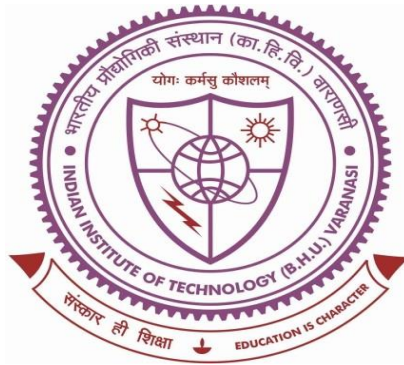


# CHEMICAL CHARACTERIZATION AND BENEFICIATION OF PERMIAN COAL FROM SINGRAULI COALFIELD, INDIA



Thesis submitted in partial fulfillment for  
the Award of Degree

**Doctor of Philosophy**

By

**Ankur Dwivedi**

DEPARTMENT OF MINING ENGINEERING  
INDIAN INSTITUTE OF TECHNOLOGY  
(BANARAS HINDU UNIVERSITY)  
VARANASI – 221005  
INDIA

Roll No. : 15151501

2023

# CERTIFICATE

---

---

This is to certify that the revised thesis entitled “**Chemical Characterization and Beneficiation of Permian coal from Singrauli coalfield, India**” is being submitted by **Mr. Ankur Dwivedi** in partial fulfillment for the award of Ph.D. in **Department of Mining Engineering, IIT (BHU), Varanasi** is a record of bonafide work carried out by him.

Date of Submission: 25.07.2023



(Supervisor)

Forwarded by:



# CERTIFICATE

---

---

It is certified that the work contained in the thesis titled “**Chemical Characterization and Beneficiation of Permian Coal from Singrauli coalfield, India**” by Ankur Dwivedi has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

It is further certified that the student has fulfilled all the requirements of Comprehensive, Candidacy and SOTA.



**(Amrendra Kumar)**

Supervisor and Associate Professor

Department of Mining Engineering

Indian Institute of Technology (BHU), Varanasi

# DECLARATION BY THE CANDIDATE

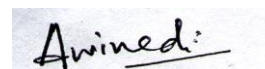
---

---

I, Ankur Dwivedi certify that the work embodied in this thesis is my own bonafide work and carried out by me under the supervision of Dr. Amrendra Kumar, at the Department of Mining Engineering, Indian Institute of Technology (BHU), Varanasi. The matter embodied in this thesis has not been submitted for the award of any other degree/diploma.

I declare that I have faithfully acknowledged and given credits to the research workers wherever their works have been cited in my work in this thesis. I further declare that I have not willfully lifted up any other's work, paragraphs, text, data, results, etc., reported in journals, books, magazines, reports dissertations, theses, etc., or available at websites and included them in this thesis and cited as my own work.

Date: 22.07.2022



Place: Varanasi

(Ankur Dwivedi)

---

## CERTIFICATE BY THE SUPERVISOR

It is certified that the above statement made by the student is correct to the best of my/our knowledge.



**Supervisor**

(Amrendra Kumar)

Associate Professor

Department of Mining Engineering

Indian Institute of Technology

(Banaras Hindu University)

Varanasi -221005, India



**Head of Department**

(Professor Suprakash Gupta)

# COPYRIGHT TRANSFER CERTIFICATE

---

---

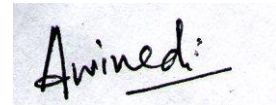
**Title of the Thesis:** Chemical characterization and beneficiation of Permian coal from Singrauli coalfield, India.

**Name of the Student:** Ankur Dwivedi

## Copyright Transfer

The undersigned hereby assigns to the Indian Institute of Technology (Banaras Hindu University), Varanasi all rights under copyright that may exist in and for the above thesis submitted for the award of the DOCTOR OF PHILOSOPHY.

Date: 22.07.2022



Place: Varanasi

(Ankur Dwivedi)

**Note:** However, the author may reproduce or authorize others to reproduce material extracted verbatim from the thesis or derivative of the thesis for author's personal use provided that the source and the Institute's copyright notice

## ACKNOWLEDGMENTS

---

---

I have great pleasure in expressing my deepest sense of gratitude to my revered supervisor, Dr. Amrendra Kumar, Associate Professor, Department of Mining Engineering, IIT (BHU), Varanasi for inspiring guidance and encouragement through the course of this study.

