures for sampling, sample

preparation and analysis to provide a reliable estimates of the desired quality parameters at minimum variance.

Detailed documented procedures are laid down in National International standards for executing the job representative sampling pertaining to different method of sampling. However, due to time, technical and other economic constrains strict implementation of the procedures are difficult to practice for routine monitoring job. As a result there are always a tag of war between producer and users.

### **10.1 Sampling of Washed Coal (SAIL/BCCL/CFRI)**

High ash coking coals are washed before despatching cleans to steel plants. Most of the coking coal washeries in eastern region have automatic mechanical samplers. Samples are drawn from the automatic samplers and results are averaged on the railway rank basis. The collected samples are reduced by offline mechanical and/or manual means to produce the final test samples.

In spite of coming sampling through unloading ends. To resolve the issue CFRI was involved as are independent third party implemented for a period of two and half year i.e. from Dec'1996 to March'1999, particularly eliminating any controversy between BCCL and SAIL economy of the both the seller and customer.

The sampling scheme designed by CFRI, if followed with all seriousness by the representatives of the two parties can significantly reduced the discrepancies in the results at both ends. If results are to be given as an average of those at the two ends, then identical means of sampling and preparation are to be adopted. Results obtained from automatic sampler at one end cannot be compared with those of manual sampling the other end, as the latter will definitely be affected by human discretions.

### **10.2 Standardization of the Procedure of Sampling and Analysis (NTPC)**

Normally the sampling methodology that is being implemented conforms to one of the standard procedures like **BIS/ISO/ASTM**, etc. However, very often due to technical, time and cost constraints strict implementation of the standard procedures becomes difficult and in such cases simplified sampling method is implemented with the mutual consent of the different stakeholders. The pertinent



point that needs to be looked into is that the fundamental principles of sampling are not compromised and more importantly the **precision** of the measurement.

It is an established fact that more than the **80% of the total variance** arising during the different stages of sample collection, preparation and analysis come from the sampling procedures. Sampling variance is a function of fuel variability so that the same number of increments, sub-lots, preparation and testing errors can yield different precision with fuels of wide variability. Efforts are to be made to reduce the sampling variance as far as practicable and both the sellers and purchasers need to agree to the limit that is practically achievable.

Quality monitoring of coal for any end use requires proper implementation of standards sampling, preparation and testing procedures. The design of sampling scheme may vary depending on the purpose like process control, evaluation of plant performance, commercial transaction etc. for which the sampling methodology has to be executed. Normally the sampling methodology that is being implemented conforms to some standard procedure like BIS/ISO/ASTMs etc. However, very often due to technical time and cost constraints start implementation of the standard procedures becomes difficult and in such cases simplified sampling method is implemented with the mutual consent of the different stakeholders. The pertinent points that needs to be looked into is that the fundamental principles of sampling are not compromised, followed by execution of standard designed test to check the effect of the modification on the value of the desired quality parameters.

National Thermal Power Corporation (NTPC) is one of the largest consumers of non-coking coals in India. Semi mechanical/manual means from the loaded wagon. In this connection NTPC has identified few Thermal Power Station for methodology of sampling and awarded job to CFRI. CFRI was engaged to prepared methods for wagon top sampling and tested.

The objective of the study was to standardize the procedure for sample collection and preparation for general analysis and total moisture determinations. The study involves a critical assessment of the sampling procedure of such large sized coal from wagon top conveyor belt, and chute, following the existing methodology and identification of the scope of improving upon the procedures to yield results with lesser variance.



## Recommendations

For all the NTPC where the standardization of sampling was studied methodology was proposed for conveyor belt, chute and wagon top sampling. The Thermal Power Station selected for above studies:

Vindychal Super Thermal Power Plant

Farakka Super Thermal Power Station (Mechanical and Wagon Sampling)

Kahalgaon Super Thermal Power Station

Talchar Super Thermal Power Plant

Talchar Thermal Power Station

### **10.3 Sampling and analysis of Imported Coal unloaded at the port ends - (SAIL & IMFA)**

At a time when the steel producers are working at full capacity to cater to the ballooning demand, the Australian vendors have enforced a 'force majeure' clause to reduce supplies of coking coal to India. This has led to the increase of Coking coal prices by nearly four-fold in the last one year. With rising internal consumption and the mining industry plagued by accidents, China is no longer viewed as a credible supplier, creating a crisis for the industry. India imports about 15 million tonnes of high grade coking coal, which is primarily used by the State-owned producers. Steel Authority of India Limited, imports coking coal from Australia, S. Africa, U.S.A, Canada, China on a regular basis. The Cargo is usually unloaded at the ports of Vizag, Paradeep & Haldia. Systematic sampling and characterization of the imported coal will not only help in judging its quality but also to see whether the supplier is keeping all the contract parameters in its right position.

Steel Authority of India is importing coals for use as a blend for coke making. The company entrusted CFRI to carry out sampling of imported coal while the coal is being unloaded at Vizag, Paradeep & Haldia ports and conduct detailed characterization tests as per their requirement. The main objective is to characterize the imported coals after collecting the representative samples when unloaded at the port end and preparing as per the standard procedures.

The Coal Preparation Division has carried out the sampling and analysis of imported coal for over forty (40) vessels since the year 2000 and the activity is still continuing and is one of the major source of ECF.

Indian Metals and Ferro Alloys, Therubali – Orissa, are importing coal for using it directly as a reductant in their furnace for the manufacture of Ferro silicon. The company entrusted CFRI to carry out sampling of imported coal while the coal is being unloaded at Vizag port and conduct detailed characterization tests for finding out the coking propensity of the imported coal.

The Coal Preparation Division has carried out the sampling and analysis of imported coal for Four (4) vessels since the year 2002.

## **11.0 Quality Enhancement of Coal for its Efficient Utilization (Taskforce Project) – X Five Year Plan**

Suitable R&D schemes have been identified under a broad theme of 'Quality Upgradation of coal for its efficient utilization'. The theme can get an implementable shape only with wide participation of expertise of R&D institutions, industries and proactive and supportive policy decisions. CSIR, with its chain of laboratories is possibly the only organization to undertake this massive work and provide the scientific and technological solutions to these challenging problems. CFRI, the nodal laboratory has impressive infrastructural facilities under a single roof, including coal washing pilot plants and test rigs for combustion to address the various aspects of coal/flyash characterization, environmental assessment and coal washing. Other laboratories like RRL, Bhubneswar, RRL Bhopal and NML also have the requisite facilities and expertise to deal with the relevant issues on characterization and coal beneficiation. *Networking of these laboratories will lead to the formation of a large number of expert professionals, who with the backup support of the collective infrastructure can produce deliverables in a scale, both in time and magnitude, which cannot be achieved through individual efforts in isolation*

The Indian coal seams formed during lower Gondwana period being categorized as difficult-to-wash due to its typical mineral matter association, which is responsible for high amount of near gravity material (NGM) making the

selection of beneficiation process unique and difficult. Planned R&D initiatives during the fifties & sixties of the last millennium got translated into development of suitable flow sheets for treating such coals and led to the installation of coking coal washeries, primarily catering to the need of the steel sector. However, with the gradual depletion of superior quality coals coupled with the advent of mechanization in mining and material handling methods, the limitations of the existing facilities in producing the optimum outputs, both in quantity and quality, came into surface. These constraints led to some serious thinking among the policy makers in meeting the newer challenges of coal washing and several Expert Committees were formed to review the situation and identify the means to circumvent the daunting problems.

The TIFAC report on Coal Beneficiation (cf. Project proposal on ECONOMIC REDUCTION OF ASH IN COAL WHILE CONSERVING SCARCE COKING COAL RESOURCES, TIFAC Programme/DST/CSIR Labs, 1990) gives a comprehensive appraisal of the situation and gives the direction of further research on the related areas. The report touched upon the problems of coking coal washing and means to improve upon the same with the indications of the need for noncoking coal washing in the future. The recommendations, however, failed to get implemented and the situation in the coking coal washing sector got further worsen with the production of Low Volatile Coals whose washing characteristics were much inferior to the coals for which the washeries were designed.

The unfulfilled tasks identified in TIFAC and the newer challenges posed by the LVC coals of Jharia & Bokaro coalfields call for a comprehensive well directed R&D approach for optimal utilization of the large reserves of difficult to wash indigenous coking coals. The under utilization of the existing washing facilities without appropriate renovation or retrofitting, the increased production of inferior raw coals, the widening gap between the demand & supply of quality coals are the factors that contribute to the increasing trend of coal import. Introduction of newer techniques/ equipment, evolving innovative beneficiation strategies using latest tools of modeling and online control systems warrant focused R&D activities in this area.

A newer dimension in the area of coal washing has come up, with the MoEF restrictions on the utilization of non-coking coals for power generation. To meet

the stipulations a large quantity of high ash noncoking coals need to be upgraded to 34% ash levels either through blending or beneficiation. The uncertainties associated with blending of highly heterogeneous coals and their impact on the different sub-processes of the 'coal energy chain', identification of the optimal level of cleaning for increased efficiency of power generation need to be effectively addressed. Very limited exercise, if any, have been done for Indian coals and an integrated approach starting from mining, cleaning and power generation is required with strong supportive R&D backup to meet the growing need of quality fuel for power, cement, sponge iron and foundry sectors. The major objectives of the project are:

- a) To identify the optimum beneficiation strategy of non-coking coals to meet the needs of different end users.
- b) To assess the effect of blending of coals, (indigenous & imported) on efficiency of power generation and evolving strategies of blending of dissimilar coals.
- c) To develop suitable beneficiation scheme for optimal utilization of the large reserves of Low Volatile Coking (LVC) coals from western part of Jharia and Bokaro coalfields
- d) Enhancing the recovery of cleans from fine coking coal using the optimum combination of different fine coal treatment processes
- e) Development of suitable reagents for treating non coking fines by flotation process.
- f) Application of different physical processes for treating non coking coal fines.

The taskforce project has been taken up in June'2004 and the work is going in steady progress has per the work plan. It is expected by March'2007 all the deliverables as highlighted in the project may be achieved.

## **12.0 Infrastructural Facilities**

### **12.1 Laboratory/Bench-Scale Facilities**

1. Different types of laboratory crushers (Single roll, Double roll, Jaw, Gyratory etc.,)
2. Grinding Mills

3. Sieve Shaker
4. Screens
5. Denver Flootation Cells
6. Oil Agglomeration Cell
7. Magnetic Separator
8. Water only Cyclone Test Rig
9. Heavy Medium Cyclone Unit
10. Ash Furnace
11. Moisture Oven
12. Balance
13. Moisture Balance
14. Davis Tube
15. Vibratory Sieve Shaker
16. Wet Tumbler
17. Leaf Filter
18. Centrifuge
19. Selective Drop Breakage Unit
20. Dryer

## **12.2 Pilot Plant Facilities**

### ***12.2.1 Large coal washing pilot plant, 40 tph capacity comprising of***

- a) Jig : Capacity 20 tph
- b) Heavy Medium Drum Separator : Capacity 20 tph

Fully equipped with transfer units, screens, crushers, PL controlled instruments to operate the plant in 8 different circuits.

### ***12.2.2 Modern Fine Coal Treatment Pilot Plant with on line instruments and PL control comprising of the following facilities/circuits :***

- i) Experimental Batch Rotary Breaker, 3.5m dia.
- ii) Primary Crushers x 100/50 mm (in closed circuit) operating on different principles
  - a. Shear
  - b. Impact
  - c. Compression
- iii) Closed Circuit Secondary Crushing House to achieve product size of 13/6/3 mm



- iv) Size classification circuit equipped with 3 mm and 0.5 mm wedge wire screens with provision of controlled spray water.
- v) 200 mm HM cyclone Unit complete with sump, pump, sieve bend and D.R. screen.
- vi) Spiral, 1 m dia, circuit with provision of size classification.
- vii) A battery of Flotation Cells with de-watering devices.

The above facility is also utilized to carryout tests on Oleo flotation process with the incorporation of centrifuge etc. for dewatering.

- viii) Oil agglomeration Unit to produce ultimate cleans from ultrafine coal, A complete unit with closed circuit continuous wet grinding and classification

### **12.3 Services offered:**

#### **12.3.1 Design of coal washeries**

Activities	Sample required	Time	Fees, Rs.	Deliverables
• F& S test	3-10t	1 Month	1.3-2.0 lakhs	Washability results
• Computer Simulation	(Data from I)	15 days	2.0 lakhs	Optimum flow sheet
• Pilot Plant testing	50t	2-3 Months	10-15 lakhs	Innovative Flowsheet Specs. of Washers, Crusher, Screen, etc., Techno-economics

#### **12.3.2 Design and installation of coal slurry flotation Plant, 10-20 tph**

Activities	Sample required	Time	Fees Rs.	Deliverables
Lab. Flotation test	25 kg	15 days	5000.00/ test	Floatability
Pilot Plant testing	1 t	1-2 Months	1.0 lakh	Techno-economical Viability
Technology Transfer/ Signing of Agreement	-	15 days	1-1.5 lakhs	Process know-how Design/Drawing

### **12.3.3 Sampling and Analysis of Imported Coal**

Activities	Sample required	Time	Fees Rs.	Deliverables
Supervision of Sampling at Port		15 days (till the vessel is unloaded) 20 days	4 to 5 lakhs	Test Report (Characterization data)
Characterization	30 kgs			

## **13.0 Conclusions and Future Programme**

Some major contributions to science and technology of coal preparation, which have been highlighted in this paper are a result of extensive R & D studies on various aspects of beneficiation of coking and non-coking coals, development of newer technologies etc., carried out at CFRI.

The Coal Preparation Scientists have to tackle a formidable task in the preparation of the difficult washing Indian coking coals which do not respond favorably to beneficiation by conventional techniques. Develop flow scheme for beneficiation of non-coking coals for different end users.

Keeping in view, the demand of quality coal for various end users the future R & D programmes identified are as follows:

- Dry beneficiation studies of non-coking coals of India for power plants.
- Development of 3/2 product schemes to produce clean coal for sponge iron and cement industries by complete utilization.
- Identifying the coals suitable for sponge iron industry and Increase the resource base for the above industries by suitable beneficiation.
- Technology to establish small capacity washeries for sponge iron and cement industry.
- Optimization and simulation of existing coking coal washeries to improve yield.
- Modernize the existing coking coal washeries by introducing advance techniques.
- Utilization of washery rejects for different non core sectors.

- Development of suitable reagents for beneficiation of non-coking coal fines and oxidized coals.
- Develop suitable beneficiation scheme for optimal utilization of the large reserves of Low Volatile Coking (LVC) coals from Western part of Jharia and Bokaro coalfields.
- Develop suitable technologies for the recovery of cleans from fine coking coal using the optimum combination of different fine coal treatment processes.



## ANNEXURE

### 1. List of Publications (1990 – 2006)

1. Application of HM Cyclone and oil agglomeration for the beneficiation of difficult-to-wash coking coals  
Int. Seminar on Fine particle beneficiation and Environmental Effects, held on 11<sup>th</sup> Dec 1990 at N.Delhi  
R.Haque  
D.D.Haldar
2. Beneficiation of Middlings for Recovery of Cleans for Coke Making by Froth Flotation *Symp- on Coal Utilization : Trends and Challenges, CFRI, Dhanbati, 13-14 Dec. (1989) FuelSci. Technol 10 (1 & 2), 11(1991)*  
Shantia, K..K. Sengupla, P.K. Kabiraj, S Mma,A.N. Daschowdhury, K.P. Konar.B.B.
3. Development of Fine Coal Flotation for Future *Symp. on Coal Utilization : Trends and Challenges, CFRI, Dhanbad, 13-14 Dec. (1989) Fuel Sci. Technol. 10 (1 & 2), 35 (1991)*  
Sen,K. Chakraborty, D.K. Roy, S.K.
4. Oleo-Flotation Pilot Plant Studies for Upgrading and Dewatering on Small and Fine Coals *Symp. on Coal Utilization : Trends and Challenges, CFRI, Dhanbad, 13-14 Dec. (1989) Fuel Sci. Technol. 10 (1 & 2), 21 (1991)*  
Chattopadhyay, J. Ghosh, B.IC Shanna, K.K. Ghosh, S. Haque,R.
5. Studies on Oil Agglomeration of Coal with Anthracene Oil as Bridging Liquid *Symp. on Coal Utilization : Trends and Challenges, CFRI, Dhanbad, 13-14 Dec. (1989) Fuel Sci. Technol. 10 (1 & 2), 17(1991)*  
Jha,G.S. Chattopadhyay, PC Haldar D.D. , B B. Konar, B.B Ghosh, S.
6. Effect of Some Operating Variables on the Absorption of Conditioning Oil on Coal Particle *Proc. HthlIntern. CoalPrepn. Cong.(ICPC), Tokyo, 22-25Oct., (1990)*  
Halder, D.D Jha,G.S. Chattopadhyav PC Konai, B.B. Ghose, S.
7. Dimensional analysis of the conditioning system in the oil agglomeration process  
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Power Coal Division

# **R&D studies in the areas of combustion of coals and related issues**

## **I. Major activities of the past**

### **1. Fluidized Bed Combustion**

The main objective for installation a 60 kw FBC unit was to undertake Research and Development work on fluidized bed combustion (FBC) for assessing the suitability of the technology using low grade Indian coals. Extensive investigations were conducted in the unit with wide range of coals ranging from high sulphur lignite to high ash washery rejects.

The studies provided the relevant data for developing basic design and process data for building up industrial scale boilers. Based on data generated from 60 kw unit, 2t/hr (Steam equivalent) test boiler was designed at CFRI in collaboration with BHEL.

### **2. 2t/hr (Steam equivalent) test boiler**

Realizing the need for development of FBC boilers using low-grade Indian coals to obtain basic design and process data for building up industrial scale boilers, 2t/hr (Steam equivalent) was installed at CFRI. In this unit the suitability of FBC technology for generation of heat, steam and power was established.

The experimental investigations in the test boiler have established that high ash low-grade Indian coals can be used efficiently in Industrial type FBC boilers for generation of process steam. Subsequent to commissioning and testing of the 2t/hr FBC boiler at CFRI, a large number of industrial type FBC boilers based on high ash non-coking coals have been installed and operated in different areas of the country.

### **3. Fluidized Bed hot air generator (75kg coal/hr)**

Fluidized Bed hot air generator was developed to explore the possibilities of supplying hot air for different purposes by a number of Industries especially for drying of tea, paddy, grains etc. using low grade coal.

The data generated from hot air generator is used for designing hot air generator in different industries other than steam generation.

### **4. Inert Gas Generators**

Mine fires in the coalfields is a serious problem in our country in terms of loss of scant resources of coal, safety and economic health of mining operations. Several organizations have tried different methods to control the fires and have met so far with marginal success. The objective of the Inert gas generator installed at CFRI for carrying studies on pilot plant capable of generating 500 Nm<sup>3</sup>/hr inert gas by FBC of different quantities of coal for combating mine fires.

The pilot plant for generation of cheap inert gas of specified quality suitable for combating mine fires designed, installed and commissioned at CFRI is capable of producing 500 Nm<sup>3</sup>/hr inert gas at ambient temperature. The plant of this capacity is suitable for commercial exploitation, hence needs no scaling up. The pilot plant is flexible in respect of quality and source of coal.

#### **5. Coal Water Slurry (CWS)**

The CWS technology developed at CFRI is having two parts. In the first part high concentration (about 70% coal loading) coal water slurry was prepared and in the 2<sup>nd</sup> part the CWS was burnt in 30 kg/hr combustion test Rig. The CWS technology was developed at CFRI for replacement of fuel oil used in the boiler for initial starting and power load variations. The fuel oil requirement is about 1% of the total fuel consumption.

With the increasing demand for importing fuel oil and its rising price there is a great need to find an indigenous alternative. It is observed that the pay back period for 1.2t/hr capacity CWS plant is about 4 years, which makes this process attractive with respect to profitability as well as indigenous substitute for fuel oil.

## II. Present Study

### 1. Combustion behaviour of Indian coals

For increasing the efficiency of the existing pulverized coal (PC) power plant a through understanding of the effect of coal quality parameters on various aspects of power generation is necessary. Limitations exist with the traditional parameters for realistic assessment of combustion behaviour. Other relevant parameters like coal maceral, coal mineralogy, surface characteristics need to be considered for better understanding of the behaviour of different coal in power generation.

The present activities are centered around the study of the combustion behaviour of coal in different scale. Detailed characterization in terms of chemical, physical, petrography, minerals are being done followed by evaluation of combustion behaviour in laboratory scale DTA/TGA, bench scale Drop Tube Furnace (DTF) and pilot scale Fuel Evaluation Test Facility (FETF). Three major projects have been undertaken in this area.

- Influence of rank, petrographic composition, level of cleaning etc. on combustion behaviour
- Combustion studies of high ash coal
- Impact of coal quality for clean power generation

The investigations are focused on identifying the key quality parameters that affect the combustion efficiencies, slagging propensity and emissions from Indian coals. Laboratory scale TGA studies help to measure the reactivity of coals and their kinetic data. Bench scale Drop Tube Furnace under a laminar flow provides the data on the behaviour of coals under high heating rates. The Fuel evaluation Test Facility with a turbulent flow closely simulates the data of an utility boiler has been used to study the burning efficiency of coals under different operating conditions along with slagging and fouling behaviour of different coals. The data generated from such studies are being used to make a relative ranking of different coals towards combustion.

To study the effect of rank and petrographic makeup of the coals on the combustion detailed investigations are being made on the macerals distribution, microlithotypes and the role of inertinites. These petrographic parameters in combination with the chemical parameters like fuel ratio, GCV have been used to correlate with the burning behaviour at different scale of operation. To relate the influence of petrographic components on the morphology of char, partially burnt samples have been drawn from the DTF and FETF at different residence times. These char have been microscopically studied to identify the nature and type of chars formed and linked with the characteristics of the parent coals.

Extensive studies have revealed that inertinites do play a significant role in combustion. In some cases, contrary to the traditional concept, the inertinite

reactivity can be more than the vitrinites. Studies are being carried to identify and quantify the type and nature of the inertinites which in combination with the traditional reactives like vitrinite and liptinite can be used to explain the behaviour of inert rich Indian coals.

To assess the effect of both macerals and minerals on the combustion behaviour studies are being made on different specific gravity fractions and at different level of cleaning. The TGA behaviour of the density fractions do not always follow the trend observed in DTF studies. follow the petrographic parameters but this trend is not reflected in the DTF studies. Attempts are being made to quantify the low rank inertinites which are likely to affect this behaviour.

## **2. Combustion studies of coal blends:**

Blending of coals in order to meet a number of specific requirements is common practice in utility applications. This practice has generally been motivated by the desire of a utility to enhance the quality of coals in terms of their milling behavior, combustion properties and emission control.

However the traditional quality parameters used for power coals like CV, ash content, moisture, VM etc., are not sufficient to assess the actual combustion behaviour of the blended coals. The blending of coals requires a through understanding of the relationship between the different aspects of combustion behaviour and the constitutional, mineralogical and petrographic make-up of the coal. This becomes more important while exploring the option of blending coals of different origin such as blending of indigenous coal with imported coal with widely varying constituents.

The utilization characteristics of blends of two or more coals may not be proportional to the ratio of the individual coals in the blends; particularly when the component coals differ significantly in petrographic and/ or ash properties. This is due to the non-linear nature of the processes, which the coal undergoes during pulverizing and combustion in pf-fired plants and also due to the interactions between some of the processes. Hence in dealing with operational and environmental issues relating to blends, it is necessary to carry out extensive investigation on grinding characteristics, the combustion characteristics and the environmental impact potential for the individual as well as different blends. On the basis of individual coal characteristics and different blend characteristics, optimum blending proportion have to be formulated so that the product coal meet the desired quality specification for utilization in thermal power plants.

The utilization of coal blends can significantly affect several areas of boiler operation, which impact overall power plant performance and generating cost. Normal practice for determining the overall quality of blended coals is to use the weighted average of the determined values of the individual coals in the blend. It is well known that properties like Hardgrove, Grindability Index and

Ash Fusion Temperature are not linear functions of the properties of the individual coals. It is the interaction between components of the individual coals in the blend that makes the evaluation inherently complex.

In pulverized coal fired boiler furnaces, the incidence of slagging, fouling and fireside deposit formation is a common phenomenon. The problem of boiler deposition is governed by a number of parameters e.g. furnace atmosphere during combustion, furnace temperature profile, nature and distribution of minerals present in coal, size distribution of pulverized feed coal, boiler design, type of furnace, etc. Various empirical indices based on the analysis and fusion temperature of ash are in frequent use to assess the propensity of slagging and fouling behavior. viz. Silica ratio, base-acid ratio, slagging index, fouling index etc. These relations are applicable with good approximation for single coal source or mixture of coals of similar geological origin. A blend of dissimilar coals with varying mineralogical chemistry and distribution pattern, the linear combination of the empirical indices are unlikely to be applicable to predict boiler deposit problems. To predict the slagging and fouling propensity of the blended coals in the boiler, it is necessary to characterize ash of the blended coals produced under representative conditions.

The combustion of coal results in the release of a number of compounds into the atmosphere, the most important being the particulate and the oxides of sulphur, nitrogen and carbon. The environmental impact of the hazardous impurities (e.g. sulphur, nitrogen & hazardous trace metals) present in the coals and their blends have to be thoroughly assessed to give guidelines for reaching at suitable blending proportions to better assess the risks and limit the possible environmental degradation and impact on health.

Studies are being made in TGA, DTF, FETF on blends of different coals, both indigenous and imported. Blending of coals of different origins, of different rank and high ash & low ash coals are being extensively studied. Binary blends of different proportions are being studied to observe their effect on grindability, combustion behaviour and slagging propensity. Non additive behaviour was observed in case of the burning behaviour in both laboratory and bench scale apparatus. The burnout is not only dependent on the coal types, their petrographic mix but also the proportions of individual components. Attempts are being made to identify some of the key parameters for optimizing the coal blends.

### **III. GHG Inventory and estimation of GHG emission from major sources**

#### **Background**

The gradual increase of carbon dioxide in the atmosphere is primarily attributed to two types of human activities: use of fossil fuel and land use including deforestation and biomass burning. Coal combustion contributes relatively more amount of carbon dioxide in the atmosphere per unit of heat energy than from the combustion of other fossil fuels. Emission of carbon dioxide flux in the atmosphere occurs when fossil fuels are oxidized by combustion and depends on the energy and carbon content of the fuels. The share of coal is about 35% in the total global CO<sub>2</sub> emissions. The consumption of coal will increase significantly in the foreseeing future to meet the growing demand of energy from the developing nations like China and India. With increasing international concern on the harmful emissions sincere efforts have been initiated to reduce the emissions from the entire 'coal-energy' chain. The United Nations Framework Conventions on Climate Change (UNFCCC), because of a growing concern over the possible consequences of global warming seeks to stabilize atmospheric GHG concentrations at levels that would prevent dangerous anthropogenic interference with global climate, which may be caused in part by increases in atmospheric carbon dioxide.

India is a party to United Nations Framework Convention on Climate Change (UNFCCC). One of the commitments of the developing country parties under the UNFCCC is to furnish an Indian National Communication (NATCOM) providing information on the inventories of Greenhouse Gases (GHG) of anthropogenic origin by sources and removal of sinks and a general description of steps taken or envisaged to implement the convention.

The Ministry of Environment & Forests is the nodal ministry for the subject of climate change and has initiated activities relating to preparation of NATCOM. One of the activities identified by the National Steering Committee and a Technical Advisory Committee is to prepare a National GHG Inventory. The job of preparing this inventory for the period 1990-2001 was undertaken by CFRI. Besides that the uncertainties associated with the estimation of GHG emissions from power, steel and cement plants was also carried by CFRI

#### **GHG Inventory**

The national greenhouse gas (GHG) emission inventories provide a tool for assessing progress in estimating emission levels and also provide a crucial reference point for other assessment tools, like climate change indicators and mitigation strategies. The national average of emission coefficients from the key sectors though available from the national inventory data may not reflect the regional and site specific variations. Estimation of CO<sub>2</sub> emission with the assumption of a single emission factor of a particular coal category and average oxidation factor does not reflect the variance associated with fuel



quality from different sources and the combustion technologies/vintage of individual power plants. The major sources of uncertainties arise from the choice of Emission Factors of different coal types, right from lignite to anthracite. The other source of uncertainty is lack of quantified data of different types of coal within a broad category being used in the non core sectors. To have a credible and comprehensive inventories of greenhouse gas emissions the uncertainties associated in preparation of national inventories need to be reduced to the possible extent.

Emission estimates from the combustion of solid fuels like coal and lignite require three parameters viz. Carbon Emission Factor (CEF), Net Calorific Value (NCV, kcal/kg) and carbon stored/unoxidised during its utilization. For preparation of National inventory the NCV value is required to convert the apparent consumption data by weight to heat units. This in conjunction with the CEF value provides the amount of CO<sub>2</sub> emissions from different coal types. The Carbon Emission Factor can be expressed in two different ways: i) the amount of CO<sub>2</sub> emission per unit weight of the fuel, ii) the amount of emission per unit heat value of the fuel, which could be either Gross Calorific Value (GCV) /Higher Heat Value (HHV) or Net calorific Value (NCV)/ Lower Heat Value (LHV). As the heat value, either the GCV or NCV is predominantly a function of the carbon content, the latter definition with NCV as the unit of heat value has been accepted as the International practice.

The national inventory for base year 1990 was prepared using the default CEF values and NCV values of 20.5 MJ/kg and 17.6 MJ/kg for coking and other bituminous coals respectively. Keeping in view the wide variation in the ash, moisture content and the petrographic makeup of Indian coals, scope exists for continuous refinement of the default data for Indian coals. Analyzing large volume of authenticated data on Indian coals and lignites can minimize the uncertainties associated with the default IPCC values of the two main parameters for different category of coals/lignites.

The refinement of the CEF and NCV' s would require estimation of the said parameters from a large volume of data set covering the wide variability of the quality parameters of various coal types from different regions. The data sets considered for estimation have been obtained from the technical reports of Central Fuel Research Institute (CFRI) and were confined to those generated since the last two decades.

The relevant data required were the Proximate, Ultimate, Gross Calorific Values (kcal/kg) of the different coals. The entire data were divided into three categories i.e., lignite, non-coking and coking coal on the basis of the carbon value on dry mineral matter free basis (dmmf), moisture and Gross Calorific Value. To ensure integrity of the data the experimental GCV's were cross checked with the values obtained from the elementary data using the formula

proposed by Mazumdar . The data for which the difference <sup>1</sup> between the experimental and calculated value was more than 150 kcal/kg were not considered. Similar approach was used by Quick et al while estimating CEF of US coals.

## **Estimation of NCV and CEF**

### **Lignite**

The total data used in the study is about 90 which covers the lignites produced from Neyveli Lignite Corporation (NLC).

The range of moisture of NLC lignites is between 45-55%. For estimating the parameters the mid value of 50% was used. The data of the relevant chemical parameters were converted to the desired moisture level and the estimates made.

### **Noncoking coal**

The data used for the study covers the major sources of noncoking coals in India. The total number of data used was 2600. The data set was first categorised into different grades as per the existing grading system in terms of the Useful Heat Value (UHV). This categorisation is necessary because to arrive at a mean value of the said parameters for non coking coal, with wide variability, one has to know the production pattern of each category. The production figures have to be linked with the estimation study because of wide variation in the NCV values from grade A to grade G

For each grade mean values of the respective parameters were obtained. While estimating the parameters only the mean values of particular grade have been considered. The variation of NCV within a grade can vary within a range 3-4 unit. This is due to the fact that coals of different region having the same (ash+moisture) will lie in the same grade but can have significantly different GCV values primarily because of the difference in <sup>2</sup> maturity and petrographic make up. The variation of CEF within a grade is insignificant and between the grades is only 0.5 units.

### **Coking coal**

The total data sampled for the study is 450. The data were categorized into the various grades and the mean values of NCV & CEF of each grade were used along with the production data of that grade to compute the weighted mean of the said parameters.

### 4.3.3 Results

	IPCC Default		Earlier values		Present values	
	NCV	CEF	NCV	CEF	NCV	CEF
	TJ/Kt	t C/TJ	TJ/Kt	t C/TJ	TJ/Kt	t C/TJ
Coking coal	19.98	25.8	20.5	25.8	24.18+3%	25.53
Non-coking coal	19.98	25.8	17.6	25.8	19.63+4%	26.13
Lignite	9.8	27.6			9.69+4%	28.95

The estimated NCV and CEF values of Indian coals and lignites have been used to prepare the national inventory. The emissions trend have been estimated from the apparent consumptions of the fossil fuels, taking into consideration the production, import, export and stock change. The % increase of CO<sub>2</sub> emission from liquid fuels, solid fuels and gaseous fuels from 1990 to 1999 are 34.3%, 33.5% and 55.9% respectively. The estimated increase of emission from solid fuel in 2011-12 is about to be 1210(Tg) compared to 637(Tg) in 1999.

The pattern of emission however reflects significant regional variations. The total emission is highest in Maharashtra (112 Tg) whereas the individual contribution from the North Eastern States and Himachal Pradesh is less than 1 Tg. Coal based emission is highest in Madhya Pradesh followed by Uttar Pradesh, Maharashtra, Andhra Pradesh, Bihar and west Bengal. The use of gaseous fuels is primarily concentrated in Maharashtra, Gujarat and Assam.

### GHG emission measurements from coal combustion in selected industries

Coal utilization is expected to increase significantly in the foreseeing future to meet the growing energy needs of the major sectors like power, steel, cement and other coal dependent industrial processes. These three sectors contribute almost half of the India's Carbon dioxide emission and majority of it comes from Coal and lignite combustion. The increased emissions from the solid fuel combustion thus call for accurate estimate of sector wise emission data from this key source category not merely to improve the quality of the national GHG inventory but for providing meaningful scientific inputs towards development of different mitigation strategies through diverse routes of enhancing energy efficiency, use of different fuel mix and judicious exploitation of the renewable energy resources.

The uncertainty of emission estimate associated with the small sectors is more compared with that from the large sectors, like power. Emissions of GHG particularly CO<sub>2</sub> can be measured or estimated and the approach to be taken depends on the availability of emissions related data, cost of generating it and the accuracy needed for the inventory. In case of power plants reliable estimates of CO<sub>2</sub>, SO<sub>x</sub> per unit of fuel or kwh can be made by the traditional mass balance approach using the carbon, sulphur, heat value and the quantity of fuel. The NO<sub>x</sub> emission being a function of fuel and the excess air used, need to be estimated from direct measurements. In case of integrated

steel plants estimate of emissions per ton of hot metal (thm) may not be feasible from direct measurements on numerous stacks of different process units. Using the Emission Factor (EF) of different fuels used in the blast furnace and their rate of consumption per ton of hot metal would give a reliable estimate of the emissions. In cement plant the emission from fuel sources can be estimated from quality and quantity of the fuel input.

In general plant measurements provide the most rigorous data for estimating emissions. Considering the cost aspect, it may not be feasible to measure all sources and estimates have to be made on measurements made on well-identified sources taken to be the representative of the population. . However, using national figures of annual fuel consumption and the carbon content of that fuel for GHG emission, may give wide variations when compared with that of Utility-specific data. This is due to variation of CEF of different quality of Indian coals and wide variation in unoxidised carbon fraction, a fuel and technology dependent parameter, in different power plants.

Direct measurements have been carried in few plants of different capacity, both public and private owned, and comparative evaluation were made between the estimates made from mass balance and measurement.