From time to time, I am grateful to research progress evaluation committee members, Professor Aarif Jamal and Professor Shyam Bihari Dwivedi, for their encouragement and interest in my work. I am also very thankful to Dr. Rajesh Rai, Dr. Ashok Jaiswal, and Dr. RP Singh for their encouragement and interest shown in my work from time to time.

My heartfelt gratitude also goes to Professor Prakash Kumar Singh, Professor Amia Shankar Naik, Dr Sanjay Kumar Tiwari, and other esteemed faculty members at Banaras Hindu University's Department of Geology.

The undersigned places on record the cooperation of Sri BN Pal, I B Pal, and Sunil Mishra, Pyarelal, and my colleagues in the department for their valuable help at various stages during the completion of this work.

I am also grateful to the other faculty members of IIT BHU's Department of Mining Engineering for their immediate encouragement in completing this work. I am thankful for the cooperation of my colleagues in the department.

The undersigned is equally grateful to the officers of Northern Coalfields Limited, Singrauli, for providing necessary facilities during the fieldwork. The financial help given by the Mining Engineering Department in this context is greatly acknowledged. Special thanks go to Dr. Arpita Dwivedi, a research scholar in the Physics Department at Banaras Hindu University. Last but not least, the blessings of my parents, wife, and elders led to the completion of this work.

Date: 22.07.2022

Ankur Dwivedi

Dedicated in the honour of late Professor M. P. Singh, a renowned international  
coal scientist



## LIST OF FIGURES

---

---

Figure 3.1. Location map of Singrauli district and drainage pattern showing the parts of coalfields (Moher sub-basin region).....	33
Figure 3.2. Regional geological map of the Singrauli coalfield, showing the part of Moher sub-basin in the northeast portion (Mukhopadhyay and Bhattacharyya, 2019) .....	36
Figure 3.3.The geological map of coal hosting the Moher sub-basin (part of the Gondwana basin), Singrauli, India (Singh et al., 2017) .....	37
Figure 3.4. Productive coal mines (Bina, Kakri, Krishnshila, Dudhichua, and Jayant) of Northeastern Coalfields Limited (NCL) (Shirin et al., 2021).....	40
Figure 3.5.Vertical profile of working face of Dudhichua Mine showing exposure of Turra seam.....	40
Figure 3.6. Generalized sequences of coal bearing Barakar Formation (Mahapatra, 2016).....	41
Figure 5.1. TGA profile of six different coal samples as (a) Coal 1, (b) coal 2, (c) coal 3, (d) coal 4, (e) coal 5, and (f) coal 6 .....	60
Figure 5.2. DTG profile of different coal samples (a-f) as (a) Coal 1, (b) Coal 2, (c) Coal 3, (d) Coal 4,(e) Coal 5,and (f) Coal 6 respectively .....	64
Figure 5.3. Qualitative XRD analysis of coal samples showing the presence of mineral phases (Q:Quartz; K:Kaolinite; M:Muscovite; O:Orthoclase; H: Hematite; Si:Siderite; G: Gypsum; P: Pyrite; S:Sulphur; J:Jarosite; and Cl: Chlorite). .....	67
Figure 5.4. SEM image showing the presence of coal macerals along with mineral particles .....	70
Figure 5.5. Energy-dispersive X-ray spectroscopy (EDX) graph of elements associated with the Indian Permian coal from Singrauli coalfield, India. The survey detected the presence of carbon, oxygen, silicon, and aluminum as significant constituents.....	71
Figure 5.6 Energy-dispersive X-ray spectroscopy (EDX) graph of elements associated with the Indian Permian coal from Singrauli coalfield, India .....	71
Figure 5.7. FTIR spectra of the different coal samples .....	74
Figure 5.8. XPS wide energy spectrum for the surface of IPC 1 coal sample .....	82

Figure 5.9. XPS wide energy spectrum for the surface of IPC 2 coal sample .....82