#### ***Power plant –1***

The emission coefficients of CO<sub>2</sub> obtained by direct measurements and those calculated by actual CEF and IPCC CEF are 0.963, 1.013 and 1.304 kg/kwh respectively. The direct measurement value and estimated from actual coal quality matches well but that determined from IPCC default values give a significantly higher estimate. The coefficients of SO<sub>2</sub> from direct and estimated from fuel sulphur values are 0.004 and 0.006 kg/kwh respectively. The NO<sub>x</sub> coefficients obtained from direct measurement are 0.001 kg/kwh. This cannot be estimated from fuel Nitrogen value, as emission of oxides of Nitrogen is a combined effect of fuel quality and plant operating conditions. The emission coefficients in terms of mass/unit mass of coal for CO<sub>2</sub> from measurement and fuel quality are 1.418 and 1.423 kg/kg respectively. The same for SO<sub>2</sub> are 0.006 and 0.007 kg/kg. The carbon oxidation factor is more than 99%.

#### ***Power plant -2.***

The CO<sub>2</sub> emission coefficients for Unit IV obtained from direct measurement, calculated using actual CEF of the fuel used and that from IPCC default values are 1.155, 1.105 and 1.270 kg/kwh respectively. The corresponding values of the Unit V are 1.138, 1.085 and 1.254 kg/kwh. The SO<sub>x</sub> coefficients for Unit IV from measurement and estimated from fuel sulphur balance are 0.0032 and 0.0045 kg/kwh respectively. The corresponding figures for Unit V are 0.0036 and 0.0039. The NO<sub>x</sub> coefficients in Unit IV and V are 0.0012 kg/kwh. The coefficients expressed in kg/kg of coal basis for CO<sub>2</sub> and SO<sub>2</sub> are identical for both the plants. The measured and calculated CO<sub>2</sub> coefficients are 1.714 and 1.719 and that of SO<sub>2</sub> are 0.005 and 0.006 respectively. The carbon oxidation factor is more than 99% in both the Units.

Direct measurements have also been carried in some smaller utilities of capacity ranging from 60 MW to 120 MW. The average coefficients from power plants of different capacity is shown in

### **Steel plant**

In integrated steel plants having coke oven facility, combustion of solid as well as the gaseous fuel takes place in different units. Both the coke oven and blast furnace gases are used for heating purpose of different process units. Neither the stack monitoring from individual stacks of each process units would be possible nor the generated data could be linked with the fuel data. Besides, flux such as Limestone or Dolomite added in the process contributes to the emissions.

The Tier I method of IPCC has been used to estimate the emissions per unit of hot metal using both the default EF of the reducing agents and the actual EF estimated from the sample of reducing agents collected during the period of study. The factors related to stored carbon in the hot metal were considered in the estimation. The uncertainty associated with the emission factor of the reducing agents (coke & coal) has been addressed by analyzing the coal and coke quality. Moreover, from the available data the plant emissions arising out of the fluxes have also been estimated. The EF of different solid fuels was used to compute the emission coefficients kg/thm considering the plant data of several months.

The default Emission Factor for coal and coke are 2.5 and 3.1 t CO<sub>2</sub>/t reducing agent. The EF estimated from the actual coal and coke samples are 2.64 and 2.89 respectively. Since the plant is an integrated one having coke oven facility the Tier I calculation was carried out considering the coal as the reducing agent. The coal consumption included coal used in coke oven and also the amount directly charged in the blast furnace as CDI (Coal Dust Injection). Since about 6% of the coal charged in the coke oven are transformed to its by products through which the carbon is practically stored, the actual coal effectively combusted during the whole chain of steel making processes was taken as 94% of the coal charged. The mean CO<sub>2</sub> emission coefficients t CO<sub>2</sub>/t hot metal estimated was 2.23. This approach would however give an overestimate of the emission coefficients. In this method the carbon stored in metal was not considered.

For detailed estimates the total data set was used by considering the feed materials to blast furnace, sinter plant, SMS, including that arising from the use of coke oven gas, taking unit hot metal production as the basis.

The actual EF of coal and coke obtained from the collected samples were used in the estimation procedure. The contribution from coke oven gas was estimated from the coke oven gas composition and the amount of the gas combusted in steel making processes. The necessary correction for stored

carbon (4% in hot metal) was also considered during the calculation procedure. The mean value of CO<sub>2</sub> coefficient was 2.04 t/thm and from the flux was 0.1161 t/thm

### ***Cement plant***

GHG emission from cement plants can arise from both fuel and the feed materials like lime, dolomite etc. Traditional methods of estimating emission coefficients from the process only, is done from the Emission Factor (EF) of the clinker. Measurement in stack would provide the emission data arising from both fuel combustion and from the feed materials. Knowledge of coal quality and the flow rate could be used to identify the contribution to the emission from the fuel combustion.

### ***Energy related CO<sub>2</sub> emissions***

This will be an estimate based on the fuel consumption pattern in the plant and their quality for a certain span of time. The combustion related emissions have been assessed following same methodology as in Power plant studies.

### **Process related CO<sub>2</sub>**

#### ***Methodology 1***

The IPCC guideline, Tier II has been followed. The plant specific data on CaO content of clinker have been used to derive the emission factor of clinker and compared with the default value. Attempts have been made to reduce uncertainty on the basis of best possible estimates for CaO content of the clinker and plant production data and cement kiln dust losses etc.

#### ***Methodology II***

Estimates were made on the basis of the material balance over the Rotary kiln and component (carbon) balance. Attempts have been made to record variation of raw materials and their composition and thereby evaluating the uncertainty of CO<sub>2</sub> emission arising out of these variations during the period of study. During the same time frame direct measurement in the stack was carried out. This measurement would provide the combined emissions from both the fuel combustion and raw material decomposition. Stack measurement was done following the standard procedures employed in case of power plants.

Process related CO<sub>2</sub> emissions calculated considering the clinker composition, feed composition and CO<sub>2</sub> emissions from carbon value of fuel. The emission of CO<sub>2</sub> from the process was estimated using the IPCC Tier I method by multiplying the default EF link with the clinker production data. The same was also determined from the actual CaO and MgO content of the clinker and during this estimation. The values obtained from the three approaches are 0.50, 0.53 and 0.56 t/t clinkers respectively. The lower value of IPCC is possibly due to the consideration of CO<sub>2</sub> arising from CaCO<sub>3</sub> and not from the contribution of other form of carbonates like MgCO<sub>3</sub>. The SO<sub>2</sub>



and NO<sub>x</sub> obtained from direct measurement are 0.0013 and 0.0067 t/t clinker respectively. The CO<sub>2</sub> emission from fuel only is about 0.42 t/t clinker considering both the semi dry and dry processes.

The EIA approach for estimating emission factors for US coals provides a better route for increasing the accuracy of the emission estimate. Two types of carbon dioxide emission factors have been developed. First are basic emission factors covering the various coal ranks by State of origin. These basic emission factors are considered as "fixed" for the foreseeable future until better data become available. Second are emission factors for use in estimating carbon dioxide emissions from coal consumption by State, with consuming-sector detail. These emission factors are based on the mix of coal consumed and the basic emission factors by coal rank and State of origin. These emission factors are subject to change over time, reflecting changes in the mix of coal consumed.

Similar studies needs to be initiated in our country by linking the fuel source, both State of origin and rank, with the detailed data on the fuel mix of major power utilities, which can considerably improve the reliability and ensure consistency of the emission estimates from this major sector.

## IV. Grading System of Coal

### Background

Grading of coal for commercial purpose was in vogue since 1924. Division of coals into separate groups or class utilizing the moisture and ash in some form or other were attempted at different point of time. The system of grading however, is distinctly different from the classification of coals into different classes/subclasses. The latter is done on the basis of the inherent properties of coal whereas the former is developed on the basis of combinations of factors, both inherent and extraneous, involving the economics and technological applications of the specific coals.

To make the Indian coals vendible and the inadequacies of the different approaches towards grading of non coking coal led to the introduction of the concept of Useful Heat Value which was essentially a technological correlation of the contemporary period having commercial implications. The approach suggested by the "Study Group constituted by Govt. of India in 1962" was to derive the UHV by subtracting "ash penalty" from the GCV, calculated from the empirical formulae proposed by CFRI for both low and high moisture coals. The cumbersome procedure of calculating UHV was obviated by using two formulae, proposed by Mazumdar (CFRI) for direct calculation of UHV for both high and low moisture coals.

**For high moisture coals:** 
$$UHV = 8900 - 138(A+M)$$

A and M are ash and moisture percentages at 60%RH & 40°C

**For low moisture low volatile coals:** 
$$UHV = 8900 - 138(A+M) - 150(19.0-VM)$$

A, M, VM are ash, moisture and volatile matter percentages on as analysed basis

This system was implemented in 1979 and has been still in vogue for grading non coking coal

The relevance of the existing system of grading and pricing of non coking coal on the basis of Useful Heat Value (UHV) has been under scrutiny since the early nineties. The need for a change to switch from UHV to Gross Calorific Value (GCV) for grading purpose was voiced from different corners particularly in view of some lacunae in the grading pricing structure and also to make the grading system compatible with international practice.

UHV is a parameter used to determine the grade and hence the price of coal. The ash penalty approach and subsequent development of the formulae were based on quality of coal used and the prevalent combustion technology at that point of time. During that period about 85% of coal was made available from underground mining and rest from OCP. Consequently the quality of the coals was superior to the presently available coals from large open cast projects.



The average variations in the quality parameters were ash% in the range 20-25, moisture% in the range 2-5 and GCV between 5000-5500 kcal/kg. In contrast the variations in the respective parameters of the presently mined coals are very wide:- Moisture% 6-11, GCV 3500-5000 kcal/kg, average ash% 45 or even more. In view of this the applicability of the formulae in the present scenario is questionable.

The basic premise of UHV lies on the subtraction of ash penalty from the Gross Calorific Value. The ash penalty that was considered in early sixties for each grade was based on the quality of coals, loss of heat through ash and more importantly the prevalent combustion technology at that point of time. During that time the most inefficient steam generation equipment was possibly the locomotive boiler which was one of the largest consumer coal and less efficient than prevalent stoker fired system. As per available document the study group had mentioned "The penalty that was considered for locomotives which, in the extreme case, have shown a justification of employment of an ash penalty of about 2.5 times the ash (The Third Railway Fuel Enquiree Committee, 1956)". However, the Study Group recommended ultimately 1.25 times the dry ash, as the penalty for ash percentages upto 9% ash level and step wise increase of 0.05 units in the rate of penalty to 1.45 times upto ash level 26% and then flat rate 1.5 times for ash level beyond 27%. These ash penalty factors may not be applicable in the present day context.

The concept of UHV is unique in this country. In international trading the heat value is considered as one of the important parameter. This heat value is expressed either on Gross Calorific Value or Net Calorific Value and not on Useful Heat Value. For trading purpose, other factors like ash, moisture, sulphur, etc., do come into play and the parameters and their tolerance limit are identified mutually between the buyer and seller. Moreover the GCV is not only an important inspection parameter for international coal trading, it is also included as the primary parameter of classification of thermal coals both internationally and also in Indian Standards.

#### **Limitation of the existing grading system:**

The relevance of the existing system of grading of non coking coal on the basis of Useful Heat Value (UHV) has been under scrutiny since the early nineties. The need for a change to switch from UHV to Gross Calorific Value (GCV) for grading purpose was voiced from different corners particularly in view of some limitations in the grading structure based on UHV and also to make the system compatible with international practice of trading of coal.

- . The other significant points that need to be considered are:
  - The constant 8900 in the formula are the highest dmmf CV of coking coals of Jharia coalfield. This value corresponds to coal having 90.5% carbon and hydrogen 4.8% in dmmf basis. This figure is not acceptable in the present day context when large quantity of coal is mined from other coalfields like Mahanadi, Ib valley, North Karanpura, etc.

- The equal weightage factor of 138 for the both the moisture and ash level is not valid for high ash coals.
- The UHV at ash+moisture value of 64.5% is zero but the GCV value in this range of ash & moisture can be even of the order of 2300 kcal/kg.
- The formula for estimating the UHV, in vogue since 1979, was introduced to avoid the cumbersome procedure of calculating the GCV and subtracting the ash penalty to arrive at the UHV for each grade, which was introduced in 1976. The basic premise of UHV lies on the subtraction of ash penalty from the Gross Calorific Value. The ash penalty that was considered in early sixties for each grade was based on the quality of coals, loss of heat through ash and more importantly the prevalent combustion technology. During that time the most inefficient steam generation equipment was possibly the locomotive boiler which was one of the largest consumer of coal and less efficient than prevalent stoker fired system. As per available document the Study Group had mentioned “The penalty that was considered for locomotives which, in the extreme case, have shown a justification of employment of an ash penalty of about 2.5 times the ash (The Third Railway Fuel Enquiry Committee, 1956). However, the Study Group recommended ultimately 1.25 times the dry ash, as the penalty for ash percentages upto 9% ash level and step wise increase of 0.05 units in the rate of penalty to 1.45 times upto ash level 26% and then flat rate 1.5 times for ash level beyond 27%. These ash penalty factors may not be applicable in the present day context.
- Low ash imported coals can give higher UHV than the corresponding GCV. Even the low ash washed fraction of indigenous coals can have higher UHV than the GCV which contradicts the basic contention that UHV is derived from GCV by deducting the ash penalty factor.
- Calorific value depends on the rank, petrographic mix other than the extraneous factors. There is a wide variation in the coal quality parameters of the noncoking coals from different region. The variations range from C%: 72-82, H: 4.5-6.3, GCV: 7200-8200 kcal/kg and O%: 6-15, all on dmf basis. Due to such variation of the said properties, coals having the same ash and moisture content can have wide range of variation in the GCV.

### **Work done at CFRI**

An extensive work has been done by CFRI on the development of equivalency chart between UHV and GCV considering six major coalfields of India through a project sponsored by MOC in 2001-2003.

As a sequel to the above work Coal India Limited and National Thermal Power Corporation jointly sponsored a pilot study for possible migration from the existing UHV (Useful Heat Value) based gradation to GCV (Gross Calorific Value) based gradation of non coking coals. The intent of the pilot study is to generate sufficient data for likely gradation of coal on the basis of

GCV and or its related parameters and also, to evolve the gradation of Indian coals based on the international practice.

## **V. Trace elements in coal: Environmental impact due to combustion**

Coal will keep its important position as a world energy source because of its relatively abundant reserves. The pollutant emissions from coal utilisation may cause serious environmental and health risks, making it an important issue with respect to environmental protection. The concern about the fate of trace elements during coal combustion (i.e. the partitioning, environmental impacts, emission control etc.) is a relatively newer field of research. Among the host of trace elements present in coal, about fifteen are considered to be potentially toxic.

From the early nineties, extensive studies are being carried out at CFRI for the assessment of concentration level of significant trace elements present in coal, lignite and other fossil fuels, their emissions and partitioning in the combustion products during combustion. Activities are also underway to develop specific adsorbents for the removal of Hg, As etc. from gas phase as well as liquid effluents.

The significant achievements made during the last decade on the subject are:

- Development of improved analytical procedure for the estimation of Hg, As, Cd, Pb and other toxic heavy elements by flame atomic absorption spectroscopic method.
- Survey on ground water contamination with arsenic at the affected zones of West Bengal
- Investigation on removal of arsenic from ground water by coagulation method.
- Solidification/stabilisation of arsenic contaminated sludge for abatement of secondary pollution
- Generation of data bank on priority trace elements in the major coal/lignite reserves of India
- Publication of monograph on 'trace elements in Indian coal'
- Study on mercury in coal and combustion products of super thermal power stations of NTPC in Singrauli region
- Preparation of draft Indian Standard on "Determination of trace elements in coal, coke and fly ash by flame atomic absorption spectroscopic method"

### **Future R&D prospects:**

- Assessment and monitoring of the emission of potential toxic trace elements included in HAP's is the need of the hour because all the coal based thermal power stations are loading the background level in soil, air and water due to fossil fuel combustion.
- Preparation of certified reference material for trace elements analysis in coal, coke and coal combustion products (fly-ash, bottom-ash) involving other reputed laboratories.

- Development of absorbents for the removal of toxic heavy metals from waste water
- Mathematical modeling on trace element emission due to coal combustion for predicting enrichment in soil, water surrounding thermal power station.
- Development of improved sample preparation and analytical technique for trace element estimation for attaining better reliability of results.

## **New Initiatives**

### **1. Oxy-fuel combustion of pulverized coal**

The awareness of the increase in green house gas emission has resulted in the development of new technologies with lower emissions and technologies that can accommodate capture and sequestration of carbon dioxide. Oxy fuel combustion coupled with CO<sub>2</sub> capture from flue gas is a near –zero emission technology that can be adopted for new or existing coal fired power plants For existing coal-fired combustion plants there are two main options for CO<sub>2</sub> capture, removal of nitrogen from flue gases or removal of nitrogen from air before combustion to obtain a gas stream ready for sequestration. In oxy-fuel combustion, fuel is combusted in pure oxygen rather than air and by recycling the flue gas in the combustor the carbon dioxide percentage in the exit flue gas is increased. In the oxy-fuel combustion the flue gas is relatively pure CO<sub>2</sub> which is easy for sequestration and also it is having the potential to reduce pollutant emissions, in particular NO<sub>x</sub>.

Data on oxy fuel combustion of Indian coals in pilot scale is scanty The activities will focus on evaluating the effect of coal characteristics and other operating parameters like oxygen concentration or recycle ratio during oxy fuel combustion on ignition, flame stability, emission and associated ash depositional problems in a pilot scale combustion facility. This will be preceded by laboratory scale studies in TGA on the effect of the presence of CO<sub>2</sub> on char combustion reactivity. This will provide relevant data for combustion behaviour in various O<sub>2</sub> & Recycled Flue Gas (RPG) mixture and help to assess the role of different technical factors on combustion performance

### **2. Co-combustion of Pulverised coal and biomass**

There is an increasing trend of utilizing biomass for energy production globally primarily due to reduction of CO<sub>2</sub> emission and utilization of large quantity of bio wastes. Among the different thermo chemical processes combustion is the most important and proven technology for biomass utilization. Limited study has been carried in this country on co- combustion of different biomass with pulverized Indian coal. The significant variations of the properties of different types of biomass and coal warrant a detailed investigations on the chemical, mineralogical, trace elements, etc., of the source materials followed by combustion of blends with the suitable coals. The biomass with different chemical makeup and ash compositions will effect the different combustion related issues when co-firing with pulverized coals.

The activities will be aimed at exploring the following important aspects of co-combustion:

- different mode of sample preparation of high moisture biomass
- co-grinding with coal
- assessment of the emissions of NO<sub>x</sub>, SO<sub>x</sub>
- slagging and fouling behaviour arising due to the variations in the ash chemistry of coal and biomass
- effect on the quality of the fly ash
- optimizing the proportion of different biomass in the blend with indigenous coals.

## ANNEXURE

## 1. List of publications

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| 1  | World Power Conference 1947 held at Hague 1-10 Sept. (1947)<br><i>J. sci. industr. Res.</i> , <b>7</b> (1), 9 (1948)  | Whitaker, J.W.                                |
| 2  | Development of Powdered Coal Gas Turbine for Locomotive<br>Purpose - A Review<br><i>J. sci. industr. Res.</i> , <b>8A</b> , 486 (1949)  | Guruswamy, S.                                 |
| 3  | Fourth World Power Conference London, July, (1950)<br><i>J. sci. industr. Res.</i> , <b>9A</b> , 442 (1950)   | Whitaker, J.W.                                |
| 4  | World Power Conference, Sectional Meeting, 1951 Delhi<br><i>J. sci. industr. Res.</i> , <b>10A</b> , 143 (1951)   | Whitaker, J.W.                                |
| 5  | Coals and Their Heating Values<br><i>Steam Users' J.</i> , <b>2</b> , 26 (1952)   | Whitaker, J.W.                                |
| 6  | Critical Oxidation and Ignition Temperature of Coal<br><i>Symp. on Hazards of Underground Fire in Indian Mines, May (1954)</i><br><i>IMMA Rev.</i> , <b>2</b> (4), 234 (1954)   | Ganguli, M.L.<br>Banerjee, N.G.               |
| 7  | Experimental Coal Stacks<br><i>Symp. on Hazards of the Underground Fire in Indian Mines, May (1954)</i><br><i>IMMA Rev.</i> , <b>2</b> (4), 222 (1954)                          | Lahiri, K.C.<br>Ganguli, M.L.<br>Moitra, A.K. |
| 8  | Spontaneous Ignition of Coals<br><i>Symp. on Hazards of Underground Fire in Indian Mines, May (1954)</i><br><i>IMMA Rev.</i> , <b>2</b> (4), 240 (1954)                         | Mukherjee, P.N.                               |
| 9  | Spontaneous Combustion of Coal<br><i>FRI News</i> , <b>4</b> (1), 1, (1954)<br><i>Steam Users' J.</i> , <b>5</b> , 25 (1955)  | Moitra, A.K.                                  |
| 10 | Problems of Energy Development : Part I<br><i>FRI News</i> , <b>4</b> (3), 2 (1954)   | Lahiri, A.<br>Das Gupta, A.K.                 |
| 11 | Problems of Energy Development : Part II - Our Requirements<br><i>FRI News</i> , <b>4</b> (4), 2 (1954)   | Lahiri, A.<br>Das Gupta, A.K.                 |
| 12 | Problems of Energy Development : Part III - Out Reserves<br><i>FRI News</i> , <b>5</b> (1), 2 (1955)  | Lahiri, A.<br>Das Gupta, A.K.                 |
| 13 | Problems of Energy Development : Part IV - Pattern of Energy Utilization<br><i>FRI News</i> , <b>5</b> (2), 34 (1955)   | Lahiri, A.<br>Das Gupta, A.K.                 |
| 14 | Coal in India<br><i>Symp. on Indian Coal Industry held at Calcutta, Aug. (1955)</i><br><i>Ind. Min. J.</i> , <b>3</b> (9), 13 (1955)  | A. Lahiri.                                    |
| 15 | Problems of Energy Development in India, Part V - Pattern of Zonal Development<br><i>FRI News</i> , <b>5</b> (3), 67 (1955)<br><i>Steam Users' J.</i> , <b>6</b> (2), 17 (1956) | Lahiri A.<br>Das Gupta, A.K.                  |
| 16 | Role of Fuel in Glass and Ceramics Industry<br><i>Indian Ceramics</i> , <b>2</b> , 85 (1955)  | Lahiri, A.                                    |
| 17 | Pattern of Energy Utilization to India<br><i>Chemi. Age of India</i> , <b>7</b> (4), 23 (1956)  | Lahiri, A.                                    |
| 18 | Problems of Energy Development in India, Part VI - National Planning and Fuel Research<br><i>FRI News</i> , <b>6</b> (1), 2 (1956)  | Lahiri, A.<br>Das Gupta, A.K.                 |
| 19 | Fuel Efficiency Organisation<br><i>J. sci. industr. Res.</i> , <b>15A</b> (3), 107 (1956)   | Whitaker, J.W.                                |
| 20 | Studies on the Inflammability of Indian Coals<br><i>Trans. Min. Geol. &amp; Met. Inst. (India)</i> , <b>54</b> (1), 43 (1957)   | Sen, M.M.<br>Das Gupta, S.K.                  |
| 21 | Pulverized Coal Firing in Fire Tube Boiler  | Lahiri, A.<br>Rao, S.G.                       |





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|    | <i>FRI News</i> , <b>7</b> (4), 100 (1957)  | Rao, S.G.       |
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| 24 | Fuel Efficiency Studies in a Glass Industry   | Rao, S.G.       |
|    | <i>Ind. Ceramics</i> , <b>4</b> , 13 (1957)   | Sen, M.         |
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|    | <i>World Power Conference, Montreal, Canada, Paper No. 45G/8</i> (1958)                                     | Iyengar, M.S.   |
|    |   | Moitra, A.K.    |
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| 29 | Requirement and Resources for Power Generation in India   | Lahiri, A.      |
|    | <i>Ind. J. Power &amp; River Valley Rev.</i> , <b>8</b> (12), 1 (1959)                                      | Ghosal, A.      |
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102	Development of an Efficient Domestic Oven for Burning Processed Solid Fuel <i>Res. &amp; Ind. Get-together Held at C.S.I.R., New Delhi (Dec. 1965)</i>	R.N. Moitra, A.K. Das Gupta, S.K.
103	Fuel and Power in Major Manufacturing Industries in India 1951 & 1958 <i>Eastern Economist, 44, Budget No. 606-623 (1965)</i>	Sinha, N.C. Basak, P.K. Moitra, A.K. Lahiri, A.
104	Problems of Energy Supply in India during the Fourth Five Year Plan <i>Symp. on Problems of Energy Supply in India during Fourth Plant held at CFRI (4&amp;5 Feb. 1966)</i>	Sinha, N.C. Lahiri, A.
105	The Ramgarh Industrial Complex <i>FRI News 16 (2), 52 (1966)</i>	Sen, S.K. Lahiri, A.
106	Combustion of Slack Coal in Vertical Boilers <i>Symp. on Combustion Organised by Combustion Institute (Indian Section), IIT, Kharagpur (27-28 Feb., 1967)</i>	Chakraborty, J. Das Gupta, S.K.
107	Prevention of Fire in Coal Pillars in Mines : Part I - Treatment of Coal to Prevent Oxidation <i>MGMI, Annual Meeting, CFRI, Dhanbad (1967)</i>	Kanjilal, K.K. Gupta, S. Mukherjee, P.N. Mukherjee, A.K. Bagchi, S.
108	Prevention of Fire in Coal Pillars in Mines : Part II - Treatment of Underground Pillars to prevent Spontaneous Combustion <i>MGMI, Annual Meeting at CFRI, Dhanbad (1967)</i>	Ghosh, S. Gupta, S. Bagchi, S.
109	The Loss of Power and Measures to Overcome It in the Reciprocating Internal Combustion Engine under Varying Conditions of Pressure and Temperature <i>III Symp. of Combustion Institute (Indian Section), Roorkee Univ., Roorkee, (15 Mar., 1967)</i>	Jagadish, S.R.
110	Wall to Particle Heat Transfer in Fluidized Bed and Its Effect on Operational Variables <i>20th Annual Session of the Institute of Chemical Engineers, Neyveli (28-30 Dec., 1967)</i>	Chowdhury, S.B. Banerjee, S. Lahiri, A.
111	Place of Coal in Electrical Energy Development <i>MGMI &amp; JWC Coal Prod. Asn., Symp. "Coal Mining Industry" [Inaug. Seasn. GSI, Calcutta (Aug. 1969)]</i>	Rao, S. Ranga Raja Sinha, N.C. Lahiri, A.
112	Role of Coal in Energy Pattern of India <i>GMI &amp; JWC Coal Prod. Assn., Symp. "Coal Mining Industry", [Inaug. Sessn. GSI, Calcutta, (May 1969)]</i>	Lahiri, A.
113	Some Techniques of Energy Forecasting <i>MGMI &amp; JWC Coal Prod. Asn., Symp. "Coal Mining Industry" [Inaug. Seasn. GSI, Calcutta (May 1969)]</i>	Sinha, N.C. Ghosh, S.N. Lahiri, A.
114	Utilisation of Slack Coal Produced in Collieries of Central India <i>Sem. of Inst. Engrs. (India), IMMA (MP &amp; Maharashtra Branch) and American Inst. Mng. Metl. &amp; Pet. Engrs. Inc. (India Sec.), Nagpur (11-13 Apr., 1969)</i>	Chatterjee, C. Kudwali, R.N. Shrikhande, K.Y.
115	Efficiency in Coal Utilization in India and Role of Education, Training and Management <i>Nat. Sem. on "Fuel Efficiency", NPC, New Delhi (5-7 Oct. 1970)</i>	Acharjee, B. Rao, S. Ranga Raja
116	Productivity and Fuel Power Utilisation in Major Indian Manufacturing Industries <i>Nat. Sem. on "Fuel Efficiency", NPC, New Delhi (5-7 Oct. 1970)</i>	Sinha, N.C. Ghosh, S.N. Moitra, A.K.
117	Some Typical Industrial Fuel Efficiency Tests conducted by CFRI	Biswas, D.K.



	<i>Nat. Sem. on "Fuel Efficiency", NPC, New Delhi (5-7 Oct. 1970)</i>	Biswas, R.R. Juneja, M.N. Mukherjee, D.K. Pal, G. Sen, M. Das Gupta, S.K.
118	The Energy Outlook for India during the Seventies <i>Nat. Sem. on "Fuel Efficiency", NPC, New Delhi (5-7 Oct. 1970)</i>	Lahiri, A. Sinha, N.C.
119	Alternate Fuels for Blast Furnaces <i>Silver Jubilee Symposium of Ind. Inst. of Metals, New Delhi (4-9 Feb., 1972)</i>	Raja, K. Moitra, A.K. Lahiri, A.
120	Coal Based Energy <i>Symp. on Special Energy Source' 64th Indian Science Congress, Bhubaneswar (3-7 Jan., 1977)</i>	Krishna, M.G.
121	Moisture Problems in Air Liquefaction and Possible Remedies <i>IV National Symposium on 'Cryogenics', IIT, Bombay, (Dec. 1979)</i>	Jagdish, S.R.
122	Studies on Rheology of Suspension of Coal in Anthracene Oil <i>Presented at 32nd Ann. Sec. of I.I.Ch. Engrs., I.I.T., Bombay (Dec. 1979)</i>	Paul, R.N. Mall, B.K. Chakraborty, M. Mukherjee, P.N.
123	Energy Problems in India for Eighties <i>Seminar on 'Energy Management', Jamshedpur (March 1980)</i>	
124	Influence of Coal Characteristic on the Rheology of Coal-Oil Suspension <i>I.I.Ch.E. 33rd Annual Session at IIT, New Delhi (16-19 Dec. 1980)</i>	Paul, R.N. Mall, B.K. Chakravathy, M.
125	Rational and Efficient Utilization of Coal <i>Seminar on "Coal" organised by Institute for Railway Transport, Eastern Region, Calcutta (Oct. 1980)</i>	Ghosh, S. Ash, P.N.
126	Cost of Fuel and Power in Selected Indian Industries after the Energy Crisis <i>Seminar on 'Energy Conservation', New Delhi (17-19 Mar. 1981)</i>	Sinha, N.C. Sinha, B.K.
127	Objective Basic Research : A Vital Tool for Management of Power Plants <i>Seminar on 'Management Interfaces for 80's', New Delhi (Dec. 1981)</i>	Rao, J.J. Rao, S.K.
128	Outlook for Tar as Biner in Road and Building Research Construction in India <i>Nat. Symp. on 'Binder Economy &amp; Alternate Binders in Road &amp; Build. Construction &amp; Conservation' held at CRRI, New Delhi (3-4 Nov. 1981)</i>	Sen, D.K. Ray, M.B.
129	Role of Coal in Water Purification <i>All India Conference on 'Water Treatment for Industrial and T.P.S. Boilers', New Delhi (10 Apr. 1981)</i>	Rao, J.J.
130	Combustion of Coals and Waste Fuels : R & D Work. CFRI <i>Seminar on Coal like a Byproducts, BIT, Sindri (1984)</i>	Verma, S.K. Talapatra, P.C. Sen, M.M.
131	Grindability of Coal and its Relation to Selection of Coal to Pulverised Fuel Fired Thermal Power Station <i>Souvenir of Govt. of West Bengal (1984)</i>	Banerjee, A.N. Bagchi, D.
132	Present and Future Pattern of Fuel Utilization in Indian Ceramic Industry <i>Seminar on Indian Ceramic Industry Preparing for 21st Century, New Delhi (15-16 March 1986)</i>	Biswas, D.K. Verma, S.K. Mazumdar, A. Mazumdar, S.K. Sen, M.M.
133	Techno-Economic Studies on the Alternative Techniques of Hydraulic Transport of Coal Indian Context	Chakraborty, M. Hazra, S.K.



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| 134 | <p>39th Annual Session of I.I.Ch.E., Osmania University (Dec. 1986)</p> <p>Beneficiation of Non-coking Coals for Power Generation in India and Its Economics</p> <p><i>MGMI Seminar on Beneficiation of Non-coking Coal, New Delhi (25-26 Apr. 1987)</i></p> | <p>Ghose, S.</p> <p>Haque, R.</p>   |
| 135 | <p>Techno-Economics Studies on the Alternative Techniques of Hydraulic Transportation of Coal</p> <p><i>International Conference on Hyddraulic Transportation of Coal, RRL (B), ISHT (Jan. 1988)</i></p>   | <p>Chakaraborty, M.</p> <p>Hazra, S.K.</p>  |
| 136 | <p>Rheology and Stability of Coal-Water Mixture Fuel (CWM) with Typical Indian Non-coking coals</p> <p><i>Symp. on Coal Utilization : Trends and Challenges, CFRI, Dhanbad (13-14 Dec. 1989)</i></p> <p><i>Fuel Sci. Technol., 9 (10), 105 (1991)</i></p>    | <p>Majumdar, S.</p> <p>Prasad, M.</p> <p>Chanda, S.K.</p> <p>Bhowmik, T.K.</p> <p>Chakraborty, M.</p> |
| 137 | <p>Studies on the Rheology of the Suspension of Lignite in Medium Viscous Fuel Oil</p> <p><i>Symp. on Coal Utilization : Trends and Challenges, CFRI, Dhanbad (13-14 Dec. 1989)</i></p> <p><i>Fuel Sci. Technol., 10 (1 &amp; 2), 89 (1991)</i></p>          | <p>Paul, R.N.</p> <p>Bose, S.K.</p> <p>Chakraborty, M.</p>  |
| 138 | <p>Optimal Utilization of Indian Coals for Iron Making</p> <p><i>Seminar on 'Coal for Blast Furnace Coke and for Injection', Indian Institute of Metal, TISCO, Jamshedpur (21-22 Sep., 1990)</i></p>   | <p>Haque, R.</p>  |
| 139 | <p>Studies on the Rheology of Suspension of Coal in Low Sulphur Heavy Stock,</p> <p><i>43rd Indian Chemical Engineering Congress, BHU (18-21 Dec. 1990)</i></p>  | <p>Paul, R.N.</p> <p>Basu, S.K.</p> <p>Mall, B.K.</p> <p>Bandopadhyay, P.K.</p>                       |
| 140 | <p>Emission Characteristics of Coal/Coke Fired Domestic Chullahas</p> <p><i>CHEMCON - 1991 II Ch E. Congress-91 (18-21 Dec. 1991)</i></p>  | <p>Mitra, S.K.</p> <p>Sengupta, A.N.</p> <p>Mall, B.P.</p> <p>Rudra, S.R.</p> <p>Mukherjee, D.K.</p>  |

## Coal Gasification Division



## **Coal Gasification at a Glance**

Coal gasification process offers a practical means of utilizing coal for meeting stringent environment control requirements. In coal gasification process broad range of coals with varying ash content, coal fine, and middling can be successfully gasified. Bulk coal reserves in India (about 73%) are of inferior grades non-cooking coals, with ash contents as high as 45-50%. Coal for Indian power plant is supplied from these reserves. The quality of coal is further deteriorating due to open cast mine.

The core competency of CFRI is on coal science. However, CFRI found a need to establish the relationship among coal characteristics, kinetics and effect of design on gasifier performance. Attempts were made in sixties to correlate the performance of gasifier to characteristics of coal by using a (800kg/h) Lurgi gasifier as well as, Entrained Bed gasifier (110kg/h) and also FBG (40kg/h). Demonstration size gasifier posed difficulties in controlling the parameters of gasifiers such that correlation can be arrived among coal characteristics, kinetics and effect of reactor configuration.

CFRI has now taken up a multifaceted gasification program to correlate gasification performance and to develop a methodology for scaling up. These program cover following aspects:

1. Setting up 50kg/h PFBG air blown test facility, however, it represents the actual hydrodynamics of large plant.
2. To correlate surface characteristics with kinetics of coal gasification using PTGA.
3. Once, the testing unit is operational the overall characteristics of coal will be correlated to gasification performance in the testing unit.

It is expected that at the completion of the study CFRI will be in the position to suggest operational parameters of Indian coals and to develop a methodology for scaling up of gasifier for particular coal.

### **Objectives of the Planned Task on Gasification Kinetics**

- Characterization of the feed materials, chars and cinders.
- Classification of coal based on surface characteristics.
- Analysis of the product gases.
- Study of the kinetics of gasification of low rank coals.

- Mathematical modeling for prediction of composition of gases as a function of operating parameters and properties of coals for gasification reaction.
- Agglomeration formation in reducing condition in pressurized fluidized bed gasification with low rank high ash coals.

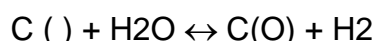
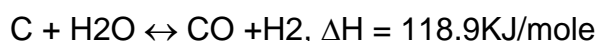
### **Facilities available at CFRI**

1. Ultrapycnometer -1000 for true density determination of solids
2. Tristar-3000 for surface area determination of solid samples.
3. Auto pore IV 9510 mercury porosimeter to study the pore structure of solids. The said instrument has already installed and working satisfactorily (Rs. 25 lakhs from CMM-12)
4. Autochem 2910 for TPD-TPR-TPO study
5. PW 2424 Magix XRF for elemental analysis of solid as well as liquid samples and XRD for minerals analysis.
6. P TGA with EGA facility, the main instrument for Gasification kinetics of low rank coal is under installation and commissioning (145 lakhs).

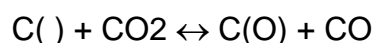
### **Estimating Reactivity of Char by Oxygen Chemisorption For Gasification**

Gasification of Carbon for Mechanistic model

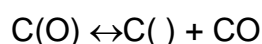
Gasification with Steam:



Gasification with CO<sub>2</sub>:



Gasification with O<sub>2</sub>:



In the entire models oxy-carbon complex formation step is the rate determining step.

Gasification of Coal/Lignite consists of two major reactions: i) Pyrolysis ii) Gasification of Char. Reaction rate depends on Char gasification since it is

slower than the devolatilisation. In studying the relative reactivity of carbon, it is quite inappropriate at the fundamental level, to express rate of reaction only in units of reciprocal of time. It only indicates the relative rate of gasification at bulk carbon materials. But for the studies of fundamental of oxidation process, in which the structure of carbon surface are being compared then not equal mass but equal accessibility of the surface has to be the normalizing factors.

### **Unit of Reactivity and Development of Reaction Model**

The unit of comparison should be gram adsorbed or lost per sq. meter surface area per unit time, may be the unit of reactivity. Therefore, the surface characterization is an important parameter in the study of carbon reactivity. Adsorption method of nitrogen at 77K determining surface area. Application of carbon dioxide adsorbent at 195K or 273K is also for surface area measurement.

In the char gasification process, char reacts with gasifying agents to produce a combustible gas. During gasification the surface area of gasified char increases rapidly with the process of gasification. Further to study reaction model with respect to change in specific surface area of char grain model and a random pore model, are used for gas and solid reaction. The grain model assumes that char particles are agglomeration of small grains, each of which follows the unburned core model.

The following equation is used to calculate specific surface area at different stage of gasification.

$$S = S_0 (1-X)^{2/3}$$

Where,  $S_0$  is initial specific area and  $X$  is conversion ratio of char. In random pore model cylindrical pores of uneven diameter are assumed and their internal surface area eroded with progress of reaction and eventually merge together.

Specific surface area according to this model is denoted as follows:

$$S = S_0 (1-X) \{1 - \Psi \ln (1-X)\}^{1/2}$$

Where,  $\Psi$  is a dimensionless parameter indicating the initial pore structure. It is calculated as follows using pore length ( $L_0$ ) and porosity ( $\epsilon_0$ ) per unit volume of solid:

$$\Psi = 4\pi L_0 (1-\epsilon_0) / S_0^2$$

Now one has to compare result with model based calculation results in respect of changes overtime conversion ratio and in reaction rate. It is easier

to make detail observation of reaction rate using high pressure TGA and the results further should be verified by P.D.T.F. and the rate equation will be denoted as follows:

$$dx/dt = K_p (1-X)\{(1-\Psi \ln (1-X))\}^{1/2}$$

Surface area determination is a complex problem as it is not directly measured and also the physical properties are different in different methods. The consciousness in literature feels direct proportionality between reactivity of carbon and concentration of active site (CT) for carbon. It also provides method for SA measurements, in the form of active surface area (ASA). A scientist has to decide the proper adsorbent, which most closely approximate to the SA available to the reaction condition.

Considering the above facts following task have been done:

1. Surface area by Nitrogen as well as carbon dioxide.
2. Active surface area using active site consideration by oxygen chemisorptions.
3. Calculation of oxygen reactivity at particular temperature /unit surface area/ unit time.
4. The rate value so obtained was correlated with different surface properties.
5. Lastly surface characteristic of char was also correlated with chemical properties of the parent lignite.

Presently, concept of reactive surface area (RSA) is introduced for quantitative understanding of char gasification kinetics. However, oxygen chemisorptions below gasification temperature is difficult to find RSA. To resolve this problem, Jenkinson and Piotrowski introduced a concept of TON “turnover number” from the quantities of CO and CO<sub>2</sub> evolved. TON really represents the product of the number of reactive sites (RSA) and the activity per site.

### **Papers in top referred journals**

- Kinetic Studies on Reactivity of Lignite char by TPD-TPO system (CFRI) has been carried out. One paper based on this reactivity study entitled “Estimating reactivity of lignite char by Oxygen Chemisorptions for Gasification” by B.K.Sharma and Sujan Saha has been published in the International Seminar- Global Coal-2005.
- A nano-particle based material has been developed for CO<sub>2</sub> sequestration (NCL-CFRI). One paper entitled “ Reverse micro emulsion mediated sol-gel synthesis of lithium silicate nanoparticles under ambient conditions: Scope for CO<sub>2</sub> sequestration” by R.B.Khomane, B.K.Sharma, S.Saha and B.D.Kulkarni (CFRI and NCL) has been published in Chemical Engineering Science, Volume 61, Issue 10, May 2006, Pages 3415-3418.
- Another paper “Estimation of Gross Calorific Value of Coals using Artificial Neural Networks” by S.U.Patel, B.J.Kumar, Y.P.Badhe, B.K.Sharma, S.Saha, S.Biswas, A.Chaudhury, S.S.Tambe and B.D.Kulkarni has been accepted by the Journal FUEL (CFRI and NCL) on 19<sup>th</sup> July 2006. Now it is ‘Article in Press’.
- Methodology for the determination of true density of coal using Nitrogen - a new technique (CFRI-NCL). A paper entitled “Density measurements of coal samples by different probe gases and their interrelation” by Sujan Saha, B.K.Sharma, S.Kumar, S.G.Sahu, Y.P.Badhe, S.S.Tambe and B.D.Kulkarni (CFRI and NCL) has been communicated to the Journal FUEL.
- Artificial Intelligence based models for classifying Indian non coking Coals (CFRI & NCL) is under preparation.
- A correlation for prediction of product gases during gasification of coal using steam/air under different operating conditions using ANN concept is under progress and the same prediction will be validated on our system ( CFRI & NCL).

### **New Know-how for Marketing/ consultancy to foreign party**

1. CFRI, Dhanbad and NCL,Pune have received jointly consultancy work from M/S Adani Energy Limited, Gujrat for preparing a report on “ Pre Feasibility Study for coal Gasification Project” of Project Fee of Rs 8,50,000/-+ service tax.
2. CFRI was also invited by Project Management Cell, UNDP/GEF Project (Steel), Ministry of Steel, Government of India for selection of appropriate Coal Gasification plant (capacity about 1500 Nm<sup>3</sup>/hr) and offered a letter for submission of project proposal.

## I. Experience of CFRI on coal Gasification

In the present energy crisis, development of efficient and economically viable process for gasification of coal is bound to play an important role. Particularly, the gas as a starting material for synthesizing fertilizers, chemicals and oils is also an efficient and convenient fuel. In this context, considerable research and development work has been done on different pilot plants at CFRI since its inception for commercial exploitation of the abundant resource of non-coking and low grade coals available in the country for generation of gases suitable for industrial and domestic uses.

### 1. Down Draft Gasifier

The pilot plant was installed in 1952 and operated with oxygen enriched (45-60%) air as gasifying medium for production of fuel gas and synthesis gas. The reactor was made of zircon tube at the top followed by mullite tubes resting on a firebrick base. Experiments with a high volatile pulverised coal of Raniganj field were carried out in this system. The coal feed rate was 22-35kg/h. The consumption of oxygen was 0.5-0.8 kg/kg coal.

#### Results obtained from Down draft gasification with oxygen enriched air

Analysis of Coal (air dried basis)		Operating conditions (range)	
<b>Proximate Analysis, Wt %</b>		Feed size, mesh	-200 (80%)
Moisture	7.5	Feed Rate, kg/h	22.7 – 34.1
Ash	14.1	Oxygen enrichment	45 - 60
VM	33.9	Temperature °C	560 - 945
Fixed Carbon	44.5		
<b>Ultimate Analysis, Wt %</b>		<b>Results obtained</b>	
Moisture	7.5	Gas Nm <sup>3</sup> /h	16.4 – 51.2
Mineral Matter	15.48	Heating Value, kcal/ Nm <sup>3</sup>	1530
Carbon	62.15		
Hydrogen	4.17		
Nitrogen	1.16		
Sulphur	0.17		
Oxygen (by diff)			

### 2. Suspended Bed Coal Dust Gasification

With a view to utilize abundantly available low grade under sized (below 200 mesh) coals a Suspended Bed gasifier ( Kopper Totzek type) of 110 kg/h coal feed capacity was commissioned in 1959. Several tests were carried out with Neyveli lignite, Kutch lignite and non-caking bituminous coals from different

coal fields including Jambad Bowla, Lower Kenda, Talcher, Singrauli, Urmer and Argada etc.

**Results obtained from Suspended bed Gasification with Oxygen & Steam**

Analysis of Coal (air dried basis)		Operating conditions (range)	
Proximate Analysis, Wt %		Feed Rate, kg/h	77.27 – 111.3
Moisture	4.3 – 7.4	Oxygen/ Coal, kg/kg	0.50 – 0.85
Ash	11.1 – 16.9	Steam/ Coal, kg/kg	0.08 – 0.27
VM	34.5 – 46.2		
Fixed Carbon	34.7 – 45.9		
Gas Composition, Vol%		Results obtained	
Carbon Dioxide	11.5- 18.1	Heating Value, kcal/ Nm <sup>3</sup>	2435 - 2635
Oxygen	0 – 0.4		
Carbon monoxide	38.1 – 54.7		
Hydrogen	28.4 – 39.8		
Methane	0 – 2.60		
Nitrogen	1.4 – 4.2		

### 3. Fixed bed high pressure gasifier (Lurgi)

A Moving Bed (Lurgi ) gasification pilot plant (designed capacity: 800 kg/h) was and commissioned in 1962 at CFRI, Dhanbad for handling coals of various ranks ranging from lignite to weakly coking bituminous coals above 6mm (6-25mm) size for generation of both town gas as well as synthesis gas. The plant was operated at pressure up to 14 kg/cm<sup>2</sup> using air& steam or oxygen & steam as gasifying agents. A number of tests with non-coking bituminous coals from Talcher, Singareni, Dobrana and Burhur coalfields were undertaken Oxygen and Steam as gasifying agents.

**Results obtained from Fixed bed high pressure Gasification with Oxygen & Steam**

Analysis of Coal (air dried basis)		Operating conditions (range)	
<b>Proximate Analysis, Wt %</b>		Feed Rate, kg/h	291 - 556
Moisture	3.0 – 7.1	Pressure, kg/ cm <sup>2</sup>	10.6 – 25.0
Ash	12.4 – 21.5	Oxygen/ kg of Coal, Nm <sup>3</sup>	0.245 – 0.322
VM	26.3 – 35.6	Steam/ Coal, kg/kg	1.441 – 1.995
Fixed Carbon	43.2 – 45.		
<b>Ultimate Analysis, Wt %</b>		<b>Results obtained</b>	
Moisture	3.0 – 8.9	Gas Nm <sup>3</sup> /h	465 - 709
Mineral Matter	13.64 – 23.65	Heating Value, kcal/ Nm <sup>3</sup>	2789 - 3023
Carbon	57.49 – 65.69	<b>Gas Composition, Vol%</b>	
Hydrogen	3.64 – 4.46	Carbon Dioxide	28.8 – 31.6
Nitrogen	1.39 – 1.84	CnHn	0.4 – 0.9
Sulphur	0.38 – 0.55	Carbon monoxide	13.4 – 19.8
Oxygen	By difference	Hydrogen	37.9 – 46.2
		Methane	8.2 – 12.5
		Nitrogen	0.3 – 2.8

Number of experiments were carried out with the coals from Jambad and Lower Kenda coalfields with air and steam as gasifying medium.

**Results obtained from Fixed bed high pressure Gasification with Oxygen & Steam**

Analysis of Coal (air dried basis)		Operating conditions (range)	
<b>Proximate Analysis, Wt %</b>		Feed Rate, kg/h	124 - 165
Moisture	2.6 – 6.1	Pressure, kg/cm <sup>2</sup>	14.0
Ash	19.1 – 20.4	Air/ Coal, Nm <sup>3</sup> /kg	1.3 – 1.6
VM	33.2 – 33.3		
Fixed Carbon	41.5 – 43.8		
<b>Gas Composition, Vol%</b>		<b>Results obtained</b>	
Carbon Dioxide	14.8 – 16.8	Heating Value, kcal/ kg	1502 - 1904
Oxygen	0.2	Gas, Nm <sup>3</sup> /h	375 - 437
Carbon monoxide	12.8 – 13.9	Thermal Efficiency%	73.8 – 76.2
Hydrogen	15.7 – 20.7	Gasification Efficiency%	74.0 – 81.2
Methane	5.3 – 6.9		
Nitrogen	41.5 – 51.2		

**4. Fluid Bed Gasification (Two Stage)**

The pilot plant (designed capacity: 40 kg coal/h) was designed and installed at CFRI in early seventies. It consists of two mild steel reactors installed in series with coal and de-dusting system. The product gas was free from tar and any liquid carbonization products. Several tests were conducted with different non-coking sub-bituminous coals (0 – 3 mm) from Rajmahal, Jhingurdahl and dakra seam coalfields. Product gas had heating value in the range of 1000-1300 Kcals/Nm<sup>3</sup>.



**Results obtained from Fluid Bed Gasification with Air & Steam**

Analysis of Coal (air dried basis)		Operating conditions (range)	
<b>Proximate Analysis, Wt %</b>		Feed Rate, kg/h	13.5 – 40.0
Moisture	2.3 – 6.6	Air Rate, Nm <sup>3</sup> /h	24.0 – 67.8
Ash	25.2 – 44.6	Steam Rate, kg/h	4.0 – 13.7
VM	20.9 – 31.5	<b>Results obtained</b>	
Fixed Carbon	28.2 – 40.5	Gas Nm <sup>3</sup> /h	36 - 101
<b>Ultimate Analysis, Wt %</b>		Heating Value, kcal/ Nm <sup>3</sup>	1045 - 1315
Moisture	2.3 – 6.6	Thermal Efficiency%	55.0 – 69.0
Mineral Matter	27.72 – 49.06	Gasification Efficiency%	74.6 – 84.1
Carbon	36.81 – 56.71	<b>Gas Composition, Vol%</b>	
Hydrogen	2.81 – 4.37	Carbon monoxide	12.6 – 19.3
Nitrogen	0.62 – 1.67	Hydrogen	10.5 – 16.6
Sulphur	0.26 – 0.47	Methane	1.5 – 6.9
Oxygen	By difference		

**5. Bio Mass Gasification**

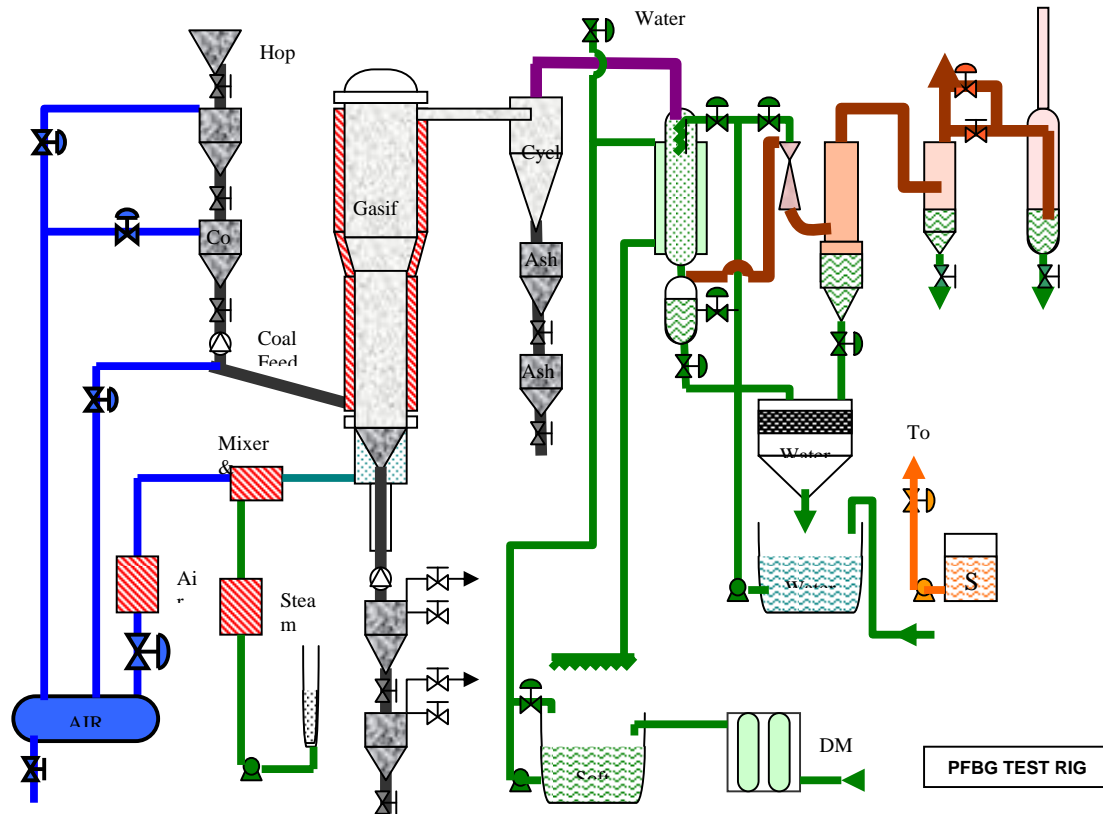
Biomass gasification unit was also developed at CFRI in late eighties of capacities 300 kg /day using saw dust, paddy husk and wooden chips as a feed.

**6. Pressurized Fluidized Bed Gasifier (PFBG)**

At present CFRI is in the process to set up a pilot plant of Pressurized Fluidized Bed Gasifier (PFBG) to gasify high ash low rank Indian coals at a gauge pressure of 3 kg/cm<sup>2</sup> and at a temperature of around 800 - 1100 °C. The gasifier plant consists of the following major sub-systems:

1. Reactor
2. Coal Feeding Module
3. Gaseous Reactant Supply Module
4. Bottom Ash Extraction Module
5. Cyclone With Fly Ash Collection Module
6. Gas Cooling And Cleaning Module
7. Exhaust System and Flare Stack

The functions of the off-site equipment are to feed preparation supply air, steam and cooling, circulating and quenching water to the process. A flow diagram is given below.



## II. Surface Characterization of Coal

Practically all aspects of coal utilization are influenced by the surface characteristics of coal. CFRI's experience in the study of surface property includes i) density measurement ii) porosity, pore size distribution and iii) surface area determination. CFRI is also equipped with TPD-TPR-TPO system.

### **Work related to porosity, pore structure, surface area: Influence of porous structure on coal gasification**

Coal Gasification is one of the oldest technologies, and continues to play an important role in coal utilization with the development of combined cycles. Understanding of char gasification kinetics and char structure is essential for the design and modeling of a coal gasifier. Char structure has been extensively studied by different authors due to its importance in char combustion and gasification processes [1-5] Char particles have been found to contain a large number of pores with radii ranging from one length of nanometer to tens of micrometers. The pores are randomly oriented and are of different shapes. Studies on char gasification show that the reaction rate not only varies with carbon conversion, reaction conditions but also very much depends on the physical structure of the char.

The gasification of coal consists of two major reactions: pyrolysis and gasification of chars. Since the rate of char gasification is much slower than that of devolatilisation, reaction rate of coal gasification depends on char gasification. During the course of char gasification, the micropores which make up approximately 95% of the total surface area [4] play a substantial role in the reactivity of char particles, while mesopores and macropores provide channels for reactant gas transportation [1,5]. Two competing phenomena determine the evolution of pore structure: growth of individual pores and coalescence of neighbouring pores [3]. Pore enlargement is dominant at low carbon conversion, resulting in the increase in the surface area and the observed reaction rate. As the reaction proceeds, the pore walls collapse and pore coalesce, leading to a decrease in the surface area and reaction rate. Presently CFRI is engaged in a project "Studies on gasification kinetics of low rank coals" which will create basis knowledge base on internal surface area, pore size distribution, porosity, oxygen

sorption study related to the gasification kinetics with special focus on Indian coals.

### **Significance of coal density**

Density is one of the important fundamental properties of coals and useful in providing information about the physical and chemical make-up. Owing to the involved complexities, density must be defined carefully and measured with at most precision. Therefore, considering the importance, it has been reviewed by different authors such as Dryden [6]. Fuji, et al. [7], Franklin [8], Bond and Spencer [9]. CFRI has also contributed in this area and Mr B.K.Mazumdar correlated true density of coal with its elemental composition [10]. Van Krevelen [11] and also Mr Mazumdar [12-15] showed how density could be used for evaluating complex coal characteristics, e.g., structural parameters such as ring condensation index, aromaticity, etc.

### **Present work on density**

Generally, density measured using helium as the probe gas represents the true density of a coal as the helium molecule is sufficiently small and there is no available volume of pores into which it does not penetrate and van der Waals forces are weak enough to permit helium adsorption on the coal surface to be neglected [8-9]. Though, the densities of coal samples were usually measured using various gases and liquids including hydrogen [16-18], determination of coal density using nitrogen as probe gas is uncommon. We have tried to introduce nitrogen as the probe gas to obtain the true densities of the coal samples because an estimation of the true density using nitrogen is new and convenient one as nitrogen is cheaper than helium as well as an easily available gas.

In this study, densities of a number of coal samples have been measured by utilizing helium and nitrogen as probe gases owing to their almost equal pore accessibility on the coal surface and the closeness of their molecular sizes and inertness to the interaction with coal. Finally, empirical relationships between helium-based and nitrogen-based densities have been developed using two modeling approaches, i.e., multivariable regression (MVR) and the artificial intelligence (AI) based nonlinear modeling technique, namely, artificial neural networks (ANN). Thus, this study provides a comparative study of coal densities measured using helium and nitrogen with a special focus on Indian coals.

## Development of empirical relationships between helium and nitrogen-based densities

### Regression Model

A preliminary MVR analysis of the experimental densities and the proximate and ultimate analyses data was conducted to choose the most influential input variables of the models from a total of six variables, namely, nitrogen-based density and weight percentages of carbon, hydrogen, oxygen, nitrogen and sulfur. Here, models were developed by considering the above-stated six variables. These models were subjected to the statistical analysis (F-test) to choose the best set of input variables. The best model given by the MVR for the experimental data collected on the DAF basis is given below.

$$\rho_t = -3.8213 + 0.6259 * \rho_{t(m)} + 0.0460 * C_O + 0.0445 * C_C - 0.0115 * C_H + 0.0938 * C_N \quad (1)$$

where,  $\rho_t$  and  $\rho_{t(m)}$  in Eq.1 refer to the helium-based and nitrogen-based densities, respectively, and  $C_O$ ,  $C_C$ ,  $C_H$  and  $C_N$  represent weight percentages (wt %) of oxygen, carbon, hydrogen and nitrogen, respectively. The coefficient of correlation ( $r$ ) between the MVR model predicted and experimental values of the helium-based coal densities is 0.93 and the standard error is 0.03 g/cm<sup>3</sup>. It may be additionally noted that most of the coal samples in the present study contain only a small percentage of sulfur (wt%) and lower sulphur content in the coals has no significant effect on the densities as observed in [19]. Hence, wt% of sulfur was also excluded from the input space of the ANN model.

### ANN Model

An MLP neural network based model with two hidden layers was developed for the prediction of helium-based coal densities. The MLP model architecture (i.e., the number of the neurons in the two hidden layers) and its weight coefficients were optimized by following the stepwise procedure given in section 2.3.3. The number of neurons in the optimized MLP architecture are as follows: 5 (input nodes), 4 (hidden layer-I nodes), 5 (hidden layer-II nodes) and 1 (output layer node). The EBP training algorithm-specific two adjustable parameters namely, the learning rate ( $\eta$ ) and momentum factor ( $\mu$ ) were optimized to be 0.2 and 0.1, respectively, and these were kept constant over the entire MLP training phase.

This model can thus be used in practice to predict the helium-based densities without actually using helium for the density determination. The methodology for the determination of the true density using nitrogen as the probe gas in place of the commonly used helium is a new and convenient one owing to the easy availability and lower cost of nitrogen vis-à-vis helium. The above-described approach for estimating the true densities although developed with a special focus on Indian coals is sufficiently general and therefore can be extended to estimate the densities of the coals of different geographical origins provided that these coals have their chemical constituents in the same ranges as those of the coals in the present study.

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## ANNEXURE

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## Coal Carbonization Division

## **Sixty Years Achievement of Carbonization Division**

Central Fuel Research Institute have been directed since its inception 60yrs ago to find solutions of the problems related to fuel and energy in general and coal in particular, arising in the wake of industrial development of India based on coal in the successive five year plans. In the initial period of the institute attention was mainly concentrated to coal related problems related mainly to steel industries. One of the major problems in the steel industries was poor quality of coking coal particularly that of high ash. In view of this major attention of institute's activity was diverted to few coal processing areas such as coal preparation, carbonization, combustion, gasification, and coal based chemicals. At the initial stage most of the work of the institutes was direct and practical help to the industrial advancement of the nation. Subsequently attempts were made in the development of new processes and technologies required during the period with a view to make them more energy efficient, environment friendly, state of the art and economically viable. Coal Carbonization Division contributed significantly to fulfill the above objectives.

In India the reserve of coking coal is very meager. Out of which the upper seams coking coal of very good quality of Jharia and Ramgarh field has been exhausted mostly in locomotives for almost 100 yrs. The available coals of lower seams were highly matured coking coals but lost its coking potentiality due to high ash. As a result, the challenge in front of CFRI was to remove the mineral matter with minimum cost and optimum yield and also to use the optimum per cent of ash in coke making suitable for blast furnace use. Obviously, the attention of coal carbonization division was diverted initially to blend optimization of coke oven feed coal for optimum ash level, use of non-caking coal in the blend and use of low volatile coal in blend.

The institute after a great deal of research and pilot plant scale experiments has suggested suitable blend of indigenous prime coking coal with appropriate amount of sub standard coking coal or weakly coking coal which can produce metallurgical coke of good quality suitable for foundries and steel plants. It was shown that about 20-40% of many weakly coking coals

and a significant quantity of non coking coal with prime coking coal can make a good coke thus contributing to the conservation of best coking coal.

Needless to say that gradual increase in production of steel in 50's,60's & 70's in the country was possible to a great extent due to successful researches carried out in the area of coal carbonization, coal preparation, and RQA. This has resulted in selection of blends for Bokaro, Bhilai, Rourkela and other steel plants.

Further as the demand for coke went on increasing with the limited reserve of coking coal in the country, the carbonization division could not had sound sleep till it developed partial substitutes of coke by making briquetted fuel. The industrial briquettes having properties equivalent to prime hard coke could be made possible by utilization of coke breeze and coal dust. The processes developed in lab scale was tested in pilot plants and subsequently commercialized.

CFRI has carried out extensive experiments on low pressure Briquetting of coal as well as Kashmir Lignite and based on these investigations a 10 Ton/hr plant was installed at Srinagar for Briquetting of Kashmir lignite The plant was to provide acceptable domestic fuel to meet up the demands of the Kashmir valley people. One 1 tph Pilot Plant is in the laboratory for testing the coal for briquette making.

CFRI has developed a pelletization process which in conjunction with a moving bed devolatilized coal produces an excellent smokeless fuel for Industrial/domestic use. Non coking coal slacks, inferior coking coals, coke breeze, char and middling/ rejects and slurries from coal washeries can be processed to produce strong agglomerated fuel using a small proportion of cheap binders at atmospheric pressure .A continuous 1 Ton/hr pilot plant was designed and installed at CFRI

Not only for the industry but in the domestic sector also, carbonization division had to face a great challenge. The domestic fuels used during the period were mainly wood, cow dung cakes and agriculture waste. Due to depletion of forest reserve at a faster rate the availability of wood became very scarce .The depletion of forest imbalanced the ecology of the environment .CFRI

took the challenge of developing domestic fuel by low temperature carbonization. A continuous low temp carbonization pilot plant was installed in the year 1961 of 20-30t /day capacity. A lot of non-coking coals were tested and the technology was commercialized. Based on the test, a LTC plant came at Dankuni though using different technology.

In the domestic sector also CFRI developed small briquettes using low pressure with coal dust and inorganic binder which could be utilized in the domestic cooking stoves. As the manufacture of domestic coke became costlier, through vertical type plant having facility for by product recovery, due to heavy capital investment, the institute diverted to development of non recovery type soft coke oven of very small capacity of 2-2.5 Tons /oven with a production capacity of 50-100 ton/day .The technology was developed in 1995-96 and was commercialized from 1996 onwards. We are very proud to say this technology has earned more than 2 crores by premia which is the highest in CSIR in a single technology and also 1 crore from different consultancy services on that technology. This may also be noted that this technology has earned two prestigious national technology awards of CSIR in 2000 and of NRDC in 2002.

Though the inventions in domestic sector in carbonization division were so lucrative, the development of technology for industrial coke making was also appreciable. In the initial days attention on carbonization division was for the utilization of inferior grade coking coal for coke making as the coke making technology were mostly imported and of by-product recovery type. Subsequently the objectives were diverted towards beehive coke ovens of smaller sizes for making flexibility in coke making. Further sole heated beehive ovens of multi chimney and in later stages that of single chimney was developed. To make the technology more Energy efficient, suitable heating and insulating systems were developed in the non-recovery coke oven in KUMBRAJ ovens.

Coal is converted into hard coke broadly in two types of ovens viz oven without byproduct recovery i.e., non-recovery and those with recovery of by products. Non recovery ovens are also known as beehive ovens. Non-recovery ovens are generally preferred due to less capital investment and

flexibility in operation compared to by product ovens. Non-recovery ovens can be easily started up or closed down as they are made of fire bricks.

KUMBRAJ oven was established with a view to utilize the large resources of low volatile medium coking coals for metallurgical coke making. It was found to be nearly pollution free. Further attempts were made in new generation non-recovery coke oven technology where ovens were made completely mechanized and environment friendly with provisions for utilizing waste heats.

World wide there are various oven types such as Benjamin Thomson Ovens (UK), Hillman Ovens, Jewell Thomsons ovens, Connalsville Ovens, PACTI (All USA), Kembla Ovens (Australia). Sesa Kembla at Goa, INDIA. However CFRI new generation technology is the cheapest.

CFRI extends all necessary expertise right from design, drawing to commissioning of plant to entrepreneurs in setting their non-recovery Coke Oven Plants based on CFRI technology.

Presently studies is on hand to suggest proper utilization of North East Coal in coke making keeping pollution problem in mind, development of stamping technology for non-recovery type coke oven, utilization of waste heat of flue gas for power generation and also other industries etc.

## **CFRI's Achievements on Coal Tar for Carbon Products**

Coal Tar is a by-product of carbonisation process and the same is considered as 'Store House' of organic chemicals as well as various industrial carbon products. Coal tar industries dominated in the chemicals area till the Second World War, after which it lost its glory to petrochemical industry. But under the condition of less availability of petroleum in near future in comparison to coal, coal tar is gaining importance in the chemical and carbon products in particular. Considerable studies have been carried out in CFRI on the gainful utilisation of high temperature/low temperature coal tar. Some important chemicals and carbon products, for which India is still dependent on others, can be obtained from coal tar. The present paper highlights the status of

recovery of coal tar by products and production of industrial carbon products in CFRI.

The temperature of carbonisation affects much on the yield and characteristics of tar. The process of coal carbonisation is of two types, depending upon operating temperature, namely i) low temperature carbonisation (LTC) at 600<sup>0</sup>C , ii) high temperature carbonisation (HTC) at 1000<sup>0</sup>C. The product pattern of carbonization depends on its condition and quality of coal. The principal objective of low temperature process is, production of semi coke or soft coke for domestic purpose and high temperature process for the production of hard coke and coal gas. The typical products of LTC & HTC are shown in the following table.

**Table-1**

	Semi coke/coke%	Tar%	Gas%
LTC	70-80	5 - 9	124-193
HTC	78-80	2-3	300-350

Coal carbonization in a by product oven (HTC) is an essential part of integrated steel plant. So steel plants remain one of the main sources of tar. The tar production during late nineties from different places are given below,

**Table - 2**

<u>Place</u>	<u>Production</u>
<b>HTC Tar</b>	
India	0.4 million ton/y
World	16.0 million ton/y
SAIL	1420 t/d
Other Industries	230 t/d
<b>LTC Tar</b>	
Dankuni	70-80 t/d
Neyveli	0.64 million ton/d

Coal tar from HTC is one of the main source for aromatic chemicals like BTX, naphthalene, anthracene, phenanthrene, carbazole, acenaphthene, pyrene, etc. The property of LTC tar is different from that of HTC tar. LTC tar is primary in nature and contains more alkylated compounds than HTC tar which is more aromatic in nature; because at high temperature, the primary tar, which evolves just after charging coal, undergoes some secondary reactions to produce more aromatic compounds containing two to four rings. The average composition of tar and their variations depending on the nature and type of carbonisation are presented in Table-3

**Table 3 Major important compounds in tars of different origin**

<b>Sl. No.</b>	<b><u>Compounds</u></b>	<b><u>Average % on tar,</u></b>	<b><u>Average % on tar,</u></b>
		<b>HTC</b>	<b>LTC</b>
1.	Benzene	0.10	0.015
2.	Toluene	0.03	0.204
3.	O-Xylene	0.03	0.121
4.	m- Xylene	0.07	0.232
5.	p-Xylene	0.03	0.090
6.	Styrene	0.02	Traces
7.	Ethylbenzene	0.03	0.100
8.	Phenol	0.50	1.52
9.	o-Cresol	0.26	1.60
10.	m-Cresol	0.32	1.55
11.	p-Cresol	0.22	1.36
12.	Xylenols	0.40	7.30
13.	HBTA	0.80	15.78
14.	Tar bases	2.00	2.08
15.	Naphthalene	8.00	0.66
16.	1-methylnaphthalene	0.60	0.24
17.	2-methylnaphthalene	1.20	0.20
18.	Diphenyloxide and its homologues	1.30	
19.	Anthracene	0.90	0.06
20.	Phenanthrene	5.00	1.1
21.	Carbazole	1.50	1.28
22.	Pitch	60.00	30.00

Out of these, five groups of compounds including pitch have been identified, which are important and could be gainfully recovered from HTC and also from LTC coal tars as evident from Table – 4 with an average composition.