Figure 5.10. XPS spectra of coal sample IPC1 for (a) C1s peak, (b) O 1s peak, (c) N 1s peak, (d) Al 2p peak, and (e) Si 2p peak, black solid line and red solid line refer to experimental curves and best-fitted curves, and the assignment of peak is also given after deconvolution with a solid line of different color..... 86

Figure 5.11. XPS spectra of coal sample IPC 2 for (a) C1s peak, (b) O 1s peak, (c) N 1s peak, (d) Al 2p peak, and (e) Si 2p peak, black solid line and red solid line refer to experimental curves and best-fitted curves, and the assignment of peak is also given after deconvolution with a solid line of different color..... 87

Figure 5.12. Raman spectra of coal 1-4 for different band ..... 89

Figure 5.13 (a-d). Raman spectrum of four different samples coal with the corresponding curve fitted bands (red dotted represents experimental Raman spectra; black line fitting spectra, different color line represent area under D1, D2, D3, D4, and G).....92

Figure 5.14. SEM image showing agglomerated glass, cenospheres and nanosphere.95

Figure 5.15. Energy-dispersive X-ray spectroscopy (EDX) graph of elements associated with the Permian fly ash. The survey detected the presence of carbon, oxygen, silicon, and aluminum as significant constituents .....97

Figure 6. 1 (a-f). FTIR spectra of (a) Raw CS-1 and DES-treated CS-1, (b) Raw CS-2 and DES-treated CS-2, (c) Raw CS-3 and DES-treated CS-3, (d) Raw CS-4 and DES-treated CS-4, (e) Raw CS-5 and DES -treated CS-5, and (f) Raw CS-6 and DES-treated CS-6, respectively .....113



## LIST OF TABLES

---

---

Table 1.1 Total Coal Production and Consumption by Country in 2021 (IEA) .....	5
Table 1.2 Gradation of different type of coal in India.....	6
Table 1.3 India's leading coal producer states.....	8
Table 1.4 Different Categories of Coal Reserve Distribution (MT) in India .....	8
Table 3.1 Generalized geological sequences developed in the Singrauli basin.....	37
Table 3.2 Geological age and lithology of coal bearing formations of India.....	38
Table 5.1 Proximate, ultimate analysis, and GCV of coal in the singrauli .....	56
Table 5.2 Burning profile parameters and combustion rate of different coal.....	61
Table 5.3 Estimated value of Conversion degree ( $\alpha$ ), Activation energy ( $E_g$ ) and correlation coefficient .....	65
Table 5.4 Characteristic peaks and respective molecular vibrations.....	73
Table 5.5 Peak area of characteristic transmittance peaks of different coal samples..	74
Table 5.6 Proximate, ultimate and gross calorific value (GCV) of IPC from the Singrauli coalfield.....	81
Table 5.7 Contents of atomic carbon, atomic oxygen, atomic silica, atomic aluminum, atomic nitrogen, and atomic sulfur on the coal surface .....	83
Table 5.8 XPS data of C 1s spectra of Indian Permian coal samples .....	84
Table 5.9 XPS data of O 1s spectra of Indian Permian coal samples .....	85
Table 5.10 XPS data of N 1s spectra of Indian Permian coal.....	85
Table 5.11 Raman structural parameters determined from the fitting of Raman spectrum.....	92
Table 5.12 The concentration of major oxides (wt%) of PFA from the Singrauli coalfield.....	99
Table 6.1. Structures of the DES used for coal treatment.....	106
Table 6.2 Swelling ratios and extraction yields of different coals samples.....	109
Table 6.3 Proximate analysis of raw and DES-treated coal wt% (ad).....	110
Table 6.4 Ultimate analysis of raw coal and DES treated coal , wt%(daf) .....	111
Table 6.5 FTIR spectra of characteristic vibrations and functional groups present in coal.....	112
Table 6.6 Peak area of characteristic transmittance peaks of different coal samples	114
Table 7.1 Grades of non-coking coals in the Singrauli coalfield .....	129
Table 7.2 Grades for Coking Coal in the Singrauli coalfield.....	130
Table 7.3 Grades of Semi-coking and weakly Coking Coal in the Singrauli .....	130