**Table 4**

<b><u>Products</u></b>	<b><u>HTC</u></b>	<b><u>LTC</u></b>
	% on tar	% on tar
BTX	0.26	0.66
Phenols, Cresols, Xylenols	1.70	13.42
Naphthalene	8.00	0.66
Me-naphthalene	1.80	0.44
Anthracene, Phenanthrene, Carbazole	7.40	2.50
Residue/Pitch	60.00	30.00

There are processes to recover major components of tar and to enrich some components by catalytic processes. CFRI has also worked on recovery and purification of some compounds from coal tar. Distillation of tar is the first step to get the concerned fraction of any compound. Table - 5 shows the distillation range for some compounds.

**Table 5**

<b>Tar Fractions, °C</b>	<b>Chemicals Present</b>
1. Light Oil, upto 210	BTX, T.Acids,T.Bases,Sol.Naphtha
2. Middle Oil, 210-230	T.Acids,T.Bases,Naphthalene
3. Me-Naphthalene, 230-270	Higher substituted Naphthalene
4. Light Creosote, 270-315	Acenaphthalene, Diphenylene oxide, Fluorene
5. Middle Creosote, 315-360	Phenanthrene,Anthracene,Carbazole
6. Pitch, >360	Chrysene,Fluoranthene,Pyrene, + high mol.wt. containing compounds

## **Recovery of compounds in tar oil fractions**

From motor benzole, enrichment and recovery of benzene was possible at CFRI through hydrodealkylation. CFRI had shown that high temperature tar fractions boiling in the range 230-300°C are quite effective for extraction of benzole from coke oven gases. The performances of LT tar fractions were

found to be similar. These fractions were found to be superior to petroleum wash oils.

CFRI has carried out extensive work on the recovery and utilization of tar acids. Dealkylation of high boiling tar acids into more useful lower phenols was studied by catalytic cracking and hydrogenolysis.

There is good demand for naphthalene for use as such material and for synthesizing other compounds. One of these compound is phthalic anhydride. CFRI studies led to the development of a process which could give naphthalene of more than 99% purity with high recovery from tar. The process involved was solvent refining and has been patented.

The possibility of direct oxidation of naphthalene oils to phthalic anhydride has been examined in detail at CFRI which is covered under Indian patent.

The importance of anthracene lies with the use of anthraquinone in dyestuffs. It is worthwhile to process anthracene oil fraction for recovery of anthracene. CFRI has developed a solvent purification process whereby a crude anthracene paste containing 20% anthracene could be treated to obtain anthracene of more than 95% purity at high recovery levels and patented. Catalytic oxidation of anthracene to anthraquinone has also been studied with 90% yield.

Next to naphthalene, phenanthrene is the most abundantly occurring hydrocarbon in HTC tar which is around 4-6% and is separated alongwith anthracene and carbazole in the anthracene cake. Phenanthrene, being used in the pesticide industry, has a great demand in India. CFRI has separated phenanthrene through solvent extraction with a purity of 98% (guarded under patent). 9:10 Phenanthraquinone has good demand in the dyestuff industry and as a selective herbicide. The oxidation reaction of phenanthrene to phenanthraquinone has been studied where 98% pure phenanthraquinone has been obtained (Indian patent).

The neutral oil of LTC tar is less aromatic, containing very little naphthalene, anthracene and much paraffinic and naphthanic bodies compared to HTC tar. Although an appreciable portion of it can be processed for diesel oil, it can be better utilized by hydrogenation. Hydrogenation was carried out at CFRI in

batch type autoclave as well as in a continuous bench-scale reactor. It has been observed that in the batch process, 200-250<sup>0</sup>C and 250-300<sup>0</sup>C fractions can be converted to diesel type fuels using CFRI developed catalyst at 400<sup>0</sup>C, 226kg/cm<sup>2</sup> hydrogen pressure with reaction time of 3 hrs. In the continuous process, a low pressure of 50 kg/cm<sup>2</sup> was sufficient, but the diesel oil produced was of inferior type (low speed type). It was possible to upgrade the diesel oil like product by removal of aromatics by sulphuric acid extraction. The fraction 300-350<sup>0</sup>C was found unsuitable for hydrogenation, as a result cracking was attempted using SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> catalyst and an active carbon catalyst. A product boiling below the initial boiling point of the feed could be obtained. The aromatic content of the product increased steadily while the content of paraffins and naphthenes decreased. It is, therefore concluded that for the production of diesel type of fuel, the cracking alone has no scope, it can be adopted for the production of simple aromatics or a feed for hydrogenation to diesel oils.

**Pitch:** The annual demand of coal tar pitch, during late nineties, in India was 1,20,000 tons.. A part of which is converted to coal tar fuel (CTF) and burnt at steel plants. The rest is mainly used in aluminium industries for making electrodes .

CFRI has done extensive work to modify pitch quality according to the needs of industries. Four products from coal tar pitch have been prepared by CFRI, namely Mesophase pitch, Needle coke, Binder pitch, Low Quinoline Insoluble (QI) pitch.

**Mesophase Pitch:** Carbon fibre is the material of modern age. It can be used in different areas such as Sports goods, Automobile industries, Air crafts, Space crafts, Biomedical application etc. The feed for carbon fibre was mostly produced from petroleum products in western countries. HTC tar is a potential source for production of Carbon fibre due to high aromatics content. CFRI has developed the special type of pitch called Mesophase pitch as feed for Carbon fibre from commercial coal tar. The project was completed successfully and appreciated by Indian Space Research Institute (ISRO) as funding agency. The characteristics of a typical mesophase pitch, prepared at CFRI are given below.

**Table 6                      Quality of Mesophase Pitch Produced at CFRI**

Toluene Insoluble	-	59.1 (wt%)
Quinoline Insoluble	-	29.0(wt%)
Coking value	-	68.3 (wt%)
Soft. Pt.	-	200°C+
Mesophase Content	-	41.6(vol%)

**Needle Coke:** In the area of Needle Coke CFRI has contributed developmental work through processing of tar from different sources and subsequent coking of the same . These cokes are used as a grist material for electrode used in chemical and electrochemical industries. Both LTC and HTC tar from different sources were explored and revealed that Pitch Coke/ Needle Coke of desired properties and optical texture could be achieved.

**Binder Pitch:** CFRI has developed binder pitch from commercially available tar and modified inferior grade tar by thermal treatment in presence or absence of air to improve its binder characteristics such as coking value, softening point, solubility of different solvent, density, viscosity etc. for use in Soderberg electrode of aluminium industries. Development of pitch bonded Refractory Block (dolomite& Magnesite)was achieved with suitable treatment of tar which were tested at Bokaro Steel Plant.

**Low QI pitch:** Quinoline Insoluble material of tar is unwanted for use the tar in producing value added products mentioned above. QI content of HTC tar is in the region 4-10 (wt %). The pitch prepared from this quality of tar has QI content around 7-16(wt %). The QI content of Pitch should be less than 1% if it has to be employed for producing value added carbon product, particularly mesophase pitch. CFRI has succeeded to achieve it using a low cost solvent.

### **Latest Non-recovery type coke oven constructed under consultancy Project**

1. M/S. Usha Fuels, Raipur, Capacity – about 5000 tons per month per battery. (Fully Mechanized)
2. M/S. M.V International, Kuttack, Capacity about 4000 tons per month (partially mechanized).

3. M/S. Uluberia Coke Plant, Uluberia, West Bengal, Capacity- about 4000 tons per battery per month ( partially mechanized)
4. 4. M/S. Aroma Coke Plant, Dhanbad Capacity- about 3000 tons per battery per month ( partially mechanized)
5. M/S. Arun Fuels, Chirkunda - Capacity- about 3000 tons per battery per month (partially mechanized)

### **Future Plan of Carbonization Division**

Coal Carbonization is CFRI's forte. Even though a good deal of R & D work has been done in the past, there is ample scope for further research, especially in the area of characteristics of blended coals using soft coal for coking; assessment of low, medium and high temperature carbonization characteristics of coals for the production of domestic and metallurgical fuels; development of suitable additives for improving the plastic properties of low grade coals ; Briquetting characteristics of high ash ,low grade bituminous coals, lignite, and washery by-products; production of metallurgical grade formed coke from non-coking coals; Development of low capital , energy efficient non-recovery type coke ovens for metallurgical as well as domestic purpose. Research initiatives will be focused in an emerging area for the utilization of non-coking coals for making coke by developing technology for stamping the charge.

Emphasis will be laid on utilization of NE coal for briquette making and also coke making directly by directly higher temperature carbonization and also for simultaneous desulphurization.

Development of Value Added Carbon Products derived from coal through Aqua Conversion Process as Clean Coal Technology.

The material for future is Carbon in its different form, fibre or carbon-carbon composites. In view of limited resource of petroleum crude, the source for carbon is coal. The availability of tar is restricted as it is injected directly in the blast furnaces with coke. The possibility of extracting pitch like material from coal rather than tar attracts scientists throughout the world. Till today only West Verginia University of USA has done some work to prepare mesophase pitch through Solvent Refined Coal (SRC) technique. They have used costly

solvents and high pressure systems to extract this pitch like material. To avoid costly process our programme is chalked on the basis of knowledge generated through development of Coal Water Emulsion (A Clean Coal Technology) and production of Mesophase Pitch at CFRI.

Assessment on the potentiality of the extraction of higher aromatic compounds from coal tar generated through carbonization of coal of different rank and evaluation on their suitability for specific chemicals.

Existing market for higher aromatic chemicals leads us to take up the project. At present tar is generated in coke ovens where different blends of coal, made of different ranks, are used. To visualize a clear picture of tar towards recovery of aromatic chemicals, coal tar from different rank of coal is worth to study.





## ANNEXURE

### 1. List of Publication

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| 6.011 | Assessment of Coke Quality : Indices for Physical Properties<br><i>FRI News</i> , <b>5</b> (2), 46 (1955)<br><i>Ind. Min. J.</i> , <b>4</b> , 1-4 (1956) | Das Gupta, N.N.<br>Das Gupta, A.K.<br>Roy Chowdhury, A.K. |
| 6.012 | Coal Price and Industry<br><i>FRI News</i> , <b>5</b> (4), 98 (1955)   | Lahiri, A.<br>Ghosh, A.<br>Bose, R.N.                     |
| 6.013 | Selection of Coal for Coking<br><i>Ind. Min. J.</i> , <b>3</b> (6), 1 (1955)   | Das Gupta, N.N.<br>Ghosh, S.R.                            |
| 6.014 | Reactivity of Coke<br><i>Trans. Inst. Chem. (Ind.)</i> , <b>28</b> (3), 281 (1956)   | Rao, V.V.<br>Das Gupta, N.N.                              |
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| 6.306 | Coal – Tar A feed stock for value added products<br><i>National Conference on Carbon(1996), DMSRDE , Kanpur</i>  | A.K.Choudhury,<br>U.Bhattacharjee<br>and Ms.<br>A.Bhattacharya  |
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| 6.312 | Modeling for suspension of coal in Multi – Phase reactor system – A statistical Approach<br><i>Fuel, Vol. 77, No.14, 1998</i>  | A.Mukherjee,<br>S.K.Hazra and<br>P.Bandopadhaya   |
| 6.313 | Problem of inferior coals and its utilization as domestic fuel<br><i>National Seminar on Beneficiation of Coals and Minerals, MCL, Sambalpur, 29<sup>th</sup> Jan. 2000</i>                      | P.N.Sinha,K.S.Bhattacharjee,<br>P.Sengupta,   |
| 6.314 | Alternative base material and binder for formed coke making<br><i>Seminar of energy development and environment safety in the new millennium: Problems and solutions, 6-7 November 2000 CFRI</i> | A.k.Sharma ,<br>B.P.Das   |
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| 6.317 | Modern trends in coal carbonization its environmental and economic Impact<br><i>All India seminar for coal carbonization for the production &amp; marketing of Town Gas and coal chemicals, 14<sup>th</sup> Feb, 2004, Dankuni Coal Complex, Hooghly</i> | S.K.Hazra,<br>A.K.Sharma,<br>R.Dasgupta,<br>S.C.Roy and<br>K.Sen  |
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| 6.322 | Development of a process for producing environment friendly fuel from High Sulphur-High ash Lignite for Heat & Power Generation,<br><i>International Symposium on Clean Coal Initiatives,Delhi,22-24 Jan.1999</i>  | Srivastava S.K,<br>Hazra S.K,<br>Mukherjee A.<br>and Singh A.P.   |
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| 6.326 | Sustainable Technologies for Domestic Fuels vis-à-vis Environmental and Economic Impact,<br><i>Seminar on Environmental Challenges of Jharkhand, CFRI,Dhanbad,18 Feb.2002</i>  | Hazra S.K   |

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| 6.327 | Potential of producing Briquetted Fuel for domestic Uses from Myanmar coal.<br><i>National Seminar on Coal Science &amp; Technology: Vision 2020.CFRI Dhanbad 20-21 April 2003.</i>                             | Sinha P.N, Ranjan R, Yadav R.S, Choudhury N, Sengupta P, & Hazra S.K. |
| 6.328 | Natural Coke (Jhama) of Jharia Coalfield: a Critical Appraisal of industrial Use<br><i>National Workshop on Re-inventing Jharia Coalfield,MGMI,Dhanbad Chapter 22-23,Aug 2003</i>                               | Singh A.K, Sen k, Choudhury N, & Hazra S.K.                           |
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| 6.330 | Enrichment of Carbon in Natural Cokes(Jhama):A Prospective Source to Carbon Industry<br><i>National Seminar on Carbon ,DMSRDE,Kanpur,26-27 Nov 2003</i>   | Singh A.K, Singh Vivek, Yadav R.S , Choudhury N & Hazra S.K           |
| 6.331 | Coal Derived Carbon- a new hope to Carbon Based Industries,<br><i>Indian Carbon Society, NPL, New Delhi, 25-26 Nov. 2004.</i>   | Singh A.K, Choudhury N & Hazra S.K                                    |

## **2. List of Reports (from 1995)**

### **1995 – 96**

1. Beneficiation and Carbonization of Joyrampur VI Seam Coal of Jharia Coalfield (Sponsored by BCCL, Dhanbad).
2. Beneficiation and Carbonization of Rajpur V,VI, VII,VIII Seam Coal, Jharia Coalfield (Sponsored by BCCL, Dhanbad).
3. Studies On Production of Briquette Fuel for Domestic Uses from Washery Byproducts/Low grade Coking coal (Sponsored by BCCL, Dhanbad).

### **1996 – 97**

1. Feasibility report on the potentiality of CPC/RPC for production of briquetted fuel for industrial/ metallurgical uses
2. Technical audit of coke ovens and by-product plant at Durgapur Projects Limited,
3. Utilization of Botswana Coal for ECUP, Botswana.
4. Reports on Beneficiation and Carbonization of Dhoru Coals.(Seam VI/VII/VIII)
5. Technical Audit of Tar Acid Recovery Of Dankuni Coal Complex.
6. Technical Audit of Retorts of Dankuni Coal Complex for High Gas Yield.
7. Utilization of CIL Coke in CFRI developed chullah.
8. Oven for Soft Coke at Hill Briquette Industries.

**1997 – 98**

1. Studies of coal tar partially distilled produces at DSP for its suitability as Boiler fuel, **Durgapur Steel Projects, Durgapur .**
2. Beneficiation and Carbonization of Dhori Coals
3. Devolatilization of Coals of Mahanadi Coalfield.
4. Beneficiation studies on lignite Devolatilization and desulphurization.
5. Techno economic feasibility studies on Devolatilization and desulphurization of lignite for its use in Indian Cement.
6. Technical audit of DPL Coke Oven.
7. Construction of 24 No. of Soft Coke Oven for Rajive Fuel Products.
8. Recoverable waste heat from raw petroleum calcite Kiln.
9. Feasibility on the Potentialities of CPC/RPC for production of Briquetted Fuels.
10. Studies of Coal Tar Partially Distilled Produced at DSP for its Suitability as Fuel in Boilers.

**1998 – 99**

1. Report on Beneficiation and Carbonization of Lodna Coals.
2. Report on Coking Potentialities of washed Lower Seam Coals of munidih colliery.
3. Interim Report on Soft Coke Ovens for Rajhans.
4. Report on Power Potentialities through waste heat utilization from existing Non-recovery Coke Oven in and around Dhanbad.
5. Reoprt on erection and commissioning of Soft Coke Ovens for Shivan Industries.

**1999 – 2000**

1. Reports on Erection and commissioning of coke oven for Delta Coke Pvt. Ltd.
2. Development of Technology for the Production of additives based on Coal Tar products for Coal Water Slurry Fuels.

**2000 - 2001**

1. Technical audit and cost estimation of coke oven for TATA Metalics Ltd.
2. Construction / supervision of soft coke oven ( 24) with pusher system for discharge products
3. studies on the utilization of Coal from Ib valley Coalfield for Soft Coke making and their undersize for other domestic fuels after Blending.
4. Report on Study of the Devolatization characteristics and Pilot Oven Products Characteristics of Two Coals for M/s Vinay Cement Limited.
5. Potentialities of making Soft Coke/Briquettes from Talcher Coal of mahanadi Coalfields.
6. Report on Identification of best sources of Coal and Condition to obtain Tar suitable to Obtain Mesophase Pitch required for Special Application Like carbon Fibres.

**2001 - 2002**

1. Utilization of Coke Breeze for making tailored made Fuel in GHCL Lime Kiln.

2. Development of Binder for making briquette from undersize coal fines of Mahanadi Coalfields Ltd.
3. Assessing the Potential of Utilizing Coke Breeze and Unburnt Carbon From Boiler for Utilization in Lime Kiln.
4. Report on Comparison of Gases Emitted in Stack by burning Flame Di-isocyanate (TDI) Tar Powder in Non-Recovery Type Coke Oven.
5. Development of Process for Cooling Soft Coke Produced from MCL coals by dry quenching.

**2002 – 2003**

1. Report on erection, commissioning and their run of Briquetting plant at Gujrat Heavy Chemicals Ltd.
2. Assessing the Potentialities of Myanmar coals for production of domestic fuel.

**2003 – 2004**

1. Report on assessing the potential of utilizing the coke breeze of TCL for Utilization in Lime Kiln of TATA chemicals Ltd.
2. Report on assessing the coking potentialities of imported Soft / Hard coals in blends with different Indian coals in various proportions in Pilot Scale
3. Report on design & drawing for Briquetting Plant for TATA Chemicals Ltd.

**2. List of Patents (From 1995)**

**1995 – 96**

A Process for the Production of Low Ash Fuel Alternative to Byproduct/Beehive Coke for Industrial/Metallurgical Purposes using Calcined Petroleum Coke with or without Mixing any other Carbonaceous Materials such as Coke Breeze.

P.N. Sinha, K.S. Bhattacharjee & P. Sengupta.

**1996 – 97**

A Process for the Production of smokeless easily ignitable pellets from carbon dust on lumps.

S.K. Ghose, S.K. Mukhopadhyay, S. Gupta, S.R.K. Rao, M. Chakraborty, and R. Haque.

**1997 – 98**

1. A device for production of soft coke and process thereof  
S.K.Hazra and P.Sengupta
2. A Process for Production of Coking agent having Low ash high fluidity(thermoplasticity) useful for blending with substandard Coals to make standard coke (PATENT No. 177069)  
Dr. M. Chakraborty and Dr. R. Haque.
3. A Direct Reading device for measuring resistivity of particulate Coke or Allid carbonaceous materials (Patent No.176522)  
A.K.Roy, M.C.Das, S.K.Mazumdar and R. Haque.

**1998 – 99**

A process for the production of low ash fuel using calcined petroleum coke and low ash fuel prepared thereof.

P.N.Sinha, K.S.Bhattacharjee, P.Sengupta

**1999 – 2000**

A process for the production of low ash fuel alternative to by-product/ beehive coke for industrial/ metallurgical purposes using calcined petroleum coke with or without mixing any other carbonaceous materials such as coke breeze

P.N.Sinha, K.S.Bhattacharjee and P.Sengupta

**2001 – 2002**

1. A process for the production of low ash fuel  
P.N.Sinha, P.Sengupta & K.S.Bhattacharjee
2. A process for Devolatilization of Lignite and an equipment therefor  
P.N.Sinha, P.Sengupta , K.S.Bhattacharjee & Dr. S. K. Hazra.
3. A process for manufacture of Active Carbon from Coke Breeze, useful for removal of toxicants from effluent water of Coke Plants.  
S.N. Mjumdar, Dr. J.Singh, S.K.Basu, P. Bhattacharya & Dr. Kalyan Sen.

**2002 – 2003**

A process for the production of Low Ash Fuel using calcined petroleum coke and low ash fuel thereby .

P. N. Sinha, P. Sengupta and K. S. Bhattacharjee ( in USA & France).

**2003 – 2004**

Process for removal of organic sulphur from high sulphur coal

A.Ali, A.Bhattacharya, S.K.banerjee, D.N.Tiwari,S.K.Hazra and R.S.Yadav

**2004- 2005**

A process for devolatization of Lignite and an equipment thereof

P. Sengupta. P.N. Sinha, K.S. Bhattacharjee and S.K.Hazra

**3. Technologies Transferred (from 1995)**

**1995 – 96**

Improved Beehive Oven (CFRI-TISCO Design): released to M/S I.B. Industries Ltd., Calcutta (2.2.96).

**1996 – 97**

1. Improved Beehive Oven (CFRI-TISCO Design) transferred to M/S Shree Mahavir Carbon Ltd., Cuttack on 12.06.96.
2. Oven for production of Coke for domestic uses (Soft Coke) transferred to M/S.

- a) Hill Briquette Industries, Bhuinore, Dhanbad on 07.06.96.
- b) Delta Coke Co. Gaighata More, Haripur, Burdwan on 07.10.96.
- c) Rajiv Fuel Product, Varanasi on 11.03.97.
- d) Pratap Fuel Industry, Chandausi, Varanasi on 14.03.97.
- e) Mayank Fuels, Varanasi on 17.03.97.
- f) Super Fuels, Hazaribag on 19.03.97.
- g) Jyoti Fuels Pvt. Ltd. Dhanbad on 26.03.97.
- h) Mukherjee Coal Traders, Rukni More, Purulia on 19.03.97.
- i) Renu Industries, Dhanbad on 27.03.97.

**1998 – 99**

About Seventy entrepreneurs from Bihar, West Bengal, Uttar Pradesh, and Madhya Pradesh entered into an agreement with CFRI for the license of Know-how of oven for Soft Coke Production.

**2001 – 2002**

- 1. Soft Coke Technology Transferred to-
  - a) M/s Diamond Soft Coke Manufacturing co., Domhani Bazar, Burdwan on 23.04.01
  - b) M/s Achchal enterprises, Charhi, Hazaribag on 18.07.01.
  - c) M/s Promac Engineering Industries Ltd. Anjanapura, Bangalore on 11.09.01.
- 2. For setting up a coal devolatilization Plant in Nigeria

**2002 – 2003**

- 1. A device for production of soft coke and process thereof licensed to M/s Bhagwati Fuels, Chandauti More, Gaya on 22.8.02.
- 2. A device for production of soft coke and process thereof licensed to M/s Hindustan Sizing Coal Centre, Santaldih, P.O. Afzalpur, Jamtara on 18.11.02
- 3. A device for production of soft coke and process thereof licensed to M/s Maa Durga Fuel Coke, Chauk Keshabganj, Kalipahari, Burdwan on 22.11.02.
- 4. Technology for Briquetted Fuel for Domestic uses to M/s Black Diamond Coal Briquette Industry, Kalyaneshwari, Kulti, Burdwan on 28.11.2002.
- 5. Technology for Briquetted Fuel for Domestic uses to M/s Alo Coke Industries, Dabor, Achra, Burdwan on 24.01.2003.
- 6. Technology for Briquetted Fuel for Domestic uses to M/s Rakhakali Enterprise, Amdiha, Lalganj, Burdwan on 24.01.2003.
- 7. Technology for Briquetted Fuel for Domestic uses to M/s Singal Fuel Coke & Briquette Coke Industries, Jamuria Bazar, Burdwan on 31.03.2003.

**2003 – 2004**

- 1. A license agreement was made with M/s United Fuel Coke, Burdwan on the process for manufacturing smokeless Briquetted Fuel from Low Grade Caking coal/washery by Products on 18.06.2003.

2. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S Uluberia Coke Oven Plant , 2, Barrotto Lane, Ist Floor, Suite No. 105, Kolkata -700069 on 14.07.03.
3. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S M.V. International Ltd., 1, Crooked Lane ,Room No. 104, Kolkata -700069.
4. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S Narayani Coke Private Ltd, B/1 Umiya Mahal, Plot 11, Sector 1, OSLO Road, Gandhidham 370201, Gujrat on 09.12.2003.
5. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S Chaman Metallicks Pvt. Ltd. R-9, Yugdharm Complex, 27, Central Bazar road, Radaspeth, Nagpur- 441010 on 31.12.2003.
6. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S Shri Ramrupai Balaji Steels Ltd, 5, Bentick Street, Ist Floor, Kolkata – 700001 on 31.01.2004.
7. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S Usha Fuels Pvt.Ltd., Ist Floor, Super Market, Dhanbad on 11.03.2004.
8. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S Keshav Casting Pvt. Ltd JVSL Old Administration Building, P.O. Vidyanagar- 583275, Toranagallu, Dt. Bellary, Karnataka on 31.03.04.
9. Process: Design Know-how for setting up a battery of Improved Beehive Ovens (CFRI-TISCO Type) for manufacture of Coke with M/S Bengal Coke Company Ltd. Sikkim Commerce House , 4/1 Middleton Street, Kolkata -700071 on 31.03.04.
10. Process: Manufacture of Smokeless Briquette Fuel from Low Grade Coking Coal/washery by products with M/s Maa Durga Enterprise Vill+PO: Dakshinkhanda, Burdwan, WB on 31.03.04.
11. Process: Manufacture of Smokeless Briquette Fuel from Low Grade Coking Coal/washery by products with M/s Maa Manasa Traders, Burdwan West Bngal on 10.12.2003.
12. Process: Setting up of Ovens for Production of soft Coke for Domestic uses with M/s Maa Durga Enterprise Vill+PO: Dakshinkhanda, Burdwan, WB on 31.03.04.
13. Process: Setting up of Ovens for Production of soft Coke for Domestic uses with M/s Ghosh Enterprise , Chora,Bahula, Bardwan-713322, WB on 05.03.04.

**2004 – 2005**

1. Process: Manufacture of Smokeless Briquette Fuel from Low Grade Coking Coal/washery by products to the firm National Fuels, Patthar Khad, PO: Kendua , Kulti- 713343, Burdwan (WB) on 15.04.04.



2. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm Metcoke Industries Ltd. Honnarahalli, Siriguppa TQ, Bellary, Karnataka on 11.05.04.
3. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm A.R. Co-Extruded Films Ltd., A-2/452, Sector-8, Rohini, New Delhi on 22.07.04.
4. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm Prime Coke Industries, Barua Market , 1st Floor ,fancy Bazar, Gauhati, Assam on 18.08.04.
5. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm Debenjana Hard Coke Pvt. Ltd. P.O. and Vill-Andalgram, Burdwan (WB) on 28.08.04.
6. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm GL Coke Pvt. Ltd. , 2<sup>nd</sup> floor, State bank of Bikaner and jaipur Building , SRCB Road , Fancy Bazar , Gauhati on 15.09.04.
7. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm Meghalaya Coke & Chemicals (P) Ltd., Fame Building, Lutramby Jantiya Hills, Jowai, Meghalaya on 15.09.04.
8. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm Kirloskar Ferrous Industries Ltd., Laxmanrao Kirloskar Road, Pune- 411003 on 07.04.04.
9. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm K.D.Cokes, Lohia House, MG Road, Guwahati, Assam on 19.10.2004.
10. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm Pride Coke Pvt. Ltd. Harlalka Bhavan, Opp. Shanti Sabha, Guwahati, Assam on 19.10.2004.
11. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm JDB Coke, GS Road, Jorabat, Kamrup, Assam on 28.10.04.
12. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm ACM Fuels Pvt. Ltd., 210 Shanti Bhavan, Bank More, Dhanbad on 03.12.04.
13. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for manufacture of Coke to the firm Tycoons Industries Pvt. Ltd., British Indian Street, Kolkata on 13.12.04.
14. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens for to the firm Jai Coke Industry, Silchar, Assam (CFRI-TISCO) Design for manufacture of Coke on 28.01.05.
15. Design: Design Know-how for setting up a battery of Improved Beehive Coke Ovens (CFRI-TISCO) Design for manufacture of Coke to the firm Avija Coke Pvt. Ltd., 55, Mahadevpur, Whitefield Main Road, Bangalore on 09.03.05.

16. Process: Manufacture of Smokeless Briquette Fuel from Low Grade Coking Coal/washery by products to the firm Maa Durga Enterprise Vill & P.O: Ethora, Burdwan (WB) on 26.04.04.
17. Process: Manufacture of Smokeless Briquette Fuel from Low Grade Coking Coal/washery by products to the firm Shyam Sunder Briquette Industries, Gourangdi, P.O. Panuria, Burdwan, W.B. on 13.07.04.
18. Design Know-how for setting up of Soft Coke Plant of 10 ovens to the firm Economical Domestic Fuel Enterprises, Baralal Street, Upper Bazar, Ranchi- 834001 on 01.07.04.
19. Design Know-how for setting up of Soft Coke Plant of 10 ovens to the firm Shiv Fuel Products , Sandi , P.O.: Barechnagar, Hazaribag on 20.07.04.
20. Design Know-how for setting up of Soft Coke Plant of 12 ovens to the firm Okasha Coke Industries, Vill- Paiki, P.O.: Digwar, Hazaribag, Jharkhand on 14.10.04.
21. Design Know-how for setting up of Soft Coke Plant of 10 ovens to the firm Jai Maa Chhinamastika Fuels, Chitarpur Mile, Hazaribag, Jharkhand on 01.02.04.
22. Design Know-how for setting up of Soft Coke Plant of 10 ovens to the firm B.M.Coke & Minerals, Kuju, Hazaribag, Jharkhand on 17.02.04.
23. Process: Manufacture of Smokeless Briquette Fuel from Low Grade Coking Coal/washery by products to the firm Sree Ganesh Fuel Industries, Village and P.O.; Jamgram, Burdwan, (W.B) on 13.07.04.
24. Process: Manufacture of Smokeless Briquette Fuel from Low Grade Coking Coal/washery by products to the firm Golden Briquette Industries Vill:- Khosnagar, P.O: Domohani Bazar , Burdwan (WB) on 28.08.04.
25. Design Know-how for setting up of Soft Coke Plant of 10 ovens to the firm Rajrappa Enterprises, Vill: Twardag, P.O: Chitarpur, Ramgarh, Hazaribag, Jharkhand on 28.08.04.
26. Design Know-how for setting up of Soft Coke Plant of 10 ovens to the firm Omkar Fuels,vill:- Khakhi Kala, P.O: Dumri, Nimiaghat,Giridih, Jharkhand on 10.09.04.

#### **4. MoU Signed**

1. MOU was Signed for Setting up a coal slurry beneficiation Plant with M/s maithan Coal Company(P) Ltd. Registered Office: Room No. 408, 4<sup>th</sup> Floor,Jhilmil Chambers,P-44, Rabindra Sarani, Kolkata-700013,Administrative Office: P.O. Chirkunda- 828202,Dist: Dhanbad on 01.07.03.
2. MOU was Signed for development of Higher version of Non-recovery Coke Oven For the Manufacture of Metallurgical/ Industrial Grade Coke with M/s Ecocoke & Power Pvt. Ltd. 14, Singapore Garden, Doddakallasandra, Kanakpura Road, Bangalore- 560062 on 27.11.2003.

## Coal Chemicals Division

# **R & D activities on Development of chemical processes undertaken at CFRI**

## **Introduction**

Petroleum, coal and Natural gas are the major source of carbon, which have been exploited extensively for the development of energy and chemicals for use of mankind on the earth. The present day energy crisis is due to the non-availability as well as price escalation of petroleum. In this context, the techno-economic prospects of replacement of petroleum by coal/natural gas or other carbonaceous materials as a source of feedstock for chemicals has assumed great importance.

Coal is the alternative source of carbon next to petroleum, which can more profitably be used, in the form of chemical feedstock, after its conversion. For the proper use of the byproducts of coal carbonization and also to valorize the products, a number of organic chemical processes have been developed at CFRI, Dhanbad. Number of organic compounds like BTX, naphthalene, anthracene and phenanthrene etc in neutral fraction and phenolic compounds in acidic fraction are available considerably and bases like pyridine bases and quinoline bases are also present in meagre quantity in basic fraction. Synthesis gas obtained from gasification of coal/biomass and also from methane (natural gas/CBM) reforming is a major feedstock for production of organic chemicals and fuels.

Hence, Development of chemical processes based on compounds of coal carbonization byproducts and methanol/synthesis gas as well as methane have been carried out. These process development work comprised (i) development of catalysts, (ii) optimization of reaction parameters (iii) characterization of products and catalysts (iv) scale up studies of the selected process for generating data for transfer of technology.

## **1. Development of chemical processes based on aromatic/heterocyclic compounds**

### **1.1 Aromatic hydroxy compounds based on coal carbonization by-products**

Among the aromatic hydroxy compounds, cresols, resorcinol and beta-naphthol are the most important due to their ever-increasing demand in the

rapidly growing chemical sectors, which include drugs, dye-stuffs, resins, pharmaceuticals, agrochemicals, tyre industries etc. Hence, with a view to meet the future requirements of these chemical industries, the Central Fuel Research Institute, Dhanbad has developed cost effective, environment friendly and performance driven processes for the production of cresol (para- and meta-/para-mixture) resorcinol and beta-naphthol from toluene, benzene and naphthalene respectively

### **1.1.1 Process for the Production of Cresols**

#### **(a) Industrial application**

Cresols are extensively used in the manufacture of resins, soaps, wire enamels, plasticizers, disinfectants, antioxidants and agricultural chemicals. The major consumer of meta-para-cresols mixture is the phenolic resin industry who prefers cresols containing 50% meta-cresol. Para-cresol is a basic raw material for the production of 2, 6- tert-butyl para cresol (DBPC), a widely used antioxidant with an extensive range of applications.

Thus para-cresol as well as meta-para cresol mixtures have a wide range of applications and their demand is increasing day by day in the country. The production of cresols is inadequate to meet the demand and therefore, there are ample scopes and possibilities for setting up commercial units for the manufacture of cresols in the country.

There are four routes for the synthesis of cresols, namely sulphonation route, chlorination route, p-cymene route and phenol methylation route. The raw materials requirements, technology and above all considering the suitability in Indian conditions, the sulphonation route has been chosen.

#### **(b) Process for the production of cresols**

Toluene is sulphonated with 98% sulphuric acid to produce predominantly para-sulphonic acid. The sulphonation mixture is subjected to isomerisation reaction when considerable quantity of para-sulphonic acid and entire quantity of Ortho-sulphonic acids are converted into meta-sulphonic acid. The acid mixture is neutralized with caustic soda and sodium sulphite to obtain sodium sulphonate and sodium sulphate. Sodium sulphonate is separated from sodium sulphate by fractional crystallisation and fused with caustic soda at around 150°C. The fused melt is quenched with water, acidified and cresols are isolated by layer separation and /or by solvent extraction. The mother

liquor is worked up to recover more sodium sulphate. Cresols are finally purified by fractional distillation. The process can yield para-cresol of 97% purity and/or meta-para mixed cresols containing as high as 65% meta-cresol. The overall conversion of toluene to cresols is around 80% of theoretical.

### **1.1.2 Process for the production of Resorcinol**

#### **(a) Industrial Application**

Resorcinol furfural resins blended with plasticized nitrile rubber give material which can be used to bind crude and vulcanized rubber to metal objects and fluorinated organic materials. Resorcinol formaldehyde resins have a special application in the bonding of textile materials to rubber in the manufacture of tyres, belting heavy-duty hose and rubberized textile sheeting. Resorcinol-formaldehyde resins have extensive use as adhesives, which are especially important in the manufacture of laminated timbers, roof trusses, laminated paper and cloth products.

As an antiseptic agent resorcinol finds use in preparation of cosmetics. It is mainly used for external applications in the form of paste and ointments for the treatment of skin diseases and superficial fungus infections. Para amino salicylic acid and resorcinol mono-acetate are derivatives of resorcinol which are used in pharmaceutical preparations.

#### **(b) Process**

Benzene is sulphonated with 100% sulphuric acid to get benzene-monosulphonic acid which is subsequently converted to benzene-meta-disulphonic acid by reaction with 60-65% oleum. The sulphonated mass is neutralized with caustic lye and sodium sulphite. The sodium metadisulphonate is isolated from the neutralized mass by crystallization. The dry disulphonate is then fused with caustic soda and molten mass is quenched with water. Quenched melt is then acidified with  $\text{SO}_2$  to liberate resorcinol. Resorcinol is extracted with an organic solvent and recovered by vacuum distillation. The overall conversion of benzene to resorcinol by CFRI process is 65-68% of theoretical.

The process can yield technical or pharmaceutical grade resorcinol (99.5%purity) with proper control of distillation. The process has been proved and demonstrated in the scale of 300g resorcinol per batch.

### 1.1.3 Process for the production of Beta-Naphthol

#### (a) Industrial application

Beta-naphthol is an important chemical required for the manufacture of a major proportion of dyestuffs in demand. It also finds application in the production of pharmaceuticals, perfumes, tanning agents and rubber chemicals. The most important category of dye intermediate manufactured from beta-naphthol is the hydroxy naphthalene sulphonic acid such as R-acid (2-naphthol-2, 6-disulphonic acid), G-acid (2-naphthol-6, 8-disulphonic acid) and Schaeffer acid (2-naphthol-6-sulphonic acid). The dyestuffs prepared from these intermediates are Crocein Orange-G, brilliant Orange-R, fast red-E, Wool green - S, cloth red- GEX, diaminogen blue-BB, toluidine Orange-G, pigment scarlet-G, alizerine black-SN, naphthol green-B, naphthol black-B etc. Beta-naphthol is also used as a coupling agent in the manufacture of several important azo and naphthol dyes such as fast red-G, lake red-G, fast red-A, Orange-11, naphthol blue, permanent red-2G and oil red-XI). Beta-naphthol is also used widely for the production of dyes directly on fabrics such as para red. These dyes do not exist as such and are directly produced on the fabrics itself from their compounds. Beta-naphthol and its salicyclic esters (Betol) find use in intestinal antiseptics, 2-naphthyl benzoate, benzo naphthol and 2-naphthol-2 lactate are produced from beta-naphthol and have applications as antiseptics and parasiticities. Beta-naphthol also finds applications for the preparation of beta-naphthyl ethyl ether (Nerolin) - a perfume for soaps and in the preparation of beta-naphthyl isobutyl ether - a perfumary ingredient.

#### (b) Process

Naphthalene is sulphonated with 98% sulphuric acid in liquid phase at a temperature of around 160°C. The alpha naphthalene sulphonic acid formed as a co-product is converted to naphthalene by steam hydrolysis and removed from the reaction mixture. The beta-naphthalene sulphonic acid is neutralized with caustic lye and sodium sulphite. The naphthalene sulphonate is removed from the neutralized mass by crystallization. The dry sodium sulphonate is then fused with caustic soda in a temperature range of 300-350°C. The fused mass is quenched with water and is acidified with SO<sub>2</sub> to liberate betanaphthol. Betanaphthol layer is separated and is further purified by distillation under reduced pressure. The purity of the product is 99.5% (confirming to IS 2741 - 1973).

#### (c) Commercial plants based on CFRI Technology

There exist two commercial plants of beta-naphthol based on CFRI process:

- (i) Eastern Naphtha Chem. Ltd., Bokaro (Cap. 660 tone/yr).
- (ii) Oswali Chemicals Ltd., Cuddalore (Tamil Nadu) Cap. 1000 tone/yr).

The above work has also been reported elsewhere (13, 16)

## **1.2 Development of Pyridine based chemicals**

Pyridine, methylpyridines and their intermediate compounds are used as precursors in the manufacture of drugs, agro-chemicals and rubber chemicals. Till now these compounds are manufactured by the old technologies using the corrosive and pollution causing materials. Pyridine, monomethyl pyridines, pyridine 3-carbonitrile and pyridine 4-carbonitrile are important feed stocks for production of drugs and pharmaceuticals like nicotinamide (pp vitamin) nicotinic acid (food additive), antihistamins (decapyrin), stimulants (retalin and pipradol), coramine, isoniazid (tuberculosis drug) etc. agro chemicals like diaquant and paraquat, reglone and gramoxone, rubber chemicals like vinyl pyridine (tyre adhesive).

### **1.2.1 Process on production of pyridine and methyl pyridines**

Pyridine and methyl pyridines have been developed by vapour phase reaction of aldehydes and ammonia, the cheap raw materials sufficiently available in the country from molasses of sugar industries and ammonia from fertilizer industries. The solid catalyst and process parameters have been developed in a single tube reactor of 100 ml catalyst volume and 60% yield of pyridine and 3-methyl pyridine achieved.

Development of this work was also reported in the form of publications (10, 39, 40) and patents (0507NF2003/WO, US).

### **1.2.2 Processes for synthesis of pyridine 3-carbonitrile and pyridine 4-carbonitrile**

These intermediate compounds have been developed by vapour phase ammoxidation of 3-methyl pyridine and 4-methyl pyridine respectively. The solid catalyst and process parameters have been developed in a single tube reactor using 100 ml catalyst volume and more than 90% yield of carbonitriles achieved.

This work has been patented (0025NF2003/EP, BR) and published (47).



### **1.2.3 Processes on synthesis of Nicotinamide and INH**

Next down stream processes which are developed in CFRI are production of nicotinamide and isonicotinamide by catalytic hydration of pyridine 3-carbonitrile and pyridine 4-carbonitrile respectively and another process for an important drug named as isoniazid (isonicotinic acid hydrazide) from isonicotinamide have been developed in liquid phase, each process upto the scale of 250g per batch. 90-95% (mol %) yield of these drug intermediates with 99% purity have been achieved.

The above CFRI processes developed for value added products are superior to the conventional processes because these are single step processes with high yield and better quality as well as environmentally friendly without emission of acid fumes and discharge of corrosive effluents.

The above processes on pyridine based chemicals have already been demonstrated /are to be demonstrated to some of the private entrepreneurs for transfer of technologies.

The work has been patented (0166NF2002/WO, US; 0157NF2002/US, EP) and published (50).

### **1.3 Development of processes on synthesis of aromatic nitriles**

Benzonitrile and phthalodinitriles are important precursors in the production of pigments, resins and polyester fibres. Benzonitrile and phthalodinitriles have been synthesized by ammoxidation reaction of toluene and xylenes (o-, m-, p-) in presence of vanadia catalyst developed at CFRI. The work has also been reported elsewhere (36). Details of all the processes are given in table 1.

**Table 1 Technology on synthesis of pyridine carbonitriles, aromatic nitriles, Nicotinamide and INH developed at CFRI.**

Technology	Raw materials	Catalyst	Reaction parameters			% Yield (by wt.)	Scale of work
			Temp °C	Sp. vel cc/cc/h	Pressure		
1. Catalytic vapour phase synthesis of pyridine-3-carbonitrile in a fixed bed reactor	3-picoline, air, and ammonia	Vanadia catalyst	350-450	2000-4000	atm	88-92	Bench scale
2. Catalytic vapour phase synthesis of pyridine-4-carbonitrile in a fixed bed reactor	4-picoline, air and ammonia	Vanadia catalyst	350-450	2000-3500	atm	95-98	-do-
3. Catalytic hydration of pyridine 3-carbonitrile to nicotinamide	Pyridine-3-carbonitrile	Transition meta oxide	100-110	-	atm	100-115	Bench/ Pilot Plant
4. Synthesis of INH from isonicotinamide (from pyridine-4-carbonitrile)	Isonicotinamide, hydrazine hydrate and alcohol	-	100-110	-	atm	100	Bench scale
5. Benzonitrile from Toluene	Toluene, air, ammonia	Vanadia, Chromia, Alumina (10-15 ml cat. Vol)	300-400	1071	atm	90.0 (BN)	Lab. Scale
6. Orthoxylene to Orthophthalodinitrile + phthalimide	Orthoxylene, air, ammonia	Vanadia, Chromia, alumina (10-15 ml cat. Vol.)	300-450	1481	atm	45 (OPN), 15.0 (phthalimide)	-do-
7. m-xylene to isophthalodinitrile	m-xylene, air, ammonia	Vanadia, chromia, boria- Alumina (10-15 ml cat. Vol)	300-450	2880	atm	70.0 (IPN)	-do-
8. P-xylene to terephthalodinitrile	p-xylene, air, ammonia	Vanadia- Chromia- Boria- Alumina (10-15 ml cat. Vol.)	300-450	1440	atm	80-0(TPN)	-do-

#### 1.4 A process on synthesis of Diphenic acid from phenanthrene

Diphenic acid is used in the production of high temperature heat resistant resins, engineering, plastics, liquid crystalline polymers, pharmaceuticals and also in agro-chemical industries. Diphenic acid acts as chain terminators on reaction with the aromatic diamine. Due to its excellent performance in the above fields, there is growing demand in the world market.

This is a liquid phase process in which phenanthrene and glacial acetic acid are added together in a reactor and heated at 75-80°C. To the resulting mixture, 100-300 ml of 30% hydrogen peroxide solution is added drop wise at 75-80°C maintaining the temperature 3-4 hrs. The resulting mixture is distilled under reduced pressure to make the volume half and cooled. Diphenic acid

crystals are obtained. It is filtered and the residue is boiled with 100% solution of sodium carbonate and active charcoal and filtered and the filtrate is acidified with HCl to maintain the pH 4.5 and cooled where diphenic acid crystallizes out (m.p. 228<sup>0</sup>C). The product is 99% pure.

The process for production of diphenic acid is important which requires upscaling for transfer of technology to the entrepreneurs.

This work has resulted a patent (0544NF2002/WO, US).

### **1.5 Process for the production of 9,10-Phenanthraquinone**

9,10-Phenanthraquinone is used as an additive for photographic or electro-photographic applications, in UV-curable coatings and adhesives and adhesives; in production of celluloid by wood pulping. An intermediate for azo pigments can be obtained by condensation with hydrazinobenzoic acid. Condensation with aromatic amines gives intermediates for pharmaceuticals (e.g. immuno-suppressives) or fungicides.

The process has been developed by catalytic liquid phase oxidation of phenanthrene in presence of dichromate as catalyst achieving 50-57% (wt. %) yield of 9,10 phenanthraquinone with 99% purity. The major advantage of the process lies in the fact that (i) the chemicals used in the process are very cheap and non-polluting in nature and (ii) all the process steps were low energy consuming.

This process has been commercialized by M/S Cstron Technologies Ltd., Dhanbad

Earlier considerable work (4, 12, 17, 25) was also carried out at CFRI, under the program of utilization of coal carbonization byproducts on the oxidation of various byproduct chemicals like benzene to maleic anhydride, toluene to benzaldehyde and benzoic acid, o- xylene and naphthalene to phthalic anhydride and anthracene to anthraquinone by developing different oxidation catalyst. These products have great industrial application in different fields- dyestuffs, paper industries, plastics, resins and plasticizers etc.

## 2. Development of chemical processes based on C<sub>1</sub>-compounds

### 2.1 Synthesis of olefins and selected fraction of hydrocarbons from methanol/syngas through modified F.T synthesis (SSRC-Department of Coal)

The lower olefins (C<sub>2</sub>-C<sub>4</sub> range) are the starting materials for a tremendous volume and variety of industrial polymers and chemicals. The manufacture of higher olefins of C<sub>5</sub>-C<sub>11</sub> range for processing to plasticizer alcohols as well as to C<sub>9</sub>-C<sub>18</sub> hydrocarbons for the production of detergent raw materials is possible through further processing of the lower olefins produced from syngas i.e. by oligomerisation of lower olefins to higher olefins. Thus, it would effect reduction in the dependence of petroleum naphtha.

It is in this context that synthesis of olefins and selected faction of hydrocarbons through C<sub>1</sub>- chemistry has become an emerging area development. C<sub>1</sub>- chemistry denotes chemical processes that form compounds with C-C bonds from C<sub>1</sub>- building block like CO (syngas), methanol, formaldehyde, HCN, methane etc. Since CO and methanol can be derived from coal, these methods of making petrochemicals currently obtained from C<sub>2</sub>-C<sub>3</sub> olefins have assumed importance. These new C<sub>1</sub>-processes are different from older technology in that more sophisticated catalysts have been used for more selective and less energy intensive processes.

#### Conversion of syngas to olefins

Different iron based and cobalt based catalyst were prepared and tested for the conversion of syngas (1:1 and 1:2) to lower olefins. It is observed that among the catalysts tested promoted and calcined iron-titania catalyst system showed highest activity and selectivity towards the formation of lower olefins. The maximum selectivity to the lower olefins (C<sub>1</sub>-C<sub>2</sub> range) achieved is 68% (wt %), the maximum yield of lower olefins obtained is 52 gm/Nm<sup>3</sup> of syngas consumed, and the maximum CO+H<sub>2</sub> conversion obtained is 45%. Using Fe-TiO<sub>2</sub> promoted and calcined catalyst the effect of following reaction parameters were studied for the conversion of syngas to lower olefins (i) pressure, (ii) reaction temperature (iii) gas space velocity, (iv) syngas composition. It is observed that for the maximum yield of lower olefins and for maximum syngas conversion the following optimum reaction parameters were maintained: reaction pressure- 2.5 Kg/m<sup>2</sup>, reaction temperature –250<sup>0</sup>C, gas-space velocity- 1060 cc h<sup>-1</sup>.

For the homogeneous phase oligomerisation, the following organometallic catalyst based on Ti and Al i.e.  $\text{TiCl}_4 + \text{Et}_3\text{Al}_2\text{Cl}_3$ ,  $\text{Ti}(\text{OBu})_4 + \text{Et}_2\text{Al}_2\text{Cl}_3$ ,  $\text{TiCl}_4 + \text{Et}_2\text{AlCl}$ ,  $\text{TiCl}_4 + \text{Et}_3\text{Al}$ , Cobalt-octate +  $\text{Et}_3\text{Al}_2\text{Cl}_3$ , were used for the oligomerisation of ethylene to higher olefins. Among the catalysts tried using  $\text{TiCl}_4 + \text{Et}_3\text{Al}_2\text{Cl}_3$ , catalyst the maximum selectivity to higher olefins achieved is 84.9% with ethylene conversion 96.6%. It is interesting to observe that using Cobalt octate +  $\text{Et}_3\text{Al}_2\text{Cl}_3$  catalyst 1-butene is the only product that is having 100% selectivity. Using  $\text{Ti}(\text{OBu})_4 + \text{Et}_3\text{Al}_2\text{Cl}_3$  catalyst for the selectivity to 1-butene is also 37.8% (wt%). For the heterogeneous phase oligomerisation, the following catalysts like Ni-SAPO-5, HSM-5 Zeolite (Si/Al = 100), 12-tungsto phosphoric acid supported on HZSM-5 (Si/Al = 100),  $\text{NiSO}_4$ -supported on alumina,  $\text{NiSO}_4$  supported HSM-5 (Si/Al = 40) and activated bentonite clays were tested for the oligomerisation of ethylene to higher olefins. The selectivity to higher olefins is not very high in comparison with that obtained by homogeneous phase oligomerisation of ethylene.

Process on synthesis of lower olefins and further its oligomerisation to higher olefins may be studied in higher scale for its commercialization.

This work resulted published papers (5-8, 29-30) and report (1).

## **2.2 Synthesis of higher alcohols from synthesis gas through modified Fischer-Tropsch synthesis (US-INDIA fund under NSF program)**

Higher alcohols are put to use, though to a limited quantity to substitute tetraethyl lead as an additive to petrol. This has gained importance considering the pollution caused by tetraethyl lead when used as an additive for petrol. Since higher alcohols are ideal substitute for tetraethyl lead, efforts are going on throughout the world to develop a suitable catalyst for the synthesis of higher alcohols from syn gas. Considering the above facts, the project is having the following objectives:

- (i) To develop catalyst for synthesis of higher alcohol from syn gas
- (ii) To optimize the process parameters such as reaction temperature, pressure, molar ratio of reactants space velocity etc. for the maximum production of higher alcohols.

It is possible to produce mixture of methanol and higher alcohols from synthesis gas by alkali promotion of the methanol synthesis catalysts and by appropriate modification of the reaction conditions. In the eighties, chemical and petroleum industries have shown a renewed and growing interest in the use of mixture of methanol and higher aliphatic alcohols. More recently the

trend to restrict the use of lead for environmental protection has focused the attention on the product performances of such alcohol mixture as high octane blending stock for gasoline. It has been shown that the addition of higher alcohol to methanol increases the water tolerance in respect of phase separation, reduces the fuel volatility and vapour lock tendency also results in higher volumetric heating values. Addition of alcohol to gasoline decreases the CO, NO<sub>x</sub> and unburnt hydrocarbon emission and could be an answer to lead additive suppression in gasoline. An adequate C<sub>1</sub>-C<sub>6</sub> alcohol mixture has a very good octane No. (RON-120-121, MON-97-98).

These considerations have reinitiated investigation on higher alcohol synthesis (HAS) from carbonmonoxide and hydrogen since the beginning of the nineties. However, the price of oil has favoured alternative solutions of the refining process and use of oxygenated petrochemical compounds such as Methyl-tert-butylether (MTBE), whose worldwide production has increased remarkably in the last few years. However, in spite of this higher alcohol can be a real alternative for MTBE, since they are entirely based on natural gas or coal and are good octane boosters and combustion improvers. Further more, they are clear fuels without sulphur, olefins and aromatics.

Keeping in mind the above facts, R&D work has been taken on the development of a suitable catalyst for the selective production of higher alcohol and optimizing the reaction parameters such as pressure, temp., space velocity and H<sub>2</sub>/CO ratio of the feed.

The following three catalysts were prepared by citrate complexation method:

Alkali promoted Co/Cu/Al with promoter	-	Cat A
Alkali promoted Co/Cu/Al with rare earth and promoter-		Cat B
Alkali promoted Co/Cu/Zn/Al with promoter	-	Cat C

These catalysts (12ml. to 35ml.) were tested in Catatest unit using syngas (H<sub>2</sub>:CO- 2:1 & 1:1). Effect of reaction variables such as pressure (50 to 65 bar), temp. (280-330°C) and space velocity (800-1200 h<sup>-1</sup>) was studied and optimum conditions for each catalyst were obtained. The results are shown in table2 & 3.

**Table 2 Reaction conditions and results for synthesis of higher alcohols**

Catalyst	Pressure Bar	Temp (°C)	Sp. Velocity (h <sup>-1</sup> )	Syn gas conversion (%)	Alcohol formed/Nm <sup>3</sup> of syn gas consumed g
Cat A	65	300	800	86.0	30.1
Cat B	65	280	848	61.1	42.2
Cat C	65	310	1000	61.6	32.5

**Table 3 Alcohol selectivity (% wt/wt) in the mixture**

Catalyst	CH <sub>3</sub> OH	C <sub>2</sub> H <sub>5</sub> OH	C <sub>3</sub> -OH	C <sub>4</sub> -OH	C <sub>5</sub> -OH	C <sub>6</sub> -OH
Cat A	17.7	9.6	48.8	14.0	-	9.9
Cat B	37.6	37.7	22.5	5.6	1.6	-
Cat C	17.1	51.2	25.3	6.4	-	-

C<sub>3</sub>-C<sub>4</sub> alcohol contained iso as well as n-alcohol.

It has been observed that reaction temperature plays a vital role in the selectivity of individual alcohol in the product. At lower temperature i.e., below 300°C, selectivity of C<sub>1</sub> and C<sub>2</sub> alcohol increased significantly at the cost of C<sub>2</sub>+ alcohol selectivity and at the same time the yield of total alcohol increased. When the temperature was increased above 300°C, C<sub>1</sub>-C<sub>2</sub> alcohol selectivity decreased and C<sub>2</sub>+alcohol selectivity increased significantly but total alcohol yield decreased. Higher reaction temperature i.e., above 300°C also favoured more formation of iso-propanol and iso-butanol. In case of Cat A although the total alcohol formation was less than Cat B, but C<sub>2</sub>+alcohol selectivity was more than 70%. In case of cat C which contained Zn also, ethanol formation was always higher than methanol and it was the main product, i.e., more than 50% selectivity. In case of all catalysts liquid hydrocarbons was formed in significant quantity i.e. parallel to alcohol some times even more along with small amounts of other oxygenates. In hydrocarbon gas formation, mostly C<sub>1</sub> to C<sub>4</sub> saturates were formed. CO<sub>2</sub> formation was also higher specially in case of using syn gas H<sub>2</sub>: CO = 1:1.

An internal report was submitted on this work (2)



### **3. Design and development of nano-material technology for pre-reforming of hydrocarbons including naphtha (NMITLI project)**

The objective of the project was

- (i) Designing a suitable reactor for pre-reforming of hydrocarbons.
- (ii) Development of nanomaterial catalysts for prereforming of naphtha

A pre-reformer SS reactor of 100 ml catalyst volume, max pressure 50 kg/cm<sup>2</sup>, temperature 600<sup>0</sup>C was designed by CFRI and fabricated by M/S Chemito Technologies, Nasik and installed at CFRI. Nanomaterial catalyst based on nickel-alumina containing alkaline-earth and rare earth metal oxides was developed by Aerogel method achieving particle size in the range of 0.60 to 12.0 µm and nickel particle size on the catalyst up to 8.0 nm. Nickel was loaded up to 40% in the catalyst. The maximum conversion of naphtha obtained was 94-98% and methane formation was 75-77% at the temperature range 425-450<sup>0</sup>C and 30 kg/cm<sup>2</sup> pressure for pre-reforming of naphtha over the catalyst packed in the pre-reformer reactor (developed at CFRI).

As a result of this work a patent has been filed and a paper has been presented (49).

### **4. Development of technology for the production of additives based on coal tar products for coal water slurry fuels (SSRC Project)**

Process for the production of anionic types of additives based on coal chemicals has been developed. The additives developed have been found to function excellently in the formulation of the Coal Water Slurry Fuels (CWSF). Slurries with high coal concentration (70%) and having acceptable viscosities have been formulated using a low-concentration of the additives (0.8-0.9 wt%, dry coal basis). Various parameters such as the coal properties, particle size distribution, affecting the CWSF characteristics and stability, have been studied.

Since the studies have been carried out in a lab/bench scale, it is suggested to evaluate the reliability/feasibility of the overall CWSF technology in a demonstration plant of reasonable capacity (e.g. 0.5-1 tone coal charge) together with the production facilities of the additive package (e.g. 10 kg/batch of NSA). The study on atomization and combustion characteristics of the CWSF, a very vital area of the overall technology but not covered under the



scope of the present work, must be carried out either in the demonstration plant itself or separately. The demonstration project would also provide essential data for calculating the costing parameters needed for setting up a commercial unit for the production of CWSF.

The work has resulted in the form of a published paper (46).

## **5. CSIR Task Force Networking projects (On-going)**

### **5.1 Conversion of methane to ethylene by oxidative coupling of methane Objective**

To develop catalyst and process parameters for selective production of ethylene by oxidative coupling of methane (OCM) in a single step. This will overcome the multi-step conventional process for conversion of methane to syn gas by reforming and further conversion to ethylene via methanol.

The abundance of natural gas and also coal bed methane (a new source of methane), together with steadily depleting oil reserves highlights methane conversion to higher hydrocarbons as an extremely attractive goal. Due to refractory nature of methane, its activation is a great challenge before the Scientists making this as one of the most extensively investigated processes in catalysis. Methane can be converted to higher hydrocarbons by indirect routes through syn-gas or by direct routes. The direct route involves either the high temperature non-oxidative coupling of methane or the oxidative coupling of methane (OCM) to higher hydrocarbons.

Recently, Nanocatalysts have got prominence in catalysis because of their higher catalytic performance over traditional catalysts. This can be synthesized by several methods; however, in this investigation solgel (aerogel) technique has been employed for the preparation of catalysts for oxidative coupling of methane. The oxidative coupling of methane to  $C_2$  hydrocarbons was studied over  $La_2O_3/MgO$ ,  $La_2O_3/MgO-CaO$  and  $La_2O_3/MgO-Al_2O_3/NiO$  catalysts. Catalysts were prepared by aerogel method using organometallic precursors. These were calcined at  $750^\circ C$ ,  $850^\circ C$  and  $950^\circ C$  and characterized by determining particle size and also by XRD and SEM. The performance of these catalysts was evaluated for oxidative coupling of methane in the presence of molecular oxygen at the temperature in the range of  $650-800^\circ C$  at atmospheric pressure in a continuous flow fixed bed SS reactor. Nickel containing catalyst showed higher conversion of

methane (30.3%) where as selectivity to C<sub>2</sub>-hydrocarbons was lower and formation of carbon oxides was high. La<sub>2</sub>O<sub>3</sub>/MgO catalyst showed lower conversion of methane but the selectivity towards formation of C<sub>2</sub>-hydrocarbons was better (26%) compared to the former catalyst. La<sub>2</sub>O<sub>3</sub>/MgO - CaO has been found better catalyst for this reaction exhibiting high selectivity to C<sub>2</sub>-hydrocarbons (56.3%).

This work resulted in the presentation of a paper in a workshop (53). Further work is in progress to develop more selective catalyst for maximum conversion of methane (>40%) and ethylene selectivity (>80%).

## **5.2 Green Technologies for Organic Chemicals: Alkylation of naphthalene**

### **5.2.1 Objective**

Conventional Friedel Craft catalysts like metal halides are causing a lot of environmental pollution for alkylation reaction. Hence, metal halides are replaced by shape selective solid acid catalysts in an eco-friendly process. Thus, it is proposed to develop solid acid catalyst and optimize process parameters for the selective alkylation of naphthalene for environmental friendly synthesis of alkyl naphthalenes.

### **5.2.2 Progress**

Alkylation of naphthalene was carried out in a continuous flow pyrex reactor with 20 mm id and length 30 cm under atm pressure, with 10 g catalyst (6-14 mesh). The reaction mixture of naphthalene and methanol (1:6 molar ratio) with mesitylene as solvent was fed using a syringe pump. The product was condensed and collected at the bottom and analyzed by GC using BPX 50 capillary column of 30 m length fitted to FID.

The reaction of naphthalene with methanol was studied over various Ce & La-modified zeolite catalyst (HZSM-5, beta zeolite, zeolite L and Y type) at the temperature 250-350°C and methanol/naphthalene mole ratio 2-6 using mesitylene as solvent. GC analysis revealed the formation of 2-methyl naphthalene (2-MN), 1-methyl naphthalene (1-MN) and 2,6-dimethyl naphthalene (2,6-DMN) as major products. The alkylation activity was enhanced due to modification of H $\beta$  by cerium. It was also observed that 2-MN was formed predominantly compared to 1-MN.

Alkylation of naphthalene was also carried out in a high pressure fixed bed reactor (Make: Chemito Instruments Pvt. Ltd, Nasik, India) using Ce-modified (large pore) zeolite as catalyst, under a pressure of 6 kg/cm<sup>2</sup>. The maximum conversion of naphthalene and selectivity to mono and dialkyl naphthalene obtained are: Conversion of naphthalene: 68%, Selectivity to 2-methyl naphthalene, 1-methyl naphthalene and 2, 6-dimethyl naphthalene are 45.0%, 34.8% and 20.2% respectively.

This work has resulted a paper, which was presented in a seminar (51). Further work is in progress to develop an active, selective and stable catalyst for alkylation of naphthalene.

**5.3 Globally competitive chemicals, processes and products: Scale up studies of the processes (i) Isonicotinic acid hydrazide (INH) from Pyridine 4-Carbonitrile via isonicotinamide (ii) Pyridine 3-Carbonitrile by ammoxidation of 3-picoline (iii) Pyridine 4-Carbonitrile by ammoxidation of 4-picoline.**

**5.3.1 Objective**

To develop catalysts and process parameters for production of (I) INH from pyridine 4-carbonitrile via isonicotinamide in the scale of 100- 250g/batch in liquid phase (ii) Pyridine 3-carbonitrile and pyridine 4-carbonitrile by ammoxidation of 3-picoline and 4-picoline respectively in a fixed bed reactor in vapour phase using 100 ml catalyst volume. Upscaling these processes will help in transfer of technology useful to pharmaceutical industry.

**5.3.2 Progress**

Scale up studies for the process on Isonicotinamide from 4-cyanopyridine have been made in two scales: 200 gm/batch and 250 gm/batch by catalytic hydration in liquid phase and five experiments in similar conditions have been conducted. All the steps of the process- reaction, filtration and drying have been standardized.

Using prepared isonicotinamide, preparation of INH in 200 g/batch and 250g/batch have been carried out by conducting three experiments under similar conditions and all the steps of the process like reaction with hydrazinehydrate, washing and crystallization have been standardized. Product has been identified by FTIR and compared with authentic sample and

also by determination of melting point. 93-95% (mole %) yield of INH with 99% purity have been obtained during this study. Melting point of INH before crystallization has been observed 167-168°C and after crystallization 171°C (melting point of pure INH). Test for IP grade INH is to be carried out at outside laboratory.

Theoretical studies have been made to examine the characteristics of -OH groups present on metal oxide catalyst responsible for catalytic activity for hydration of nitrile to amide. FTIR studies of the catalyst heated at 400°C have been made, which showed a significant decrease in OH stretching, due to which the yield of isonicotinamide decreased in hydration of 4-cyanopyridine. The catalyst heated at 110°C (used in the reaction) has been tested by chemical method, showed lower pH i.e. more OH groups, which is responsible for higher yield of amide.

Attempt has been made to standardize the process on "Synthesis of 3-Cyanopyridine from 3-picoline" by conducting experiments on ammoxidation reaction over vanadia-molybdena phosphorous oxide catalyst based on zeolite and pillared clay using 20g catalyst. A VMPO catalyst was also prepared by reducing vanadium pentoxide by hydroxylamine hydrochloride. The catalyst was also tested in a single tube glass reactor by using 10ml catalyst as such and by loading over zeolite, result is encouraging. Studies on the preparation of bulk quantity of catalyst and its activity test are in progress.

Experiments for verifying the maximum reaction cycles of the nicotinamide process are in progress for demonstrating the process to the private entrepreneurs.

As a result of this work a number of papers (47, 50) have been published.

#### **5.4 Monomer for specialty chemicals**

Alkyl phenols are chemicals and chemical intermediates mostly used in Agro industries. Dialkyl phenols such as 2:6 xylenol and 2:4 xylenol are used in the manufacture of industrial grade plastics e.g. polyphenylene oxide (PPO & Paints), trialkyl phenols are plasticizers and precursor for synthesis of Vitamin E.

## Progress

Different catalysts were prepared using oxides like  $\text{La}_2\text{O}_3$ ,  $\text{CuO}$ ,  $\text{ZnO}$ ,  $\text{Al}_2\text{O}_3$  promoted on alkaline earth metal oxide and their activity were evaluated in a SS single tube reactor for alkylation of phenol with methanol. The reactions were performed varying the feed ratio and temperature at atmospheric and also at  $2\text{kg/cm}^2$  pressure. On optimizing the reaction conditions with different catalysts, it was revealed that the highest selectivity of 2,6- xyleneol was achieved (76.9%) with the catalyst containing  $\text{CuO}$  over alkaline earth metal oxide ( $\text{MgO}$ ). Following are the reaction conditions and the results of different products with the best catalyst:

Phenol:methanol 1:4, Temp. :  $470^\circ\text{C}$ , Feed rate 5 ml/hr, Catalyst vol. 25 ml.

Conversion to total xyleneol about 84% details as below:

		<u>w/w%</u>
2:6 xyleneol	-	76.90
2:5 xyleneol	-	1.00
2:3 xyleneol	-	1.20
3:4 xyleneol	-	4.50
2:4:6 Trimethyl Phenol-		1.50
Rest is mono alkylated phenol.		

Another process for the production of p-hydroxybenzoic acid is underway. The process involves blocking of  $-\text{OH}$  group of p-cresol using dimethyl sulphate/benzene sulphonyl chloride followed by oxidation employing  $\text{KMnO}_4$  /  $\text{Na}_2\text{Cr}_2\text{O}_7$  in both acidic and alkaline medium.