## SYMBOLS AND ABBREVIATIONS

---

NCL	:	Northern Coalfield Limited
NTPC	:	National Thermal Power Corporation
AMD	:	Acid Mine Drainage
CFA	:	Coal Fly Ash
FA	:	Fly Ash
WCA	:	World Coal Association
NCL	:	Northern Coalfields Limited
CIL	:	Coal India Limited
CCT	:	Clean coal technology
ROM	:	Run-of-mine
SEM	:	Scanning electron microscopy
XRD	:	X-ray Diffraction
TEM	:	Transmission electron microscopy
FTIR	:	Fourier transform infrared spectroscopy
XPS	:	X-ray photoelectron spectroscopy
EDS	:	Energy dispersive spectroscopy
IEA	:	International Energy Agency
ASTM	:	American Society for Testing and Materials
GDP	:	Gross domestic product
TG <sub>spi</sub>	:	Thermogravimetric index (% °C <sup>-1</sup> min <sup>-1</sup> )
TGA	:	Thermogravimetric analysis
TG	:	Thermogravimetry
DSC	:	Differential Scanning Calorimetry
DTG	:	Derivative thermogravimetry
wt %	:	Weight percent
ad	:	Air dried basis
daf	:	Dry ash free basis

GSI	:	Geological Survey of India
HI	:	Hydrogen Index
WCA	:	World Coal Association
FC	:	Fix Carbon
VM	:	Volatile Matter
BT	:	Billion Tonnes
MT	:	Million Tonnes
DES	:	Deep Eutectic Solvents
GCV	:	Gross Calorific Value
$T$	:	Temperature ( $^{\circ}\text{C}$ )
$T_0$	:	The starting temperature value of the thermo-analytical measurement
HR	:	Heating rate
FR	:	Fuel ratio
$T$	:	Time (min)
C	:	Carbon
H	:	Hydrogen
N	:	Nitrogen
O	:	Oxygen
S	:	Sulfur
Hg	:	Mercury
U	:	Uranium
Th	:	Thorium
As	:	Arsenic
Zn	:	Zinc
Mg	:	Magnesium
CO <sub>2</sub>	:	Carbon dioxide
CO	:	Carbon monoxide
CH <sub>4</sub>	:	Methane

H <sub>2</sub> O	:	Water
km	:	Kilometer
m	:	Meter
cm	:	Centimeter
%	:	Percentage
°C	:	Degree Celsius
μm	:	Micro meter
km <sup>2</sup>	:	Square kilometer



## PREFACE

---

Coal in Singrauli occurs in different basins. The qualitative and quantitative studies of these coals have been insufficient to understand the nature and type of coal. Five coal fields, namely Bina, Kakri, Krishnshila, and Jayant Dudhichua, have productive open-cast mines and have been selected for the study. In the present work, the main emphasis has been laid on the chemical and mineralogical aspects. The proximate analysis involves the study of moisture, ash, VM, and fixed carbon, and the ultimate analysis measures the elemental composition of coal. In addition to X-ray diffraction and Fourier Transmission Infrared Radiation, XPS, SEM and Raman spectroscopy were used, and an attempt was made to determine the quantitative distribution of each organic and inorganic constituent of coal. Finally, the present study aims to classify the Singrauli coal according to available Indian classification schemes and to focus on the chemical beneficiation and potential utilization of this coal and derived fly ash with less harm to the environment. The entire work has been divided into nine chapters.

**Chapter 1** Provides an introduction of the available literature on the Gondwana coal with a description of the other coals in India.

**Chapter 2** deals with the exhaustive literature review of past studied in chemical quality of coal and its chemical beneficiation.

**Chapter 3** The geological setting and geography has been presented in this section with the description of the mode of occurrence of coal seams in Singrauli Basins.

**Chapter 4** includes the detail of sample collection procedure and various methodologies adopted to study coal and fly ash.

**Chapter 5** The Experimental study and results of analyzed data have been presented in this section.

**Chapter 6** gives an insight into the beneficiation of coal using choline, chloride, and urea-based deep eutectic solvent.

**Chapter 7** the discussion of the entire analytical results has been presented in this chapter.

**Chapter 8** reports the conclusion based on the research work. The entire work has been summarized. Finally, some recommendations for future work have also been incorporated in this chapter of the thesis.