Based on this work a patent has been filed on "Alkylation of phenol to 2,6-xyleneol"

## ANNEXURE

### 1. Published papers on development of chemical processes

1. Effect of Quaternary Ammonium Compounds on the Physico-Chemical Properties of Amorphous Silica-Alumina. Bandopadhyay, A.K. Das, J. Roy, S.K. J. Catal., 124, 241 (1990).
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**2. List of patents on chemical processes**

NFNO	FD	TL	IV
0080NF2002/US	3/26/2002	A METHOD OF ENHANCING THE PURITY OF PHENANTHRENE AND A DEVICE THERE FOR	TIWARI K K, RAO S R K, THAKUR S S K, BANERJI SOM NATH
0080NF2002/WO	3/27/2002	A METHOD OF ENHANCING THE PURITY OF PHENANTHRENE AND A DEVICE THERE FOR	TIWARI K K, RAO S R K, THAKUR S S K, BANERJI SOM NATH
0157NF2002/WO	12/23/2002	PROCESS FOR THE SYNTHESIS OF ISONICOTINIC ACID HYDRAZIDE	RAY SC, NANDI L N, SINGH B, PRASAD HL, MAHARAJ S, SARKAR P K, DUTTA P, ROY SK, YADAV SN, BANDOPADHYAY AK.
0544NF2002/WO	12/23/2002	PROCESS FOR THE PREPARATION OF DIPHENIC ACID	TIWARI K K, BIT K C, THAKUR S K, MISHRA K K, RAO S R K
0166NF2002/WO	12/23/2002	PROCESS FOR CONVERSION OF CYANOPYRIDINES TO NICOTINAMIDES AND CATALYST THERE FOR	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
0166NF2002/US	3/20/2003	PROCESS FOR CONVERSION OF CYANOPYRIDINES TO NICOTINAMIDES AND CATALYST THERE FOR, PROCESS	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
0544NF2002/US	3/20/2003	PROCESS FOR THE PREPARATION OF DIPHENIC ACID	TIWARI K K, BIT K C, THAKUR S K, MISHRA K K, RAO S R K
0157NF2002/US	3/20/2003	PROCESS FOR THE SYNTHESIS OF ISONICOTINIC ACID HYDRAZIDE	RAY SC, NANDI LN, SINGH B, PRASAD HL, MAHARAJ S, SARKAR P K, DUTTA P, ROY SK, YADAV SN, BANDOPADHYAY A K.
0025NF2003/WO	3/31/2003	PILLARED CLAY BASED VANADIA CATALYST AND PROCESS FOR PREPARATION THEREOF	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN
0507NF2003/WO	12/31/2003	A CATALYST USEFUL FOR SYNTHESIS OF 2-&4-PICOLINES	P DUTTA, S C ROY, S K ROY, T K GOSWAMI
0507NF2003/US	12/31/2003	A CATALYST USEFUL FOR SYNTHESIS OF 2-&4-PICOLINES	P DUTTA, S C ROY, S K ROY, T K GOSWAMI
0229NF2002/EP	3/5/2004	A METHOD OF EXTRACTION OF DIMETHYLPHTHALATE	KUMARES CHANDRA BIT
0229NF2002/WO	3/5/2004	A METHOD OF EXTRACTION OF DIMETHYLPHTHALATE	KUMARES CHANDRA BIT
0229NF2002/US	3/5/2004	A METHOD OF EXTRACTION OF DIMETHYLPHTHALATE	KUMARES CHANDRA BIT



0166NF2002/EP	6/28/2005	A PROCESS FOR THE PREPARATION OF A CATALYST USEFUL FOR CONVERSION OF 3-& 4-CYANOPYRIDINES TO USEFUL PRODUCTS	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
0166NF2002/CN	8/2/2005	A PROCESS FOR THE PREPARATION OF A CATALYST USEFUL FOR CONVERSION OF 3-& 4-CYANOPYRIDINES TO USEFUL PRODUCTS	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
0544NF2002/CA	6/23/2005	A PROCESS FOR SYNTHESIS OF DIPHENIC ACID FROM PHENANTHRENE	TIWARI KK, BIT KC, THAKUR SK, MISHRA KK, RAO SRK
0544NF2002/JP	8/8/2005	A PROCESS FOR SYNTHESIS OF DIPHENIC ACID FROM PHENANTHRENE	TIWARI KK, BIT KC, THAKUR SK, MISHRA KK, RAO SRK
0166NF2002/AU	6/24/2005	A PROCESS FOR THE PREPARATION OF A CATALYST USEFUL FOR CONVERSION OF 3-& 4-CYANOPYRIDINES TO USEFUL PRODUCTS	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
0025NF2003/JP	9/30/2005	A PROCESS FOR THE PREPARATION OF A CATALYST SUPPORTED ON TITANIUM PILLARED CLAY, USEFUL FOR PRODUCING HETROAROMATIC NITRILES	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN
0229NF2002/AU	9/5/2005	A METHOD OF EXTRACTION OF DIMETHYLPHTHALATE	KUMARES CHANDRA BIT
0025NF2003/ZA	9/29/2005	A PROCESS FOR THE PREPARATION OF A CATALYST SUPPORTED ON TITANIUM PILLARED CLAY, USEFUL FOR PRODUCING HETROAROMATIC NITRILES	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN
0025NF2003/KR	9/30/2005	A PROCESS FOR THE PREPARATION OF A CATALYST SUPPORTED ON TITANIUM PILLARED CLAY, USEFUL FOR PRODUCING HETROAROMATIC NITRILES	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN
0025NF2003/CN	11/15/2005	A PROCESS FOR THE PREPARATION OF A CATALYST SUPPORTED ON TITANIUM PILLARED CLAY, USEFUL FOR PRODUCING HETROAROMATIC NITRILES	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN



0025NF2003/BR	9/30/2005	A PROCESS FOR THE PREPARATION OF A CATALYST SUPPORTED ON TITANIUM PILLARED CLAY, USEFUL FOR PRODUCING HETROAROMATIC NITRILES	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN
0025NF2003/AU	10/19/2005	A PROCESS FOR THE PREPARATION OF A CATALYST SUPPORTED ON TITANIUM PILLARED CLAY, USEFUL FOR PRODUCING HETROAROMATIC NITRILES	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN
0025NF2003/EP	11/20/2005	A PROCESS FOR THE PREPARATION OF A CATALYST SUPPORTED ON TITANIUM PILLARED CLAY, USEFUL FOR PRODUCING HETROAROMATIC NITRILES	ROY SK, RAY SC, DUTTA P, NANDI LN, YADAV SN
0544NF2002/NZ	7/11/2005	A PROCESS FOR SYNTHESIS OF DIPHENIC ACID FROM PHENANTHRENE	TIWARI KK, BIT KC, THAKUR SK, MISHRA KK, RAO SRK
0166NF2002/NZ	7/11/2005	A PROCESS FOR THE PREPARATION OF A CATALYST USEFUL FOR CONVERSION OF 3-& 4-CYANOPYRIDINES TO USEFUL PRODUCTS	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
0544NF2002/AU	6/27/2005	A PROCESS FOR SYNTHESIS OF DIPHENIC ACID FROM PHENANTHRENE	TIWARI KK, BIT KC, THAKUR SK, MISHRA KK, RAO SRK
0157NF2002/JP	6/22/2005	A PROCESS FOR THE SYNTHESIS OF ISONICOTINIC ACID HYDRAZIDE (INH) FROM ISONICOTINAMIDE	Ray S. C, Nandi L N, Singh B, Prasad H,L, Maharaj S, Sarkar P K, Dutta P, Roy S K, Yadav S N, Bandyopadhyay A K.
0157NF2002/EP	6/28/2005	A PROCESS FOR THE SYNTHESIS OF ISONICOTINIC ACID HYDRAZIDE (INH) FROM ISONICOTINAMIDE	Ray S. C, Nandi L N, Singh B, Prasad H,L, Maharaj S, Sarkar P K, Dutta P, Roy S K, Yadav S N, Bandyopadhyay A K.
0166NF2002/CA	6/23/2005	A PROCESS FOR THE PREPARATION OF A CATALYST USEFUL FOR CONVERSION OF 3-& 4-CYANOPYRIDINES TO USEFUL PRODUCTS	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
0544NF2002/CN	7/27/2005	A PROCESS FOR SYNTHESIS OF DIPHENIC ACID FROM PHENANTHRENE	TIWARI KK, BIT KC, THAKUR SK, MISHRA KK, RAO SRK
0229NF2002/JP	9/5/2005	A METHOD OF EXTRACTION OF DIMETHYLPHTHALATE	KUMARES CHANDRA BIT



0166NF2002/US	1/24/2006	A PROCESS FOR THE PREPARATION OF A CATALYST USEFUL FOR CONVERSION OF 3-& 4-CYANOPYRIDINES TO USEFUL PRODUCTS	RAY SC, SINGH B, MAHARAJ S, PRASAD H, SARKAR PK, DUTTA P, ROY SK, BANDOPADHYAY AK, SEN R
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1. Report on utilization of syngas from coal- synthesis of higher olefins through oligomerisation of olefins from syngas. No. TR/CFRI/3.01/1999-2000.
2. Report on synthesis of higher alcohols from synthesis gas through modified Fischer-Tropsch synthesis. No. TR/CFRI/3.02/2000-01.
3. Report on development of technology for the production of additives based on coal tar products for coal water slurry fuels. No. TR/CFRI3.03/1998-99.



## Coal Liquefaction Division

# Prospects of Oil from Coal in India

## Abstract

History of coal liquefaction dates back to early 20<sup>th</sup> century. Germany had successfully used both direct and indirect routes for production of oil during Second World War on commercial scale. South Africa under geopolitical compulsion produced most of its oil requirement at Sasol through indirect process from mid 1950's to 1980's. Incomprehensible price rise during 1970's was instrumental in the development of a large number of coal liquefaction processes, some of which were studied upto pilot plant scale. However, none of these processes could clear the techno-economic viability tests.

Discovery of new oil fields and slump in oil prices led to shelving of most of these projects. Activities pertaining to coal to oil is being pursued in India since 1960's primarily at CFRI, Dhanbad. R & D activities on kinetics and mechanism, action of catalyst, solvent quality, characterization of products and comparative amenability of Indian coals towards hydrogenation had been carried out in details. Based on these studies CFRI has developed two low severity processes. One of which is for conversion of coal to oil with high distillate yield(~90%) while the other one deals with development of coal-water emulsion. China has recently gone all out to produce coal to oil commercially to meet its energy needs while India is still exploring possibilities of such coal to oil projects in near future.

## Introduction

Coal is a solid fuel with a high carbon content but with a hydrogen content of ~5%. Compared with liquid fuels (derived from petroleum crude oil) it is inconvenient to handle and unsuited to some applications, e.g., transportation.

Coal may be used to produce liquid fuels suitable for transportation applications by removal of carbon or addition of hydrogen, either directly or indirectly. The first approach is usually known as carbonisation or pyrolysis and the second as liquefaction. As the cost of converting coal into useful liquid fuels is higher than the cost of refining crude oil, it is the relative price of the raw feedstocks that provides the main incentive to pursue the technology.

Given the gradual depletion of oil reserves and skyrocketing prices of petroleum crude and products derived from it, time is ripe to look for alternate source which can supplement it as a short term measure and eventually

replace it when its availability becomes scarce. Coal liquefaction is one such source, backed by large recoverable coal reserves globally.

Significant coal liquefaction research and development (R&D) was started up in the early 1970s, particularly in the USA, the UK and Japan, in response to various oil price shocks. Since the 1980s, developmental works in coal liquefaction have been largely put on hold with the notable exception of South Africa. This country with large reserves of coal but without any oil or gas reserve and penalized with trade embargo on account of its rigid apartheid policy over three decades beginning in mid-1950 to the mid-1980 had little option except to go for coal liquefaction (indirect mode) to meet its oil requirements. Of late Japan and China have shown keen interest in developing commercially viable technologies for coal liquefaction.

Many different 'direct' processes have been developed primarily in USA during 1970's and 1980's but most are closely related in terms of the underlying reaction chemistry. Common features are the dissolution of a high proportion of coal in a solvent at elevated temperature and pressure, followed by hydrocracking of the dissolved coal with hydrogen gas and catalyst. The basic differences among these processes have been the reactor configuration and the catalyst used. Direct liquefaction is the most efficient route currently available. Liquid yields in excess of 70% by weight of the dry, mineral matter-free coal feed have been demonstrated in favourable circumstances. Overall thermal efficiencies (% calorific value of the input fuel converted to finished products) for modern processes are generally in the range 60-70% if allowance is made for generating losses and other non-coal energy imports, as compared to 50% of indirect liquefaction route. These processes generally have been developed to a level of process development unit (PDU) or pilot plant scale and the main technical issues have been resolved. However, no demonstration or commercial-scale plant has yet been built.

The only operating process for the 'indirect' liquefaction of coal is South Africa's Sasol process, with three operating plants. The only 'core' unit specific to indirect liquefaction is the synthesis reaction step and the majority of recent work has therefore been concentrated on the development of improved catalysts, which need not be specific to a particular process.

With lower oil prices since 1985, activities pertaining to coal liquefaction for the production of transportation fuels remained at low key till recently. However, Japan has been relentlessly pursuing activities in large-scale



process development, with a 150 tonnes/day plant in operation, whilst China (increasingly a net oil importer and containing areas remote from sources of oil) presents the strongest adoption prospect. China is currently co-operating with each of the USA, Japan and Germany on feasibility studies and moving ahead with the plan of installing the first commercial coal liquefaction (direct) of the world in late 2007.

The commercial viability of coal liquefaction rests with the overall economics of the process. This depends on the availability of significant quantities of poor quality, low cost coal, and the unavailability or otherwise relatively high cost of oil (and gas). With present level of oil price which in all likelihood would continue to rise further in coming days, possibilities of full-scale commercialization never looked so real.

### **Coal to Oil : An overview of its status & prospects**

Coal may be used to produce liquid fuels suitable for transportation applications by removal of carbon or addition of hydrogen, either directly or indirectly. In any hydrogenation process, coal gasification has been considered as an intrinsic part for the supply of process hydrogen. The general feature of the coal suitable for hydrogenation and also for gasification are that the coals should have a fairly high reactive (vitinite and exinite together) and ash content should be preferably low. Restriction on ash content is also governed partly by process configuration. For processes employing very high pressure, ash content should be as minimum as possible in order to cut down the corrosion and erosion problem of the reactor and sensitive control devices like valves etc. Another important parameter that needs consideration is the rank of the coal. Higher rank coals tend to be less amenable towards hydrogenation. The ideal coals for hydrogenation and gasification should have reflectance ( $R_o$ ) between 0.5 and 0.7. The reactive contents of the coals belonging to these ranks should be between 60 % and above in ROM coal.

Low ash coal is generally preferred for hydrogenation and gasification since most of the hydrogen processes employ very high pressure. Ash in large proportion is neither desirable from the point of operation and also from energy consideration despite its catalytic role for coal to oil conversion. However, R&D trends on hydrogenation had been directed for developing processes under low severity condition. The concept of Multi Stage

Hydrogenation development at CFRI, envisages conversion of oil to syncrude at a much less severity condition compared to the other processes.

### **International status of coal liquefaction**

Significant coal liquefaction research and development (R&D) was started up in the early 1970s, particularly in the USA, the UK and Japan, in response to various oil price shocks. In the 1980s, developments have been largely put on hold with the notable exception of South Africa, which has large reserves of coal but no oil or gas reserves. In South Africa it is reported that up to 60 % of the fuel requirements for transportation are met from the coal through indirect hydrogenation route. With lower oil prices since 1985, interest in coal liquefaction for the production of transportation fuels has declined. At present, only Japan is active in large-scale process development, with a 150 tonnes/day plant in operation, whilst China (increasingly a net oil importer) present the strongest adoption prospect. China is currently co-operating with USA, Japan and Germany on feasibility studies.

### **Indirect liquefaction**

Indirect liquefaction involves, as a first step, the complete breakdown of the coal structure by gasification with steam. The composition of the gasification products is then adjusted to give the required mixture of  $H_2$  and  $CO$ , and to remove sulphur containing catalyst poisons. The resulting 'synthesis gas' is reacted over a catalyst at relatively low pressure and temperature by Fischer Tropsch (FT) process. The products may be paraffins, olefinic hydrocarbons or alcohols (particularly methanol), depending on the catalyst selected and the reaction conditions used.

The Sasol process, the only commercial process is based on the Fischer-Tropsch (FT) liquefaction process operated on a large scale in Germany and elsewhere in the 1930s and 1940s, although over 40 years' continued development by Sasol has resulted in substantial improvements in the synthesis step. The production of synthesis gas by the Lurgi gasification of coal has remained relatively unchanged except for an increase in gasifier size. The first plant, Sasol 1, was built at Sasolburg in South Africa in the mid-1950s with a capacity of ~6000bbl/day of gasoline. The much larger Sasol 2 and 3 plant were completed at Secunda in 1980 and 1982 respectively. These plant were each designed to produce 50,000bbl/day of gasoline, together with substantial quantities of other products for use as chemical feedstocks, from the processing of 30,000 tons/day of coal.

The developments by Sasol have resulted in several changes to the processes now used. Sasol uses low-temperature Fischer Tropsch (LTFT) and high-temperature Fischer Tropsch (HTFT) for synthesis. LTFT is used exclusively at Sasolburg and comprises the older Arge fixed-bed technology as well as the newer-generation slurry-phase FT process. At Secunda the HTFT process is used exclusively and comprises older circulating fluidisedbed technology as well as new-generation Sasol Advanced Synthol (SAS) technology. SAS technology, first used in Secunda in 1995, gives the Sasol 2 and Sasol 3 plant a capacity of ~150,000bbl/day of crude equivalent of white products plus chemicals.

### **Direct liquefaction**

Direct liquefaction processes aim to add hydrogen to the organic structure of the coal and breaking it down only as far as necessary to produce distillable liquids. Direct liquefaction processes can conveniently be divided into two main groups, single stage process and two stage process.

- A single-stage direct liquefaction process gives distillates via one primary reactor or a train of reactors in series. Such processes may include an integrated on-line hydrotreating reactor, which is intended to upgrade the primary distillates without directly increasing the overall conversion.
- A two-stage direct liquefaction process is designed to give distillate products via two reactors or reactor trains in series. The primary function of the first stage is coal dissolution and is operated either without a catalyst or with only a low-activity disposable catalyst. The heavy coal liquids produced in this way are hydrotreated in the second stage in the presence of a high-activity catalyst to produce additional distillate.

### **Single Stage Processes**

In the mid-to-late 1960s, all of the available processes were single-stage. Most development therefore continued to adopt a single-stage approach. The single stage processes developed during this period include, Kohleoel (Germany), NEDOL( Japan), H-Coal (USA), EDS( USA), SRC-I & II ( USA), Imhausen high pressure ( Germany ), Conoco Zinc chloride( USA). Some developers added a second stage during the 1970s, following the oil crisis, to increase the production of light oils. Most of these have since been superseded and abandoned. Two exceptions are the Kohleoel and NEDOL processes, both of which are considered ready for commercialization by their developers.

## **Two Stage Processes**

Most two-stage direct liquefaction processes were developed in response to the oil price rises of the early 1970s, often as a development of earlier single-stage processes. These processes include, CTSL (USA), LSE (UK), BCL(Japan), CSF( USA), Lummus ITSL (USA), CCLP (USA), Kerr-McGee ITSL(USA), Mitsubishi Solvolysis (Japan), Pyrosol (Germany), Amoco CC-TSL (USA), SGE (UK). Work was carried out in many countries, but relatively few processes were developed beyond the laboratory scale and many were generically very similar. Development of the following processes continued beyond the late 1980s.

### ***Catalytic Two-Stage Liquefaction (CTSL)***

### ***Liquid Solvent Extraction (LSE)***

### ***Brown Coal Liquefaction (BCL)***

The two stage processes are the outcome and understanding of the basic studies on Coal Liquefaction and its mechanism. It has been known that build up of refractory coal derived substances during liquefaction stage lead to severe adverse effect on distillable products. The two stage processes take into consideration to use optimized conditions for dissolution of coal without generating any refractory type materials. These studies also brought out clearly the role of retrogressive reactions which eventually affects the quality of the product and also the quality of regenerated solvent. In general, in these processes pressure varies between 136 and 170 atmosphere with accompanying temperature between 400 and 450°C. In the stage two pressured used varied from 136 to 186 atmosphere and the temperature varied from 330 to 449°C. Distillable syncrude yield (C4 - 450°C) varied from 39 % (ITSL) to 57 % (CTSL). These processes made distinct improvement in the distillate yield and also on reaction severity. But even then the conversion level was not high enough for making the process economically viable.

The greatest impediment in eventual commercialization of the currently available technologies is the conversion level of as low as ~60 % (maximum). Other hurdles include, high reaction severity leading to coke formation at regular interval during operation of plants resulting in erosion and regular replacement of coal prohibitive valves and above all liberal consumption of high cost hydrogen gas. All these add up to a highly uneconomical product slate and rules out the commercialization aspects of the available processes.

## **Status & prospects of coal liquefaction in India**

In our country, coal to oil as an alternative route to supplement the fuels has been periodically reviewed and considered at root level. Several expert committees have been constituted since 1948. The recommendations of which are shown in the Table 1.

Yet no clear guideline or policy on synthetic fuel emerged till date as an outcome of the reports submitted by these expert committees. The indecision and lack of clear understanding of the strategic importance of development of indigenous coal liquefaction technologies resulted in extreme reluctance on the part of concerned ministries to support activities in developing commercially viable coal liquefaction technologies suitable for Indian coals. As such, studies on coal liquefaction particularly coal hydrogenation had been restricted to laboratory scale R&D activities[1-33] principally at Central Fuel Research Institute. One 0.5 tpd high pressure plant was set up at CFRI to study the hydrogenation of coal. The single stage process yielded 25 % oil and operation problems were too many e.g., valve erosion, choking of the reactors, etc. Basic studies on reaction kinetics and mechanism, action of catalyst, solvent quality, characterization of products and comparative amenability of Indian coals towards hydrogenation had been carried out in detail and based on the information generated as an outcome of these basic studies a new generation indigenous and innovative process named as “Multi Stage Hydrogenation of Coal was developed at CFRI

### **Development of Multi Stage Hydrogenation of Coal Process [28,32]**

Based on these studies CFRI has developed a process on the concept of Multi Stage Hydrogenation of Coal (MSHC) to improve the quality and quantity of product, recycle solvent at low severity condition using less hydrogen. The basic principle on which the process has been developed rests on the identification of several retrogressive reactions which are inherent with coal to oil conversion. In the two stage liquefaction processes also this factor had been identified and had been attempted to be rectified. The process has been developed through batch autoclave studies in stages. This is a distinct three-stage process using temperature 400°C at 70 kg/cm<sup>2</sup> in the first stage, 400°C at 100 kg/cm<sup>2</sup> in the second stage and 400°C at 170 kg/cm<sup>2</sup> hydrogen pressure in the third stage. The process made distinct improvement in distillate yield, solvent quality and quantity and less hydrogen at a much less severity. A comparative technical performance statement as illustrated in Table 2 below amply justifies the uniqueness of the MSH process developed at CFRI

**Table 1 Expert Groups on Coal to Oil Conversion and their recommendations**

Year	Group/Committee	Recommendations
1948	J. C. Ghosh	Feasibility report from Koppers for 150,000 tons liquid fuels, at Rs. 35 crores investment. Aviation spirit to cost Rs. 0.81/gallong
1954	K. D. Malaviya	To set up a coal to oil plant.
1955	J.C. Ghosh	Invited project reports from Lurgi, Koppers and Kellog for a 0.1-0.3 m. t. liquid fuels (Aviation fuel 80,000-85,000 tons).
1958	C.F.R.I.	The Ghose committee report was updated
1969	C.F.R.I.	Integrated coal, petroleum and natural gas based plant in Assam. 2.5 mt of liquid fuels
1972	N.C.S.T (F.I.L)	To set up a prototype plant for production of 0.5-1.0 mt/yr distillates.
1974	Planning Commission	Coal gasification and also synthetic oil from gas
1974-77	K.R.Chakraborty	K.T. Gassification and F.T. synthesis by Kellog process at a capital cost of Rs. 700 crores (1 mt/yr)
1979	Working group of energy (N.B.Prasad)	To establish the need for R&D and draw a comprehensive plan.
1984	Expert group on conversion of coal to Gaseous and Liquid Fuels (G.S.Sidhu).	Bench scale (0.5 tpd coal) development work to be followed by a demonstration unit of 15-30 tpd liquid products with special reference to Assam coal by direct hydrogenation route. Pilot plant for a 1.0 m <sup>3</sup> catalyst volume (1.5 tpd C <sub>3+</sub> ). Ultimately demonstration unit of 20,000 - 50,000 tpy in VIII Plan through F-T route.

**Table 2 Comparative Technical Performance**

Stage	Lumas ITSL	CTSL	H-coal	EDS	LSE	CFRI-MSHC
T	450	400	445	450	475	400
P I	136	170	117	136	42	70*
T	-	-	-	-	-	400
P II	-	-	-	-	-	100
T	400	400	-	-	480	400
P III	136	170	-	-	350	170
conv.	38	58	43	37	23	90
solvent	A/P	A/P	A/P	A/P	A/P	A/P free

Hydrogen input in first two stages of MSHC is nil \* Steam pressure

T (Temperature in °C) and P (Pressure in kg/cm<sup>2</sup>)

A - Asphaltene, P - Preasphaltene, conv. - % conversion

In the MSH (Multi Stage Hydrogenation) process coal is dissolved in a hydrogenated solvent at a short contact time (SCT) in absence of any gaseous hydrogen to a predominantly preasphaltene rich product without allowing the build up of asphaltene and other retrogressive products like coke precursors. Conversion of preasphaltene to oil progressively needs much



less energy than the conversion of asphaltene. In the MSH process steam under certain condition has been used to interact with the preasphaltene rich product to degrade it to a lower molecular intermediate without allowing the build up of asphaltene and also the coke precursors. This intermediate product is a stable emulsion of coal intermediate and water having much less nitrogen and sulphur as compared to coal. The emulsion can be an ideal product for power generation since it has a high calorific value. This product on catalytic hydrogenation produced 90 % conversion of coal into oil, a solvent free of asphaltene and preasphaltene and hydrogen consumption was cut down to an appreciable level.

The expected benefits of MSH process are :

- *Very high conversion level - 85 % distillate yield*
- *Low reaction severity resulting in low operating cost*
- *Low hydrogen consumption thus further lowering of production cost*
- *Significant removal of sulphur and nitrogen content during conversion to oil thus improving product quality and simultaneously lowering cost for product quality augmentation at a later stage.*
- *Coal intermediate-water emulsion is a clean fuel for power generation*

### **Development of Reconstituted Coal–Water Emulsion as clean coal technology for power generation [33]**

CFRI, Dhanbad developed a water aided coal water emulsion process( funded by SSRC, Ministry of Coal, Govt. of India ) in a modular continuous reactor system at near atmosphere pressure in presence of cheap throwaway catalyst and water above its critical temperature in continuation of its earlier studies “Multi Stage Hydrogenation of Coal” with following features:

- ◆ Low process severity.( Temp. 400°C, Pressure 20 kg/cm<sup>2</sup> )
- ◆ Product GCV is comparable with fuel oil.
- ◆ Significant desulphurisation (~ 30%) & denitrogenation (~ 90%) of the feed could be achieved.
- ◆ No coke or asphaltene formation was observed.
- ◆ Process is suitable for all Indian non coking coals including NE coals and lignites
- ◆ Combustion tests conducted for product. Flame temperature measured through optical pyrometer was found to be > 1200°C and Flame Length was determined as > 2 meter.
- ◆ The emulsion developed is stable for >30 days and up to a temperature of ~80°C.

- ◆ The product (coal-water emulsion) developed as an outcome of the study is a new product and has not been developed anywhere else in the world till date

The product appeared to be a promising one and a more sophisticated scale up study is essential for further improvement in the quality of the product in terms of its fuel value and gathering necessary technical data as a first step for exploring the possibilities of commercial exploitation of the process. A project proposal for scale up studies is under consideration of Ministry of Coal, Govt. of India.

## **Recent happenings in coal to oil status & prospects**

### **China [34-37]**

1. HTI Inc., a subsidiary of Headwaters Inc., USA has a process licence agreement with Shenhua Group Corporation Limited, China's biggest coal company for a direct coal liquefaction plant to be built in China. The \$ 2 billion plant will have an ultimate capacity of 50,000 barrel/day capacity. The first likely to start production in late 2007. Shenhua Group intends to construct three more such plants in near future
2. China has signed a MoU with energy major Royal Dutch Shell to develop a Coal Liquefaction Project in Ningxia Region of China.
3. Chinese miner Ningxia Coal Industry Group Ltd. Is looking at a liquefaction plant needing more than \$ 3.6 billion in investment, with Sasol, South Africa.
4. The International Finance Corp(IFC), the private sector arm of the World Bank will invest \$ 10 million in Chinese company Xinao to support its efforts to convert coal into environmentally friendly fuel. In addition, IFC will grant Xinao a \$ 40 million loan and is also raising an additional loan upto \$ 128 million from commercial banks. The production is likely to begin in 2008.

### **Indonesia [38]**

Largest Coal Mining Company of Indonesia , Bumi Resources, is studying possibilities of liquefaction investment because much of Indonesian coal is low grade in terms of heat content and therefore not an attractive coal for export.



### **Philippines [36]**

Studies are underway in Philippines to explore possibilities of coal liquefaction

### **India [39-42]**

1. HTI Inc. have carried out a feasibility study for Oil India Limited to explore commercial viability of Assam Coals for coal liquefaction in direct mode. Coal India and OIL have signed an MoU in this regard for supply of 35 lakhs tons of coal annually on a joint partnership basis for running the proposed commercial plant.
2. Indian Oil Corporation Limited is evaluating a proposal for construction of 80,000barrel/day coal liquefaction plant based on Sasol technology.
3. Sasol is considering investing \$ 1 billion ultimately rising to \$ 6 billion in India for a project on coal liquefaction but is asking a government guarantee to buy its output at \$ 45/barrel if the world price falls below this price.

## **CONCLUSIONS**

With upcoming commercial ventures for coal liquefaction in both direct and indirect mode in China, the energy scenario has suddenly brightened up for India also. Reports of possible tie up of OIL and IOC with HTI Inc., USA and Sasol, South Africa respectively have certainly shown that our policy makers have finally decided to go for coal to oil and follow the Chinese model to improve our energy security. The prospects of developing indigenous technologies for coal liquefaction also looks bright but need proper nurturing and adequate funding. Proper synergy among various stakeholders is the need of the hour. There is an urgent and pressing need for going all out for coal liquefaction technologies to provide secured energy resources to fast expanding Indian industries to sustain the booming economic growth of the country. The policy makers of the country and executors of the policies need to take a careful look at the facts and figures noted below to finally decide whether we really need coal liquefaction to maintain our present pace of growth and become a economic super power in coming decades as has been projected internationally.

- ***India has large reserves of coal amounting to ~10% of the world's recoverable coal reserves( ~200 billion tons ) which is good enough to last for at least coming 200 years***

- ***Oil and gas reserves of our country are limited( 0.8% of global gas and oil reserves) and indigenous production hardly meets 25% of our oil requirements. India imported 75% of its oil requirement in 2004-05 which is expected to rise to 94% by 2030 costing \$2,000 billion a year.***
- ***Much of world oil supply originates from most politically unstable countries of the world. As such steady and uninterrupted supply of oil from these countries cannot be taken for granted***
- ***Commercial ventures in coal liquefaction will improves national energy security by lesser dependence on foreign oil as well as improved balance of trade***
- ***Since technology development is getting well established to manufacture transportation fuels from coal, it would be a wise policy to start a national programme on coal to oil conversion.***
- ***It may be noted that our neighbour China has opted to go all out for coal liquefaction to sustain its economic growth. China is likely to have its first commercial coal liquefaction plant in full operation in late 2007. Do we emulate them or wait for oil prices to come down and/or hope to find new oilfields in our country.***

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## **R & D achievements of the Central Fuel Research Institute in Fischer-Tropsch Synthesis**

### **Abstract**

The Central Fuel Research Institute, Dhanbad is the premier coal research organization in India. Fischer-Tropsch Synthesis (FTS) is one of the major areas of research in this institute for last several decades. The intensity of research in FTS varies from time to time and is more related to strategic and variation of the petroleum crude price. The most expensive section of an FT complex is the production of purified syngas and so its composition should match the overall usage ratio of the FT reactions, which in turn depends on the product selectivity. Initially several iron based catalysts have been developed in sixties for FTS where syngas source was supposed to be from coal gasifiers. A Process Development Unit of 3.5 L catalyst volume was installed and studies were performed on iron based catalysts. However, in nineties FTS was persuaded at CFRI in collaboration with Russia for the synthesis of wax where cobalt based catalysts were developed. Presently, extensive studies are being conducted for development of a commercial catalyst under the Taskforce Projects of CSIR, Govt. of India and three cobalt based catalysts have been developed for this purpose. A sophisticated laboratory with analytical and catalyst testing facilities has been established at this institute and is promised to deliver its full potential to add to the oil pool of the country through FTS route.

### **Introduction**

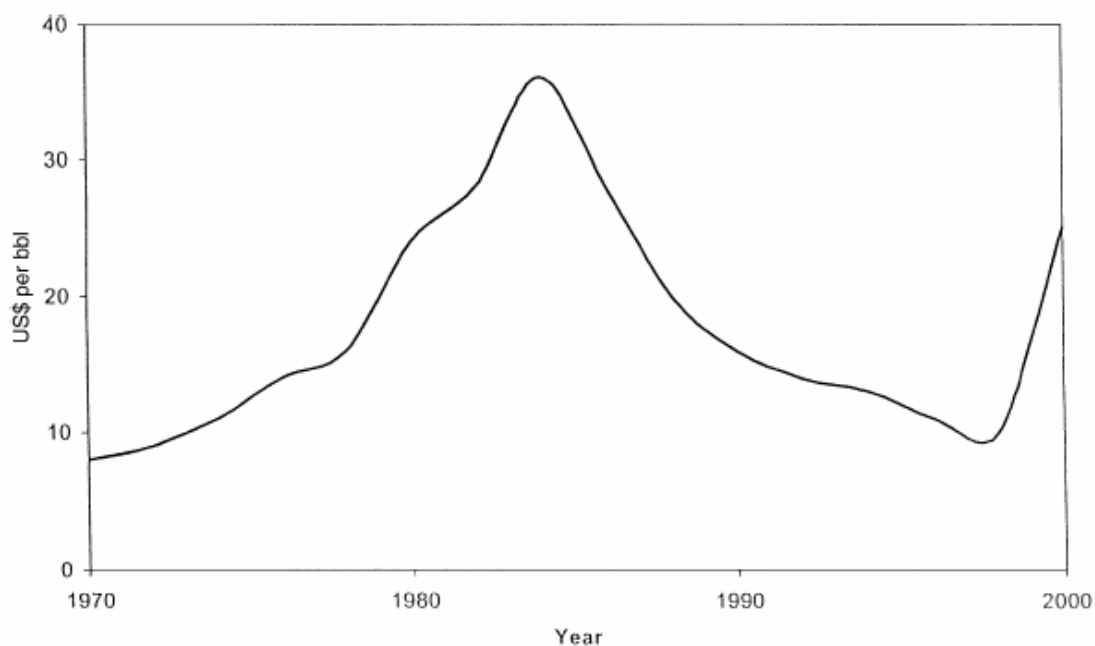
Predominant source of world's fuel and chemical is petroleum crude oil and estimated reserves of methane & coal exceeds that of crude oil by factors of about 1.5 and 25, respectively. Production of syngas from methane or coal and conversion of the syngas to a range of fuels and chemicals could become increasingly of interest as the reserves of crude oil are depleted and/or the price of crude rises. The industrial application of the FT synthesis started in

Germany and by 1938 there were nine plants in operation having a combined capacity of about  $660 \times 10^3$  t per year [1]. These plants ceased to operate after the World War II; however interest in the FT process remained because at this stage there was the persistent perception that the reserves of crude oil were very limited. Based on syngas produced from methane, an FT plant with a capacity of  $360 \times 10^3$  t per year was built and operated in Brownsville, TX, during the 1950s but a sharp increase in the price of methane caused the plant to be shut down. During the same time period, based on the world-wide prediction of increasing crude oil prices an FT plant based on coal came on stream in Sasolburg, South Africa. Even before construction of this plant was completed, however, the huge oil fields of the Middle East were discovered and consequently the predicted rise in the price of crude oil did not materialize and interest in the FT process all but disappeared.

The economic viability of the FT synthesis depends on the price of crude oil and as it has varied considerably over last three decades (Fig. 1), the decision to construct an FT plant is always associated with risk. The oil crises of the mid 1970s prompted Sasol to construct two much larger coal-based FT complexes and this time things worked out better as the two plants came on-line in 1980 and 1982 when the price of crude exceeded US\$ 30 per barrel. At that stage the combined capacity of the three Sasol plants was about  $6000 \times 10^3$  t per year. Based on methane, the Moss gas plant in South Africa and the Shell plant at Bintuli, Malaysia, came on stream in 1992 and 1993, respectively. At that stage, however, the price of crude had dropped to about US\$ 15 per barrel and so the timing of these two plants, with capacities of about  $1000 \times 10^3$  and  $500 \times 10^3$  t per year, respectively, was not as fortunate as had been the case for the two new Sasol plants. Crude oil prices continued to decline to about US\$ 10 per barrel, but in 1999 there was a dramatic increase and at the time of writing the price is again above US\$ 30 per barrel. Environmental/political considerations are also factors. Thus, for instance, certain large new oil fields are associated with high levels of natural gas the

flaring of which is prohibited. In order to recover the crude oil the construction of FT plants to convert the methane to oil is being considered.

The FTS is a catalyzed chemical reaction in which carbon monoxide and hydrogen are converted into liquid hydrocarbons of various forms. Typical catalysts used are based on iron and cobalt. The principal purpose of this process is to produce a synthetic petroleum substitute for use as synthetic lubrication oil or as synthetic fuel. FTS involves three process steps: (1) the generation of the synthesis gas, a mixture of CO and H<sub>2</sub>; (2) the conversion of the syngas to a mixture of hydrocarbon and oxygenate products; (3) hydroprocessing of synthesis products to produce transportation fuel products, today with emphasis on diesel.



**Fig 1: Change in Global Crude Oil Price**

A number of approaches are available for the conversion of synthesis gas. Two catalysts—iron and cobalt based—have been widely used and these are the only ones receiving serious consideration today. The major difference between these two catalyst types is the formation of the oxygen containing



products: with cobalt the oxygen in the syngas is rejected as water and with iron the oxygen is rejected as carbon dioxide. Since cobalt catalysts do not have water-gas-shift (WGS) activity, the feed gas must have a  $H_2/CO$  ratio of about 2.0–2.3. Iron based catalysts have significant WGS activity so that much lower  $H_2/CO$  ratio feed gas can be utilized. Thus, the perception has developed that the iron catalyst is preferable for syngas with a low (0.5–1.3)  $H_2/CO$  ratio, typical of those produced from coal as the carbon source, and that the cobalt based catalyst is preferred for high (2.0+)  $H_2/CO$  ratios, typical of those produced from natural gas. Since natural gas usage is the primary driving force for FTS today, cobalt has been receiving much attention. However, utilization of coal indicates that iron catalysts should not be automatically eliminated from consideration for syngas derived from natural gas.

The Central Fuel Research Institute, Dhanbad being the premier Coal Research organization in the country, has been working on FTS for last five decades. Initial research on FTS was aimed for liquid fuel synthesis from coal derived syngas and iron based catalysts was studied for this purpose. However, during last one decade, attention has been focused towards the utilization of vast natural gas reserve of India hence cobalt based catalysts are studied for conversion of natural gas derived syngas for synthesis of liquid hydrocarbon or wax (Fig 2).



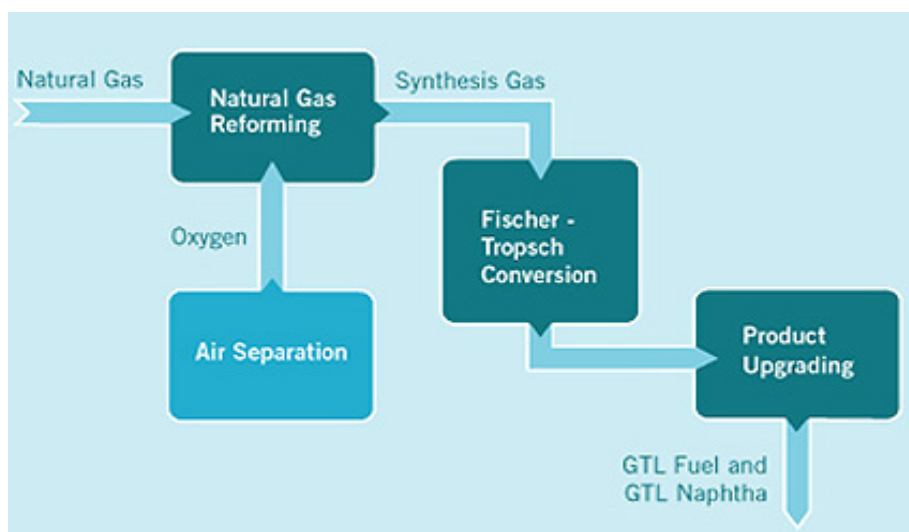
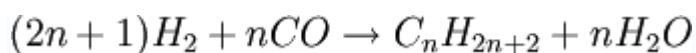


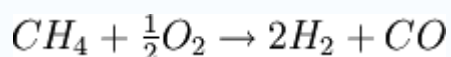
Fig 2: Pathway of conversion of natural gas to liquid fuels through FTS

## Reaction Chemistry of FTS

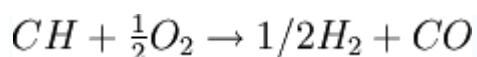
The original Fischer-Tropsch process is described by the following chemical equation:



The initial reactants in the above reaction (i.e., CO and H<sub>2</sub>) can be produced by other reactions such as the partial combustion of methane (in the case of gas to liquids applications):



OR by the gassification of coal or biomass:



The mixture of carbon monoxide and hydrogen is called synthesis gas or syngas. The resulting hydrocarbon products are refined to produce the desired synthetic fuel. Chain growth pathways and possible secondary reactions of olefins in the FT-Synthesis are shown in Fig 3.

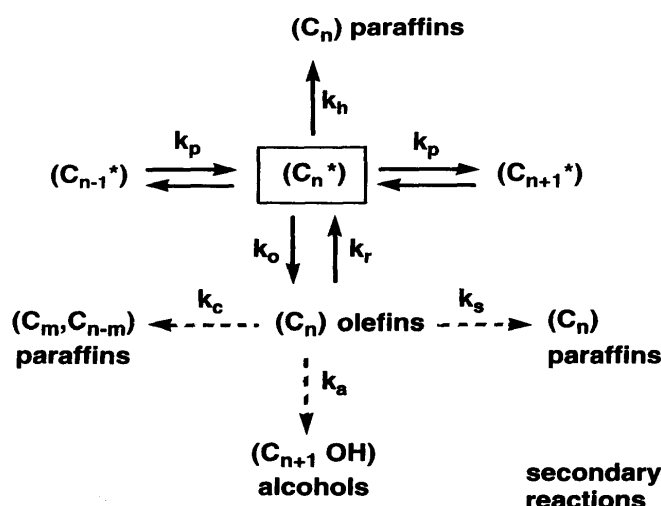


Fig 3: Chain growth pathways and possible secondary reactions of olefins in the FT-Synthesis

## International Status

Since 1955, **F-T synthesis** is being exploited commercially by **SASOL** in South Africa. Sasol is a synfuel technology supplier established to provide petroleum products in coal rich but oil poor South Africa. The firm has built a series of FT coal to oil plants, and is one of the world's most experienced synthetic fuels organizations and now marketing a Natural Gas To Oil Technology. It has developed the world's largest synthetic fuels project, the *Moss gas* complex at Mossel Bay in South Africa that was commissioned in 1993. To increase the proportions of higher molecular weight hydrocarbons, Sasol has modified its Arge reactor to operate at higher pressures. Sasol has commercialized four reactor types with the **Slurry Phase Distillate** process being the most recent. Its products are more olefinic than those from the fixed bed reactors and are hydrogenated to straight chain paraffins. The slurry phase distillate converts natural gas into liquid fuels, most notably superior quality diesel. Another technology uses the **Sasol Advanced Synthol (SAS)** reactor to produce mainly light olefins and gasoline fractions. Sasol has developed high performance cobalt based and iron based catalysts for these processes. The company claims a single module or sasol slurry phase

distillate plant which converts 100 MM scfd of natural gas into 10,000 barrels/day of liquid transport fuels can be built at a capital cost of US \$ 25,000 including utilities, off-site facilities and infrastructure units. Due to steeply growing market demand and for the utilization of large natural gas reserves many new players has come in GTL market which are now licensing their GTL technologies. SASOL, SHELL & SYNTROLEUM are the major licensing companies and other companies like Exxon, Statoil, Rentech are also spreading their business worldwide. Shell has carried out R&D since the late 1940s on the conversion of natural gas, leading to the development of Shell Middle Distillate Synthesis (SMDS) process, a modified FT-process. This process aims on maximizing the yield of middle distillates. First commercial SMDS project started in 1993 at Bintulu-Malaysia. In this process natural gas is first partially oxidized into syngas ( $\text{CO} + \text{H}_2$ ) in Shell gasifier. This syngas is then converted into heavy paraffins in a fixed bed multi-tubular reactor. This heavy paraffin is then selectively hydrocracked & isomerized for getting the middle distillates. The product obtained by this process is free from contaminants, sulphur, aromatics and heavy metals. Syntroleum Corporation is the developer, user and licensor of the Syntroleum Process, which is a proprietary process for converting natural gas (or synthesis gas from coal) into synthetic liquid hydrocarbons. The Syntroleum Process produces synthetic liquid hydrocarbons, also known as synthetic crude oil, that are virtually free of contaminants that are normally found in products made from conventional crude oil. These synthetic liquid hydrocarbons can be further processed into higher margin products through conventional refining techniques, including Syntroleum's proprietary Synfining Process. These products include: Ultra-clean liquid fuels for use in internal combustion engines and fuel cells; specialty products such as synthetic lubricants, process oils, waxes, etc. Costs to produce these products using GTL technology are increasingly competitive with conventional process technologies. Moreover, the ultra-clean properties of GTL fuels meet or exceed the new and proposed environmental requirements that will soon go into effect for the U.S. and Europe.

Key advantages to the Syntroleum Process include its use of air in the conversion process, which is economically competitive and inherently safer than the requirement for pure oxygen in other GTL technologies; and Syntroleum's proprietary catalysts, which enhance conversion efficiency of the catalytic reaction. These features help to reduce capital and operating costs of plants, and permit smaller plant sizes, including mobile plants that can be mounted on barges for offshore service. Research indicates that the Syntroleum Process can be economically applied in plant sizes from less than 20,000 to over 100,000 barrels per day.

RENTECH proposed electrical power and FT fuels project in West Australia as announced October 29, 1999. Rentech Inc. is the developer and licensor of a patented and proprietary Fischer-Tropsch, gas-to-liquids process, for conversion of synthesis gas made from natural gas, industrial off-gas, or solid or liquid carbon-bearing materials into high-value fuels, products and chemicals. These products include cleaner burning, sulfur- and aromatic-free diesel fuel, naphtha, waxes and fuel for fuel cells.

Preliminary project sizing is for a 200-megawatt electrical power plant and a 6,000-barrel per day Fischer-Tropsch plant. It is expected that Texaco's gasification technology will be used to gasify the lignite to product a syngas consisting of hydrogen and carbon monoxide. The syngas will in turn be used to produce electricity and feed the Rentech-designed Fischer-Tropsch reactor. With its large gas reserves, Norway's STATOIL has been developing catalysts and process reactors for the FT-process to produce middle distillate from natural gas. This process employs a three-phase slurry type reactor in which syngas is fed to suspension of catalyst particles in a hydrocarbon slurry, which is a product of the process itself. The process continues to be challenged by catalyst performance and the ability to continuously extract the liquid product.

EXXON has developed a commercial FT system from natural gas feed stock. They claim for the slurry reactor and proprietary catalyst systems result in high productivity and selectivity along with significant economy of scale benefits. Exxon employs a three-step process: fluid bed synthesis gas generation by catalytic partial oxidation; slurry phase FT-synthesis; and fixed bed product upgrade by hydro isomersation. The process can be adjusted to produce a range of products. More recently they have developed a new chemical method based FT-process to synthesize diesel from natural gas. Exxon's better catalysts and improved oxygen-extraction technology have reduced the capital cost of the process and is actively marketing the process internationally.

One GTL-Microsystems of UK claims to have developed a process -GTL Microsystems modular Process Intensification (PI) reactors for Steam Methane Reforming (SMR) and Fischer-Tropsch process, by using micro reactor technology. This has reduced size of about one tenth of conventional GTL plants, which uses a unique metallic substrate catalyst with high conversion of C<sub>5+</sub> hydrocarbons. This process has low capital cost and superior economics, compact in design, high efficiency and no compressed air, oxygen or water is required for its operation.

Another company VELOCYS of USA is also claiming for the micro channel reactor technology application to FT synthesis reactions.

A group of Japanese companies intent on making gas-to-liquids (GTL) fuels a commercially viable overseas business by 2011 plans to start building a trial GTL plant in Niigata. This facility is targeted to begin operation in 2008 with a daily output capacity of about 500 barrels per day of synthetic fuels and chemicals using natural gas.

The partners include Nippon Oil, Nippon Steel, Japan Petroleum Exploration, Inpex, Cosmo Oil, Chiyoda and the government-owned Japan Oil, Gas and

Metals National Corp (JOGMEC), along with support from the Ministry for Economy, Trade and Industry (METI).

JOGMEC has been researching GTL technology since 1998, with an emphasis on the efficient production of the syngas used in the Fischer-Tropsch (FT) process that actually produces the end products.

Working in partnership with Chiyoda (which developed the catalyst), JOGMEC is developing a GTL process that eliminates the need for three units usually found in the syngas section of a conventional GTL operation, thereby potentially significantly reducing the cost. There shall be no CO<sub>2</sub> removal unit, oxygen plant and syn gas conditioning unit. The JOGMEC process has only been tested in very low production pilots (7 bpd). This trial is designed to help perfect in support of building an overseas plant (likely in Indonesia) with a production capacity of 15,000 barrels per day. The major GTL Technology overviews are shown in Table-1.

It is evident that continuous developmental work on F-T synthesis is being carried out since the beginning of this century to develop alternative source of liquid fuels for particular situation and to meet the need of coming decades.

It is found that fundamental work F-T process development is being carried out in Universities & Institutes and application part is being carrying out by Multinational & Large Companies.

### **CFRI's Status**

The research work on F-T synthesis in India is considered to have begun within two decades of the invention of the process in 1913 in Germany. The first Indian research paper was published in 1935. Since then, investigational studies on F-T synthesis is continuing. Most of these studies were to examine the effectiveness of catalyst and obtain data on adsorption kinetics and

mechanism aspects of F-T synthesis. These studies were initially started at Universities of Bengal and followed up by IISC (Bangalore), CFRI (Dhanbad), IIT (Kharagpur).

In the early 1960s, IIT (Kharagpur) put up a pilot plant of considerable size (product capacity: 100 gals per day) and operated the same. This was by far the biggest F-T synthesis unit in the country. However, this unit used fixed bed reactor of Pre-World War II period, was not suitable for engineering developmental studies.

CFRI, which started investigations on F-T synthesis from 1952 put up a fluidized bed reactor of maximum catalyst capacity of 1.2 liter by the end of 1950s and operated the same for about a decade. By the end of 1960s, studies on F-T synthesis practically stopped at IIT (Kharagpur) and IISC (Bangalore). Studies at CFRI, however, continued on low-key basis till the middle of 1970s.

In the early 1970s, when the oil crisis occurred, interest on F-T synthesis was revived and IICT (Hyderabad), IIT (Bombay) took up projects on F-T synthesis. In the early 1980s, IIT (Madras) also started working on F-T synthesis. From 1976, CFRI started working on the design of scaled up F-T synthesis unit with fixed bed reactor system to install the same and study the catalysts developed at CFRI.

Since the above studies were taken up institution wise, the contributions to the science of F-T synthesis were diverse in nature. During the early part of investigation, nickel, cobalt and iron based catalysts were studied. These studies were aimed to examine the yield of liquid hydrocarbons, activity aspect of the catalyst, role of promoters as well as basic studies like adsorption of CO and H<sub>2</sub> over these catalysts. After the Second World War, emphasis was put to develop iron catalyst for F-T synthesis as iron had some advantages over cobalt and nickel. From mid-50s, investigations, therefore,

concentrated on the development of iron based F-T synthesis catalysts. Considerable amount of studies had been carried out metals of group I, II, III, IV, VI and VII of periodic table as promoters for iron catalysts.

**Table-1 The major GTL Technology overviews.**

Sr. No.	Company (Location)	O <sub>2</sub> Plant	Syn gas Production (Catalyst source)	FT-Synthesis (Catalyst/ Source)
1.	GTL-Microsystems (UK)	None	Steam-Methane-Reforming in PI Reactor (Microsystems)	Micro Channel reactor (Microsystems)
2.	VELOCYS (USA)	None	SMR (Velocys)	Micro Channel reactor (Velocys)
3.	JOGMEC (Japan)	None	Tubular reformer (Chiyoda)	Slurry bed (Co/NSC)
4.	SASOL (SA)	Reqd.	Auto thermal reformer (Topsoe)	Slurry bed (Co/Sasol)
5.	SHELL (Malayasia)	Reqd.	POX (Shell)	Fixed bed (Co/Shell)
6.	SYNTROLEUM (USA)	None	Auto thermal reformer (Syntroleum)	Slurry bed (Co/Syntroleum)
7.	Exxon Mobil (USA)	Reqd.	Auto thermal reformer (ExxonMobil)	Slurry bed (Co/ExxonMobil)
8.	Conoco (USA)	Reqd.	CPOX (Conoco)	Slurry bed (Co/Conoco)
9.	BP (USA)	Reqd.	Compact reformer (BP)	Slurry bed (Co/BP)

At IISc (Bangalore), investigation to understand the mechanism of FTS reactions were carried out using carbon balance method, in addition to chemisorption studies on cobalt and iron catalysts. Such carbon balance method helped to understand the product distribution pattern and reaction mechanism in respect of intermediate complexes had also been studied.

At IIT (Kharagpur), Fe based F-T catalyst was tested in the pilot plant and sufficient product was obtained for characterization. However, only one catalyst was studied and because of using obsolete type of reactor, the plant



was not suitable for engineering development studies. Further, bench scale slurry phase reactor system was studied for some time.

At IIT (Bombay), some studies on the preparation technique and nature of supported mono and bimetallic catalysts using metals of group VIII had been carried out. It was reported that these catalysts, however, did not produce higher hydrocarbons during synthesis.

At IIT (Madras), some studies on the basic aspects of F-T catalysts was done using modern instrumental technique. These studies were related to the role of support and promoter in F-T catalysts and also mode of carbon monoxide activation and adsorption.

At IICT (Hyderabad), some studies on F-T catalysts have been carried out recently. Different methods of preparations of supported F-T catalysts and characterization of these catalysts have been carried out and studied. Synthesis over multi-metallic catalyst system has been reported.

At CFRI, extensive studies on iron catalysts from basic and applied angles had been carried out. Three different types of F-T synthesis reactors, viz., fixed bed; fluidized bed and slurry phase had been tested. The emphasis was mainly on fluidized and fixed bed type of reactors. Fluidized bed reactor system has been found to be suitable for olefins, gasoline and chemicals like alcohols and ketones production. The fixed bed reactor is suitable for paraffinic and high molecular weight hydrocarbons, like middle distillate and waxy products. The studies in lab scale slurry phase reactor showed production of low molecular weight ( $C_1$ - $C_4$ ) gas phase hydrocarbons.

In the field of catalysis, catalysts preparation technique, role of promoter and changes in catalyst phases during their use have been studied. Based on such studies, CFRI has developed catalyst for the production of liquid fuels with high yield of middle distillate from synthesis gas. The results of iron based FT catalysts studied at CFRI are presented in Table-2.

**Table 2 Previous GTL Runs of Iron catalyst in Liquid Fuels Section**

Catalyst Name	H <sub>2</sub> /CO	Pressure Kg/cm <sup>2</sup>	Rxn. Temp. °C	GHSV	Run time (h)	Yield Aqs / Oil / Wax
FT/CS- 23/151	1.56- 1.70	15-15.5	200-205	450-460	129	108.2/ 148.5 /18.69
FT/CS-14/77/167	1.70	15-16	200-215	450	119	134 /84 /---
USA Catalyst	1.70	15-16	220	450	48	8 / 4/-----
FT/CS/15/79	1.70	15-16	200	300	134	153 /93/----
FT/CS- 23/151	1.56- 1.70	15-16	200-205	450-460	53	137.5 /56.5/ -
FT/CS/23/B3/181	1.55	10-13	205-210	450	76	41 / 51/---

The laboratory scale studies of catalysts were translated into scale-up studies. For this purpose, Fixed Bed Tubular Reactor (Catalyst Capacity 150 ml) and Process Development Unit (Catalyst Capacity 3.20 liter) have been installed at CFRI. The promising catalysts developed earlier were undergone large scale testing in the Process Development Unit.

Most of the FTS studies were performed under the in-house projects of CFRI but in 1998, a project titled **“Fischer-Tropsch (F-T) Synthesis: Development of Catalysts for Wax Production”** was sponsored by Dept. of Science and Technology, Govt. of India under the Indo-Russian collaboration scheme (ILTP). This study was done in the temperature range of 180 – 220°C. It was observed that although pressure increases the selectivity for higher alkanes, due to limitations by the material construction of the STR, it was kept at 1.2 – 1.25 MPa. The work on the conversion of syngas to wax was initiated with iron catalyst. Three iron-based catalysts – two supported on clay having different proportion of promoter and the third with silica support were prepared by precipitation technique. One Fe-based clay supported catalyst showed appreciable wax yield which was tested for 208 h at GHSV 450h<sup>-1</sup> and 1.2 MPa of pressure. At 205°C and H<sub>2</sub>/CO ratio of 1.7, wax yield was 15% whereas for H<sub>2</sub>/CO ratio of 1.6 and 210°C, wax yield was 32%. The wax yield

for the Fe-catalysts was low so cobalt-based catalysts, which are known for its selectivity towards high molecular weight compounds, were taken up. Two cobalt-based catalysts (CFRI/C1 and CFRI/C2) which were prepared differently and showed maximum activity, were compared with one Russian catalyst (cobalt-based). The wax yield for CFRI/C1 was 18.3 wt% of C<sub>5+</sub> fraction at 195°C, H<sub>2</sub>/CO =1.7 and GHSV~550h<sup>-1</sup> and the other catalyst CFRI/C2 showed maximum wax selectivity 58.3 wt% C<sub>5+</sub> fraction at 1.2 MPa, 200°C, GHSV~ 650h<sup>-1</sup> and feed H<sub>2</sub>/CO = 2.0. Analysis of the wax component by simulated distillation showed the maximum carbon number of wax was 62. Very recently extensive investigations have been carried out on alumina supported Cobalt catalyst under the Taskforce Project of CSIR, India, titled, **“Development of Gas to Liquid Technology for Fischer-Tropsch & DME Fuels”**. Three different formulations of Cobalt based catalyst (FT-Cat12, FT-Cat12A and FT-Cat14) for middle distillate synthesis have been prepared, characterized and its activity evaluated. All the catalysts have been tested at 500-1000 h<sup>-1</sup> GHSV's, 20 bar reaction pressure, H<sub>2</sub>/CO ratio 2.0, and at 220-250°C reaction temperature.

FT-Cat 12, FT-Cat 12A and FT-Cat 14 have been tested for 125 Hrs, 122 Hrs and 78.5 Hrs of continuous operation respectively. FT-Cat 12, FT-Cat 12A and FT-Cat 14 have yielded 207 ml, 147 ml and 101 ml of C<sub>5+</sub> Hydrocarbon Liquid respectively, which was characterized in GC-FID-SIMDIST. The SIMDIST results show the yield of naphtha (b.p up to 140°C) varies from 4-25 %, middle distillate (b.p 141-360°C) varies from 61-83 % and waxy oil (> 360°C) varies from 8-33 % of total liquid product. The CO conversion varies from 73-95%. The selectivity of C<sub>5+</sub> hydrocarbon at 500 h<sup>-1</sup> GHSV of FT-Cat 12, FT-Cat 12A and FT-Cat 14 were 77, 72 and 66% respectively.

It has been observed that CO conversion increases and C<sub>5+</sub> hydrocarbon liquid selectivity decreases with increase in space velocity of feed. The C<sub>5+</sub> Hydrocarbon Liquid selectivity is maximum at low space velocities, which may be due to larger residence time.

Also, as per SIMDIST result, IBP of liquid hydrocarbons of product varies from 70-75 °C and FBP varies from 400-450 °C. As per the yield and selectivity pattern of the developed catalyst system it can be emphasized that these may be successful formulations leading to commercialization for converting the huge Natural Gas Reserves of our country and adding up in the current oil pool.

Further, in the 11<sup>th</sup> plan of Govt. of India, a project titled, “**Development of Coal to Liquid Technology**” is submitted which has been cleared by the Sectoral Committee on ‘Energy Resources & Technology’ during its meeting on 15<sup>th</sup> July 2006 at CSIR New Delhi. For the CTL technology it has been targeted to develop suitable catalyst and system/process for converting the coal derived syngas into liquid hydrocarbons specifically the middle distillate which is a mixture of diesel and kerosene.

## **Conclusions**

CFRI being actively involved in coal research from its inception, coal gasification remains one of its main research topics. Dhanbad is in the heart of coalfields of Eastern India and availability of coal is beyond doubt and hence syngas generation via coal gasification is a viable route. Moreover, immense Coal Bed Methane (CBM) reserve is estimated in this region. CBM may also be used for the production of syngas. CFRI has already patented several iron based FT-catalysts for middle distillate/wax production via FT synthesis and presently achieved considerable success for the development of cobalt-based FT-catalysts for the production of middle distillates i.e. diesel and kerosene.

CFRI has developed three cobalt-based catalysts, which have been named as ALCOZAR-I, II and III<sup>®</sup> for synthesis of middle distillate from syngas. FTS products, which are manufactured from natural gas, are extremely pure and free from heavy metals, sulphur, nitrogen and aromatics. These products are environment friendly and sold globally for variety of applications. These

catalysts have shown promising results and expected to be commercial catalyst for which India is struggling for last several decades. Initiatives have already been taken with private and public sector enterprises for scale up studies of the catalysts and collaboration are in the pipe line with the enterprises. In the present context, commercial FT fuels production may be possible by collaboration of Research Institution and Industrial/ Engineering partners in foreseeable future.

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## ANNEXURE

## 1. List of Publications

Sl. No.	Title	Authors	Journal
1.	Fischer-Tropsch synthesis, Part-I, Activity of Iron catalyst at atmospheric pressure.	N.G.Basak, K.K.Bhattacharya	Journal of Scientific & Industrial Research: 12B, 617(1953)
2.	Fischer-Tropsch synthesis, Part-II, Studies with Fe-Cu-Co-K <sub>2</sub> O Catalyst at normal pressure.	N.G.Basak, T.V.Subramaniam, J.N.Bhatnagar, A.K.Roy	Journal of Scientific & Industrial research: 12B, 622(1953)
3.	Fischer-Tropsch synthesis- A study of reaction mechanism	N.G.Basak, K.K.Bhattacharya	Journal of Fuel: 27, 195, (1954)
4.	An X-Ray study on the nature of iron catalyst used in decomposition of alcohols.	B.K.Banerjee, S.R. Srinivasan,, N.G. Basak, A. Lahiri	Proceedings of symposium on 'Contact Catalysis' Calcutta May 4-5, 1956, p59-64.
5.	Crystal chemical structure of FT catalyst in different forming stages and in active synthesis as revealed in X-Ray diffraction studies.	D.P.Dutta, K.K.Bhattacharya, N.G. Basak, A. Lahiri	Proceedings of symposium on 'Contact Catalysis' Calcutta May 4-5, 1956, p65-79.
6.	Pre-sorption of CO & H <sub>2</sub> on an iron FT catalyst.	N.C.Ganguly, K.A.Kini, N.G. Basak, A. Lahiri	Journal of Scientific & Industrial research: 16B (2), 86 (1957)
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| 37. | Hydrogenation of Carbon Monoxide on Unsupported Fe-Mn-K Catalysts for Synthesis of Lower Alkenes : Promoter Effect of Manganese | Das, C.K.<br>Das, N.S.<br>Chowdhury, D.P.                                      | <i>Appl. Catal.</i> , 111 (2), 119 (1994)  |
| 38. | Processing and Utilization of Iron Ores as Catalyst in Fischer-Tropsch Synthesis  | Dasandhi, Y.C.<br>Basu, S.<br>Bandyopadhyay, P.                                | <i>Indian Chem. Engineer</i> , 37 (1 & 2), Section A, 45-52 (1995)   |
| 39. | Synthesis of Olefins from Syngas by Modified Fischer-Tropsch Catalyst   | Basu, P.K.<br>Roy, Sunil K.<br>Sarkar, P.K.                                    | <i>Minetech.</i> , 17 (2 & 3), 41 (1996)   |
| 40. | Synthesis of Liquid Hydrocarbons (Diesel Range) from Synthesis Gas Through Modified FT Synthesis Over                           | Roy, Sisir Kumar<br>Sharma, M.P.<br>Sharma, B.K.<br>Roy, S.K.<br>Kumar, Suresh | <i>Minetech.</i> , 17 (2 & 3), 64 (1996)   |





	Catalyst	Sarkar, P.K. Roy, Sisir Kumar	
41.	Synthesis of Lower Olefins from Synthesis Gas through Modified Fischer-Tropsch Process	Basu P.K. Roy, Sunil K. Sarkar, P.K. Roy, Sisir Kumar	<i>Proc. Int. Symp. Coal-96, CFRI, Dhanbad</i> , 18-19 Nov. (1996), pp.451-469
42.	Studies on Iron-ore based Fischer-Tropsch Synthesis Catalysis	Dasandhi, Y.C. Bandopadhyay, P.	<i>Chem. Engg. World</i> , 32 (1), 47 (1997)
44.	Studies on cobalt based FT catalyst & characterization using SEM & XPS technique.	B.K.Sharma et al	Applied Catalysis A: General, 211(2001).203-211.
45.	FT Synthesis with Co/SiO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub> catalyst & steady state modeling using artificial neural network	B.K.Sharma et al	Fuel, Vol 77,no-15, p1763-1768
46.	Theoretical feasibility for catalytic property of certain ores	Sudip Maity	Current Science, V 80, No. 5, 2001, p 628 – 630

## 2. List of Patents

Sl. No.	Title	Patent No. & Date	Inventors
1.	A process for the preparation of an iron catalyst useful for the production of synthetic liquid fuels having 62-69 % yield of middle distillate fraction from synthesis gas.	175209, 08/12/1995	Samiran Basu, G.C.Nandi, S.B.Basu, U.Bhattacharya, V.A.Krishnamurty & Dr. R. Haque
2.	A process for the preparation of an iron catalyst useful for the production of synthetic liquid fuels having 62-69 % yield of middle distillate fraction from synthesis gas.	179798, 28/08/1998	Samiran Basu, G.C.Nandi, S.B.Basu, U.Bhattacharya, V.A.Krishnamurty & Dr. R. Haque
3.	A process for production of wax from syngas over an iron catalyst.	30/12/1994	Samiran Basu, G.C.Nandi, P.K.Basu, S.B.Basu, U.Bhattacharya, S.K.Mitra, Y.C.Dasandhi, S.S.Bhattacharya
4.	A process for preparation of catalyst for production of wax from syngas over an iron catalyst.	30/12/1994	Samiran Basu, G.C.Nandi, P.K.Basu, S.B.Basu, U.Bhattacharya
5.	A process for preparation of Fe-Mn catalyst for production of lower olefins from syngas.	183629, 15/09/2000	C.K.Das, N.S.Das
6.	Preparation of catalyst useful for low temperature steam reforming of hydrocarbon.	Filed-2003	S.K.Roy, P.Samuel, Sudip Maity, D.K.Sakhre, S.K.Maitra & S.N.Mukherjee
7.	A Process for The Preparation of a Catalyst, Useful for the Production of Hydrocarbons from Synthesis Gas	Filed in 2005, 8-Countries	Samuel P, Maity S, Dasandhi YC, Mukherjee S, & Maitra S K
8.	Catalyst for Synthesis of Hydrocarbons from Synthesis Gas.	Filed in 2005, 8-Countries	Samuel P, Maity S, Dasandhi YC, Mukherjee S, Maitra S K



# Environmental Management Division

# **Environmental R & D Activities of CFRI: An Appraisal**

## **Abstract**

Central Fuel Research Institute (CFRI) being one of the premier research institutes of Council of Scientific & Industrial Research (CSIR) is actively involved in performing R & D work on the exploitation of coal and its conversion products since its setting up in the year 1946. As per the requirement of the cleaner technologies and environment, the R & D activities also comprise of resolving the problems of environmental concerns of coal-based industries including the monitoring and abatement of the various pollutions arising from such applications. In view of that various efforts have been and presently being taken up by the institute through its existing Environmental Management Division. These include, waste management in general and fly ash being generated from TPPs in particular, reclamation/bioremediation of waste/degraded land, low lying area, mine spoil and abandoned ash pond, mitigation of green house gases (GHGs), monitoring of air-water-soil pollution, pollution due to vehicle in the surroundings of coal-based industries. Despite, the desulphurization of solid and liquid fuels by radiolytic and oxidation/solvent extraction methods have also been studied, respectively. In the present paper, an epigrammatic focus on the various activities (previous, present and future) since the beginning of the environmental activities in the Institute has been made.

## **Introduction**

CFRI is actively engaged in carrying out research and development (R & D) work on the utilization of coal and its conversion products. The environmental problems being inseparably associated with coal conversion and processing, the institute also does R & D work in the area of environmental science and technology related to coal-based industries. This comprises of the management of solid waste from coal-based industries, principally the fly/bottom/pond ash from TPPs and mine refuse/spoil produced during mining operations; development of environmental standards; making environmental impact assessments and management plans; mitigation of GHGs; addressing the associated environmental problems like monitoring of the quality of air, water, soil and suggesting/developing appropriate methods for their minimizing/ abatement. In the context of sustainable management of solid waste from TPPs, systematic R & D work has been carried out, over the past two decades or so. This comprises detailed physico-chemical characterization and determination of potentially toxic trace and heavy metals and radionuclides in coal ash and its bulk use in brick making, civil construction materials, agriculture/forestry sector (as liming agent, soil modifier and amendment, source of essential plant nutrients, and also for the reclamation

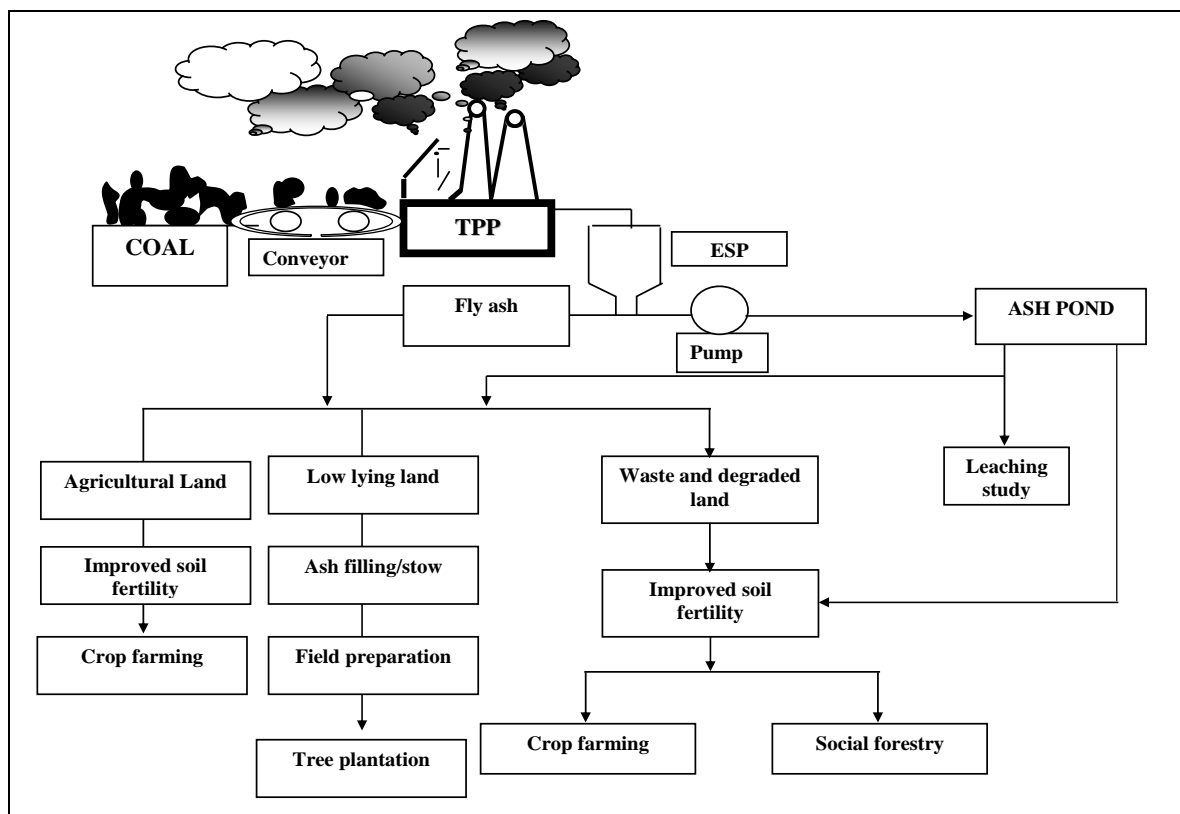
of waste/degraded lands/mine spoil/abandoned ash ponds) and other value added products such as scouring powder, etc.. In recent past, R & D work has been extensively intensified for developing fly ash soil amendment technology (FASAT) by gainfully utilizing fly ash/ pond ash alone and in combination with biological/organic amendments and industrial wastes like press mud, etc. The problems of air and water pollution caused by coal-based industries such as coal mining, washeries, coke ovens, TPPs, steel making and other industrial areas of the country have been studied and the strategies for their minimization/abatement have been put forward. Additionally the environmental concerns of SO<sub>2</sub> being generated from the combustion of high-S coal and diesel have also been undertaken into earlier study and new methods such as radiolytic and oxidation/solvent extraction have been developed.

### **Management of fly ash/ pond ash for agro-forestry**

If the energy generation from coal in TPPs is the most attractive side of coal utilization, the simultaneous generation of fly ash is its gloomy side. Presently about 110 million tons of fly ash is being generated per annum from 85 TPPs in the country, which is likely to exceed 170 million tons per annum by the end of XIth five year plan in view of annual growth in the energy demand. From the consideration of insufficient gainful utilization (hardly 42.0%), there is imperative need for the development of an eco-friendly technology-capable of utilizing it on bulk scale on sustainable basis. Subsequently, availability of about 20 million ha of wasteland with its potential to be reclaimed needs the development of an eco-friendly technology. Utilization of fly ash in agriculture as soil modifier or in wasteland/mine spoil reclamation is of considerable interest from the point of view of disposing and exploiting its potential advantages as a source of plant nutrients and also for improving the physico-chemical properties of soil, wasteland and mine spoil. Fly ash contains almost all the nutrients required by plants except nitrogen and humus, which can be supplemented by application of organic matters. Taking a cue from the inherent pozzolanic and important physico-chemical properties of the Indian fly ash such as low bulk density (~1.0 g/cc), high WHC (50-60%), porosity (50-60%), rich in silt sized particles (40-60%), alkaline in nature (pH: 8-10), unburnt C (1-3%, some times even more), negligible solubility and being a source of plant nutrient (P, K, Ca, Mg, S, Cu, Zn, Mn, Fe, B, Mo), extensive R&D work on bulk use of fly ash/pond ash in agro-forestry sector and wasteland/mine spoil management has been carried out at CFRI right from 1984 (when the utilisation of fly ash was almost negligible) till date. The

demonstration trials were carried out from pot experiment to field scale through execution of various projects (16 nos) funded by different organizations of the country. The demonstration trials include different doses of fly ash/pond ash alone and in combination with other amendments such as bio-fertilizer, humic acid, press mud, gypsum, etc. in the vicinity of various TPPs under different agro-climatic conditions and soil types. Based on these, CFRI has successfully developed fly ash soil amendment technology (FASAT), which has established improvement in the fertility status and biological activity of the agricultural/waste land, with promising yield/growth performance of a variety of crops/multipurpose plant species on sustainable basis.

These studies promoted CFRI to successfully translate the useful results of this technology into actual pilot field demonstration trials on both short and long-term basis and also in different farmers' fields and State Adoptive Research Farms (SARF) under different soil types: red and lateritic soil (pH 4.5- 5.5); alluvial soil (pH 7-8); black cotton soil (pH, 8-10); wasteland (pH 7-8); mine spoil ( pH 5.5-7.5) in the close proximity of different TPPs and coalfields not only as a concrete step towards confidence building measures but also to demonstrate the usefulness and success of this soil amendment technology through execution of short-/long-term projects sponsored by different agencies such as NTPC, WBPDC, SSRC, Dept. of Coal, MSEB, MoEF, MCL, SSRC, UPRVUNL, TISCO Jamadoba, etc. (CFRI Reports-1988, 1996, 2000-2004; CFRI Patent 2002; Ram et al, 2000, 2003, 2005, 2006; Srivastava et al, 2002, 2003, 2005; Tripathi et al., 2005).



**Flow chart for utilization of fly ash in agro-forestry**

The advantageous effects of fly ash application as soil amendment and for the reclamation/management of waste/degraded lands/mine spoil/abandoned ash pond through biological means and other amendments have been fully sustained and demonstrated in large scale field trials, which establish the versatility and vast scope of the applicability of FASAT developed at CFRI for rendering the problematic, unproductive soils and mine spoil fertile and more productive, making them suitable for agriculture/forestry purposes that include cultivation of cereal and oil yielding crops, fruit and wood value tree species, medicinal and aromatic plants. This technology is quite efficient in growing various crops with significant improvement in the characteristics of the soil, nutritional value of the crop produce and increase in the yield (20-50 per cent) over control. The demonstration trials carried out include different agro-climatic conditions and soil types together with no adverse effect on the quality of crop produce, soil and surface and ground water bodies especially in respect of uptake/carry over of toxic trace and heavy metals (Pb, Co, Ni, Cr, Cd, As, Se, Mo, etc), thereby providing experimental evidence for dispelling such apprehensions. The various plant/tree species grown on the reclaimed soil have shown encouraging growth performance. Histopathological tests in animals fed with food grains obtained from fly ash amended field after feeding

them over a period of time also did not reveal any toxicological symptoms and rather the produce are continuously being consumed by both human being and animals as well.

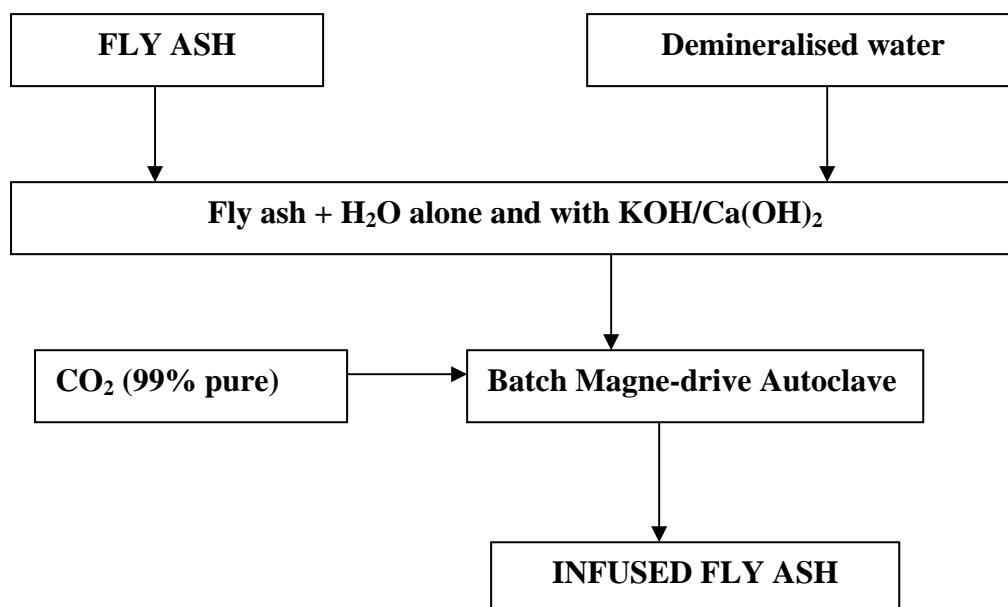
Through the transfer of FASAT and demonstration of successes via large field trials in the farmers' fields in the vicinity of various TPPs in the country, farmers are now more than convinced about various useful effects of fly ash/pond ash application in their agricultural field/waste land for increasing the yields of various crops and improvement in the fertility status of soil. They are coming in larger number to adopt it. However, more extension programs and popularization measures to educate the farmers for making them aware about beneficial use of fly ash/pond ash are still in progress.

### **Carbon Sequestration**

#### *Through Infusion*

The emission of flue gas and fly ash generation in TPPs is of much environmental concern, wherein the former comprises CO<sub>2</sub> as one of the important contributors to the GHGs in the environment and the latter is of equal concern from the consideration of environmental pollution, particularly in respect of contamination of soil, plant, crop produce and surface and ground water bodies due to the presence of many of the toxic trace and heavy metals and radionuclides. Indian coals being derived of drift origin and having higher content of ash (mineral matter) are likely to produce much more quantities of ash as compared to that from coal of in-situ origin, particularly in overseas countries. As such it is crucial to take necessary measures for abatement of GHGs either through development of much more eco-friendly technology or suitable modifications in the existing technology for coal combustion in TPPs. Further, if fly ash generated from TPPs, prior to its disposal on the surface, can be of any use to consume CO<sub>2</sub> -the principal component of flue gas, it will be of worth in mitigating the GHGs. As such, the in-situ infusion of fly ash with CO<sub>2</sub> can be of much help in reducing the CO<sub>2</sub> emission to the environment with accompanied decrease in the leaching of potentially toxic/ heavy metals and radionuclides together with its suitability for gainful utilization in an eco-friendly manner. Keeping these in view, CFRI has developed a process for in-situ infusion of fly ash with CO<sub>2</sub> under ambient conditions: i.e CO<sub>2</sub> introduced into moist fly ash under slightly elevated pressure and at room temperature. The level of infusion achieved is up to 18% of the total mass of the fly ash i.e. almost comparable to the total CO<sub>2</sub> present in the flue gas. The mechanism of this infusion is based on the transformation of basic oxides (Ca, Mg, K oxides) present in fly ash to their respective carbonates. The leachability of the

infused fly ash is reduced particularly in respect of trace and heavy metals. Also the infused fly ash has the potential for application for agro-forestry, which will further contribute to carbon sequestration through photosynthesis (CFRI Report, 2003).



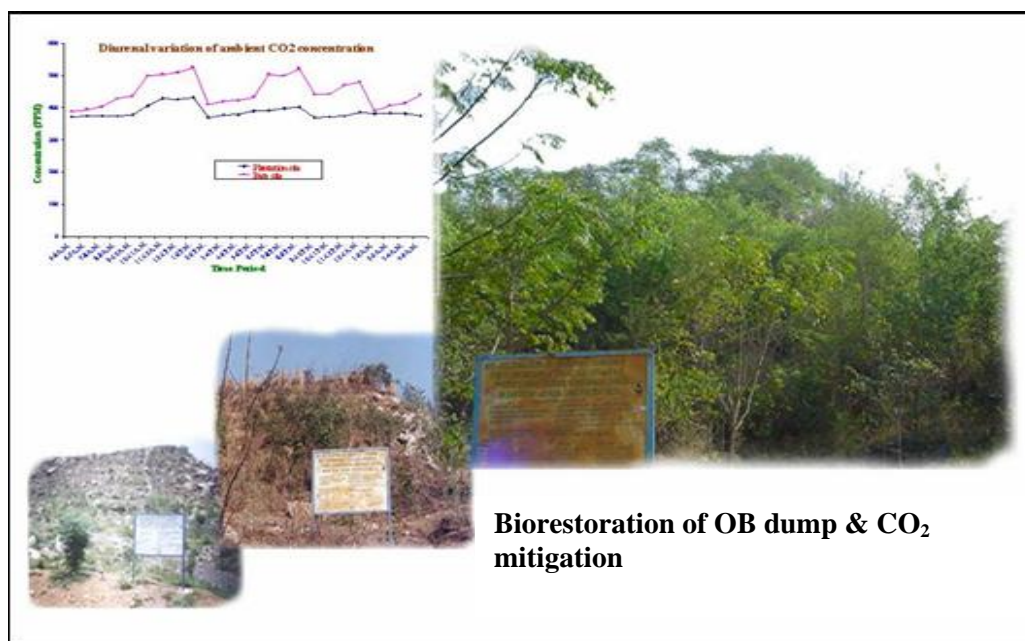
**Flow sheet for mitigation of GHG via in situ infusion of fly ash with CO<sub>2</sub>**

#### *Through plantation*

Afforestation is unique method for reducing CO<sub>2</sub> concentration and other pollutants including particulates and some gases appreciably depending on different stages of plant growth and seasons. From these contemplations, the present study was carried out by observing the role of different forest covers (manmade), as a sink of CO<sub>2</sub> in Indian context, through the determination of the rate of photosynthesis and other physiological parameters of predominant plant species (*Acacia auriculaeformis*, *Albizzia lebbek* and *Dalbergia sissoo*) common to different plantation sites of different age groups made in the mine spoil/OB dump/waste land of MCL, Orissa, and accompanied reduction in the air pollutants, like particulates, SO<sub>x</sub>, NO<sub>x</sub>. The concentration of various pollutants including CO<sub>2</sub> was well above adequate level at the bare site. Of these predominant species, *Acacia auriculaeformis* evinced relatively better photo-synthetic activity, transpiration rate, stomatal conductance and leaf area than *Albizzia lebbek* and *Dalbergia sissoo*. Also increase in the values of these parameters with the growing stages of the plant species was noticed. The predominant species were quite effective in reducing these pollutants



depending on their inherent ability and growth stage that make them suitable to grow under specified edaphic conditions and seasonal variation, where the concentration of these pollutants including CO<sub>2</sub> was minimum in the rainy season. As such potential plant species playing major role in the CO<sub>2</sub> sink and reducing SO<sub>x</sub>, NO<sub>x</sub> and particulates emission can be planted in the vicinity of GHGs emitting sources like TPPs and coal mining areas affected by coal extraction in the form of huge generation of mine spoil/OB dumps in order to mitigate the CO<sub>2</sub> and other gases including particulates from the atmosphere in an eco-friendly manner and make the environment healthy. Also the forest land already degraded by anthropogenic stresses can be managed in terms of plantation of such efficient photosynthetic species leading to conserve the biodiversity as well as GHG mitigation (CFRI Report, 2005).



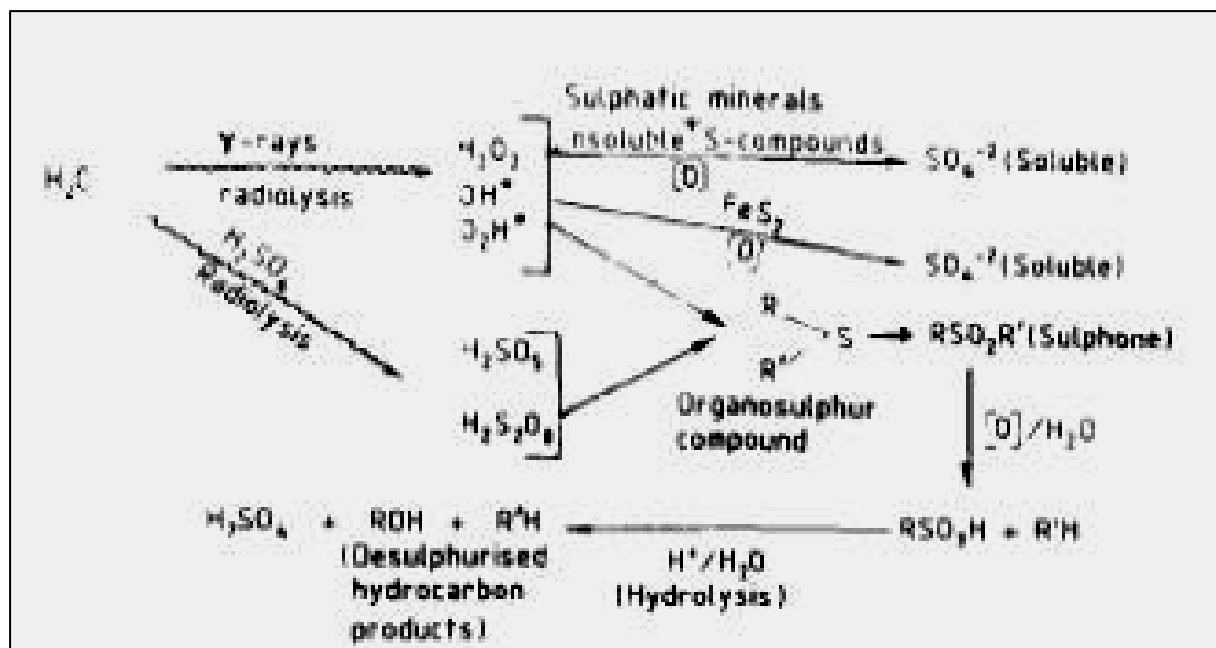
## Desulphurisation

### Coal

The harsh pollution caused by sulphur in coal during its combustion is considered to be responsible for acid rain and several other harmful effects on the environment. The necessity for gainful utilization of high-S coals has galvanized coal researchers world-wide to focus their attention on finding effective, economic and technologically viable methods of desulphurization. India possesses over 2818 Mt of high-S but otherwise good-quality (some

highly caking) low-ash coals', for which the removal of sulphur to below 1 wt% is crucial, so as to make them suitable for direct combustion in an eco-friendly manner and also for blending purposes. Numerous methods of desulphurising coal before combustion are being tried with varying degrees of success and are at different stages of development. While most of these methods are effective for almost complete removal of inorganic (pyritic and sulphate) sulphur, they are less effective in removing the organic sulphur without affecting or degrading the coal structure and also without loss in heating value. As such there is an imperative need to develop an effective selective method for desulphurisation of coal which is quite capable of removing both inorganic and organic forms of sulphur in a single step without affecting the coal matrix and its heat value.

From these in consideration, a novel radiolytic method utilizing the radiolysis of water and sulphuric acid by gamma rays has been developed by CFRI where the generation of  $H_2O_2$  and other exited molecules/free radical formation takes place and this causes the oxidation of S-compounds particularly pyrites and sulphate-S to their soluble sulphates and organic-S compounds to their oxides i.e. sulphones which on further hydrolysis undergo the formation of alcohols, water and sulphuric acid. The technology thus developed is simple yet effective method of simultaneous desulphurisation and demineralisation of high-S coals. Both the inorganic and organic forms of sulphur are removed from the coal without appreciably affecting the coal matrix or causing any substantial loss in combustible matter and rather with improvement in the heat value. The method is technologically feasible and has been worked out d to be economical. The potential application in hydraulic transportation of coal slurry through pipelines is thus promising. The removal of S and mineral matter from coal takes place through selective oxidation by the radiolytic products generated in situ (Ram et al., 1992; Tripathi et al., 1998).



Reaction mechanism of radiolytic desulphurization and demineralization of high-S coal

### Diesel

The S compounds in petroleum fuel are unwanted from the considerations of refinery processes, emission control and the quality of liquid products. In particular, the low-S has its tremendous utilization potential in the combustion of fuel and diesel, driven transport vehicles and other combustion engines, where it can be burnt in an eco-friendly manner with minimum emission of particulates and acid rain contributing  $SO_x$  into the atmosphere, besides the formation of gummy deposits responsible clogging the filter of fuel handling system of automobiles and other engines. Also they pose corrosion problems in the part of combustion engines, reduction in the activity of catalytic converters, deactivation tendency towards catalyst in down stream processes and up gradation of hydrocarbons. As per clean air Act, the S content in diesel is to be reduced to the level of 0.05 wt % in our country. The current commercially available hydro-desulphurisation process for diesel is catalytic and involves hydrogen at high temperature and pressure, apart from its being expensive. Taking these in view, a novel process for desulphurisation of diesel by oxidation and solvent extraction under ambient conditions has been used, where the concept of S compounds present in diesel along with the organo-sulphones, formed as a consequence of oxidation, having more polarity than that of S atoms, followed by their extraction in the non-polar solvent, has been taken into the consideration. This process is quite effective

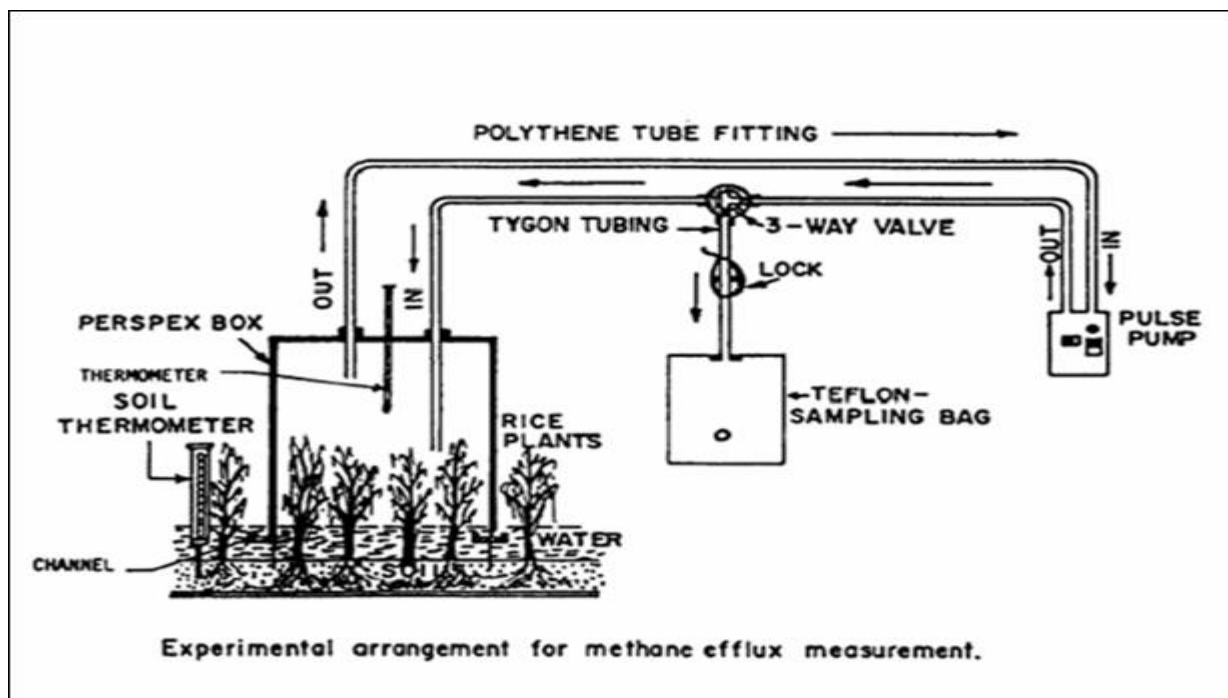
in removing the S to the tune of 0.05 wt % without affecting and rather improving the characteristics and yield of the desulphurised diesel in a cost effective and eco-friendly manner (CFRI Patent, 2000).

### **Bio-solubilisation of lignite to humic acid**

Six fungal stains (*Aspergillus fumigatus*-two strains, *Fusarium udum*, *Fusarium solani*, *Aspergillus oryzae*, *Aspergillus sydowii*) and two bacterial strains (*Baccillus species* & *Baccillus licheniformis*) were identified and found suitable for lignite solubilisation. The maximum solubilisation of lignite was up to 26% in 2- 3 weeks fermentation/incubation period. Qualitatively the obtained humic acid was quite comparable with the standard humic acid and rather superior to chemically synthesized one from nutritional point of view, apart from relatively less content of trace/heavy metals, negligible PAH value and effectiveness in increasing the yield of crops grown in comparison to chemically extracted humic acid together with better soil fertility on sustainable basis (CFRI Report, 2003).

### **Monitoring of methane emission from paddy fields of India**

In 1990s the emission of methane from paddy fields particularly in Asia including India was apprehended as major contributor to global methane emission budget. In order to have a more reliable methane budget estimate from Indian paddy fields, the 1991 methane campaign (MC91) was organized. It involved more than 15 organizations, including various laboratories of the Council of Scientific & Industrial Research (CSIR), Institutes of Indian Council of Agricultural Research (ICAR), and a number of universities. The measurements covered the various rice ecosystems in the major rice-growing regions during wet season from June to November 1991. CFRI was actively involved in the team of this network project to measure the methane budgets from the paddy field of the then Bihar. The methane budget from Indian paddies has been estimated to be around  $4.0 \text{ TgY}^{-1}$  with a range between 2.7 to  $5.4 \text{ TgY}^{-1}$ . This value is an order of magnitude low as reported earlier by IPCC, and the consequence of these low values is that the global CI-L emissions from paddies are probably lower than the mean value of  $60 \text{ TgY}^{-1}$  used in the IPCC 1992 update (Parashar et al., 1996)



### **Other activities in the area of water and air pollution**

Besides above, the activities (previous and ongoing) include: Quantification of Respirable particulate matter in the ambient air of the residential/commercial area of coal based and other industries; Meteorological data generation; Determination of  $\alpha$ -quartz in respirable air borne dust (Testing Analysis job) in coal mining areas; Measurement of emission of GHGs and SPM; Monitoring atmospheric biotic/abiotic particulates and preparing calendar for them including the associated health problems, etc. (Bharathi et al., 2004, 2005, 2006) Other activities include development of National Environmental Standard for suspended particulate matter in stack emission of beehive coke oven. The various projects (completed/ongoing) funded by different agencies are as follows:

- Traffic & Emission Survey in Jharia Coal Field Area (Spon: Norwest Mine Services, Canada, 1996).
- Monitoring of Emissions from Coal Mines under Fire at Different Levels of Jharia Coalfield (Spon: GAI-MET CAEM, Canada, 1996).
- Development of National Environmental Standard for Coal Washeries in respect of Air, Water and Noise Level (Spon: CPCB, New Delhi, 1995-96).
- Quantification of Respirable Particulate Matter in the Ambient Air of the Residential/Commercial Area of BCCL (Spon: BCCL, Dhanbad, 1996).

- Assessment of Stack Emission and Ambient Air in the Kolaghat Thermal Power Plant, West Bengal (Spon: WBPDCCL, 1996).
- Environmental Data Generation for Kunderkocha Gold Mine (Spon: MIN MEC, New Delhi, 1996).
- Studies on Power Generation Potential through Waste Heat Recovery from Existing Non-Recovering Coke Ovens in and Around Dhanbad Area (Spon: Min. of Power, GOI).
- Sampling and Characterization of Ambient Air and Stack Emission of Coke Ovens (Spon: BCCL, Dhanbad).
- Rapid And Comprehensive Environmental Impact Assessment Studies for Integrated Hazardous Waste Management Facilities at The Industrial Sites of TALOJA, Tarapur Mumbai (Spon: MIDC, Mumbai.)
- Meteorological Data Generation at IOC, Barouni (Spon: POLMET, New Delhi).
- Determination of  $\alpha$ -quartz in Respirable Air Borne Dust – Testing Analysis (Spon: different organizations).
- Studies on the Measurement of Emission of GHGs (SO<sub>x</sub>, NO<sub>x</sub>, CO<sub>2</sub>, CO) and Aerosols from TPPs, Steel & Cement Plants and N<sub>2</sub>O and CH<sub>4</sub> from Rice/wheat Fields and their Mitigation.
- Studies on the Impact of Atmospheric Biotic/Abiotic Particulates on the Environment of Jharia Coalfields and their Abatement Strategies (Spon: SSRC Dept of Coal).
- CSIR Task Force on: Pollution Monitoring Mitigation Systems and Devices (on going projects)

Programme: Development of state-of-art sensors for pollution and receptor monitoring

*Activity (1.5):* Insect as Pollutant-specific bio-sensors for coal mining and processing industries (Washaries, coke oven plants) and Thermal power plants

*Activity (1.6):* Pollutant specific bio-aerosol sensors for open cast coalmines and thermal power plant environments. (SMM-05)

### **Impact of the Significant Studies**

The various impacts of the studies made are encapsulated as below:

- i. The FASAT developed by CFRI has its tremendous economic, environmental and societal impact. In that FA/PA if applied in wasteland @ 100 tons/ha, 20 million ha of waste/degraded land available in our country and having the potential for its reclamation, is sufficient for consuming total



ash generation for 20 yrs. The availability of productive land (being used for ash dump) for agriculture together with increase in yield (20-50%) of different crops grown in the amended soils will contribute to enhance financial output. It will lead to waste utilization, mitigation of several environment related problems (air, soil, water pollution), reclamation of waste/degraded lands/mine spoil, apart from sustainable growth of crops on FA/PA amended soils i.e. with no carry over/uptake of toxic trace/heavy metals beyond permissible limit. Apart from this benefit to the farmers through increased crop yields and improvement in their normal life style, Nutrition-rich food to the users and opening of new avenues for employment generation during different stages of fly ash/pond ash amendment. The various beneficiaries are TPPs, NTPC, SEBs, CPCB, SPCBs, MoEF, Min.of Agri., Farmers, etc.

- ii. The findings on in situ infusion of fly ash with CO<sub>2</sub>, if adopted commercially, may go a long way in mitigating the CO<sub>2</sub> emission from TPPs. Infused FA can be potentially utilized for agro-forestry leading to further C- sequestration in the soil through photosynthesis. The reclamation of waste land, mine spoil/OB dumps, abandoned ash pond through biological amendments for agro-forestry, another means for mitigation of GHG, is also of much socio-economic impact.
- iii. The radiolytic process for desulphurisation of high S coals is quite capable of removing both inorganic and organic forms of S to a significant extent without affecting the coal matrix with enhanced calorific value is of tremendous environmental impact. The process developed for the desulphurisation of diesel by oxidation and solvent extraction has the potential to replace conventional process of Hydro-desulphurisation to a major extent and is cost effective and eco-friendly.
- iv. The study on monitoring of the emission of methane from paddy fields particularly in Asia including India, which was apprehended as major contributor to global methane emission budget by some of the dominating countries, in fact, has been totally dispelled. As such the continuation of the paddy crop is rather a big contribution to the economy of our country, apart from meeting the ever-growing threat of fast growing population
- v. The activity on the aspect of impact of atmospheric biotic/abiotic particulates on the environment of Jharia coalfields and their abatement strategies is totally virgin area and many of the people residing/working in

the surroundings of coalfields of Jharia suffer from various ailments due to these particulates. The findings of this study will lead to the identification of various particulates, exploring their impact on human health and finding the remedial measures for treatment, thereby making the study of great socio-environmental impact.

### **Future Studies**

CFRI has proposed to carry out R & D work in the following different areas in next five years under XIth five year plan, which include the up-scaling/popularization of the technologies/processes developed and many new thrust areas projects in the area of environmental management submitted for their funding by different agencies such as UPRVUNL, NMBA, DST, NTPC, BCCL, CSIR Network, ICAR, etc, cost of the submitted project proposal being Rs 35 crore. These are:

1. Popularisation of agro-forestry application of fly ash/pond ash amongst the farmers in the vicinity of different TPPs and other parts of the country
2. Bio-reclamation of waste/degraded land and mine spoil through selective plantation vis-à-vis carbon sequestration
3. Photo-biological  $H_2$  production from bio-depolymerized lignite/agro-residues
4. Biotechnological conversion of lignite to methane
5. Evaluation of the potentials of coal mine water for its use as irrigation water and nutrient
6. Phyto-extraction of heavy metals from contaminated soils in mining/coal based industrial area through rhizosphere modification
7. Development of soil quality/ health index
8. Impact of different industries & identification of soil indicators, detecting baseline & target values for optimum soil/ ecological functioning
9. Impact of coal mining on microbial biodiversity of higher edible fungus (endemic to coal field) and its conservation strategies
10. Pilot scale development of the process for mitigation of GHG through in-situ infusion of fly ash with  $CO_2$
11. Heavy metal sequestration as an in situ technology using VAM fungi for coal washery effluent contaminated site/ mine spoil of Jharia coalfield
12.  $H_2$  production through microbial photosynthesis
13. Atmospheric corrosion studies over polluted environment of coal based industrial areas



14. Atmospheric bio-pollution and its impact on human health in and around coalfield/coal based industrial areas
15. Myco-remediation of xenobiotic substances/ bound residues/ PAH in coalmine soil by ligninolytic enzymes of white rot fungi
16. Production of *Fly ash-Humic acid-Bt toxins-Copper (FA-HA-Bt-Cu) Nanocomposites* for inbuilt pest/plant pathogen
17. Reclamation of mine water for the development of potable water
18. Assessment of status of fire in X seams of East Bhagatdih colliery, BCCL
19. Jatropha plantation in the low lying ash filled area of TISCO
20. Response of soil microbial activity and microbial community structure to fly ashes under laboratory and field conditions

## ANNEXURE

### 1. List of Publications

- Bharti A.K., Deepali Rathore, N.K. Srivastava, L.C. Ram, R.C. Tripathi, S.K. Gupta, P.S.M. Tripathi and G. Singh (2003), Eco-restoration of over burden dumps through the plantation of high photosynthetic/soil binder species presented in National Seminar Coal S & T Vision 2020 held at CFRI, Dhanbad on 20-21 April, 2003
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### **3. List of Patents**

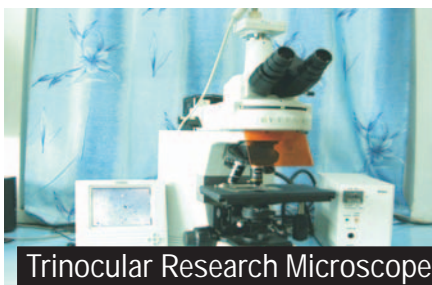
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- A synergistic fly ash based soil conditioner cum fertiliser composition, G. Singh, L. C. Ram, S. K. Jha, R. C. Tripathi, N. K. Srivastava, Patent No. 031 NF 2002.
- Mitigation of Green House Gases via in situ Infusion of Fly Ash with CO<sub>2</sub> in Thermal power Plants by Lal Chand Ram, Gulab Singh, Awadesh Kumar Sinha and Ramesh Chandra Tripathi (applied in PCT, USA and India).



*Key facilities available...i*



DMRXP HC



Trinocular Research Microscope



HPLC System



ASS



XRF



UV - Vis Spectrometer



Surface Area Analyzer



GC - MS



TPD / TPR





Coal Preparation Pilot Plant



Spiral



Modular Continuous Reactor System



FETF



High Pressure Tabular Reactor for FTS



Process Development Unit (PDU)



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