CHAPTER 3

FIELDWORK AND METHODOLOGY

3.1 Study Area

Northern Coalfields Limited (NCL), a subsidiary company of Coal India Limited (CIL) is operating Opencast Mines in Singrauli coalfield. The Singrauli Coalfield is divided into two basins namely Main basin and Moher sub-basin. Moher sub-basin having an area of 312 Km². Out of which 80 Km² on Eastern side lies in Sonebhadra District of Uttar Pradesh and rest in Singrauli District of Madhya Pradesh. The Main basin is located west of Waidhan. The Moher sub basin is the center of mining activities. The southern half of the Moher basin is preserved while the northern half was up thrown by a metamorphic fault and is eroded, hence called Moher Sub basin.

The coalfield is divided into 10 mining projects i.e., Kakri, Bina, Khadia, Dudhichua, Jayant, Nigahi, Amlohri, Gorbi block 'B', and Gorbi mine (abandoned). A location map of Singrauli is given in the **Fig. 3.1** and mine blocks of Singrauli Coalfield in **Fig. 3.2**.

Singrauli Coalfield is spread across the districts of Singrauli and Sonebhadra in the Indian States of Madhya Pradesh and Uttar Pradesh, mostly in the basin of the Son river. The Singrauli Coalfield is located between latitudes 24⁰12' N and 23⁰47' N. It is spread over nearly 2,200 km² but only a small part of the coalfield, around 220 km^2, has been identified as promising by the Geological Survey of India. The north-eastern part of the coalfield sits on a plateau with an altitude of 500 m above mean sea level, well above the lower plains of 280 m altitude. Northern Coalfields Limited was carved out of Central Coalfields Limited in 1986, to take care of operations in the Singrauli Coalfield. The proved reserves in the Moher basin are about 3 billion tonnes out of which 2.3 billion tonnes have already

been planned for mining, in an area of about 84 km². The method and methodology are shown in the flow chart (**Fig. 3.3**).

3.1.1 Topography

The Singrauli coalfield stands as high plateau over the surrounding planes covered by Talchir sediment. The Barakar sediment opencast over the Talchir outcrops as scrap faces. Towards North, the Gondwana sediment abuts against Precambrian rocks, which from a series of East-West trending prominent ridge. The Singrauli plateau rises to a height of over 500 m and above mean sea level, (M.S.L.) from the Southern plane, which has a general level of about 275 m from above M.S.L. The plateau, which is referred to as Mohar plateau, has step-like scrap faces towards south. The steps appear to represent different stages of pen planation. The different platforms are of remnant type, resulting from erosion of gently inclined sedimentary strata of varying resistance. Physiographically, the eastern part of the coalfield in Uttar Pradesh, is characterized by a cluster of hills and plateau to the north and undulating plains to the south. The western part of the coalfield also comprises a platform with a slope towards east and south, where there are abrupt drops of 50 to 60 m in altitude. Towards southwest, several high peaks such as Popari (508m) and Burma (564m) are prominent topographic features.

3.1.2 Climate, flora and fauna

The coalfield experiences a tropical monsoon type climate. Though, the winters are rather cold (5-10⁰C), the summer temperature often shoots upto 46⁰c in the day time. The area enjoys a rather heavy monsoon and the annual rainfall varies between 125 cm and 150 cm. The wild animals are depleting and many have migrated to more remote areas due to mining and allied activities. The ground especially the elevated areas, is mostly covered with open

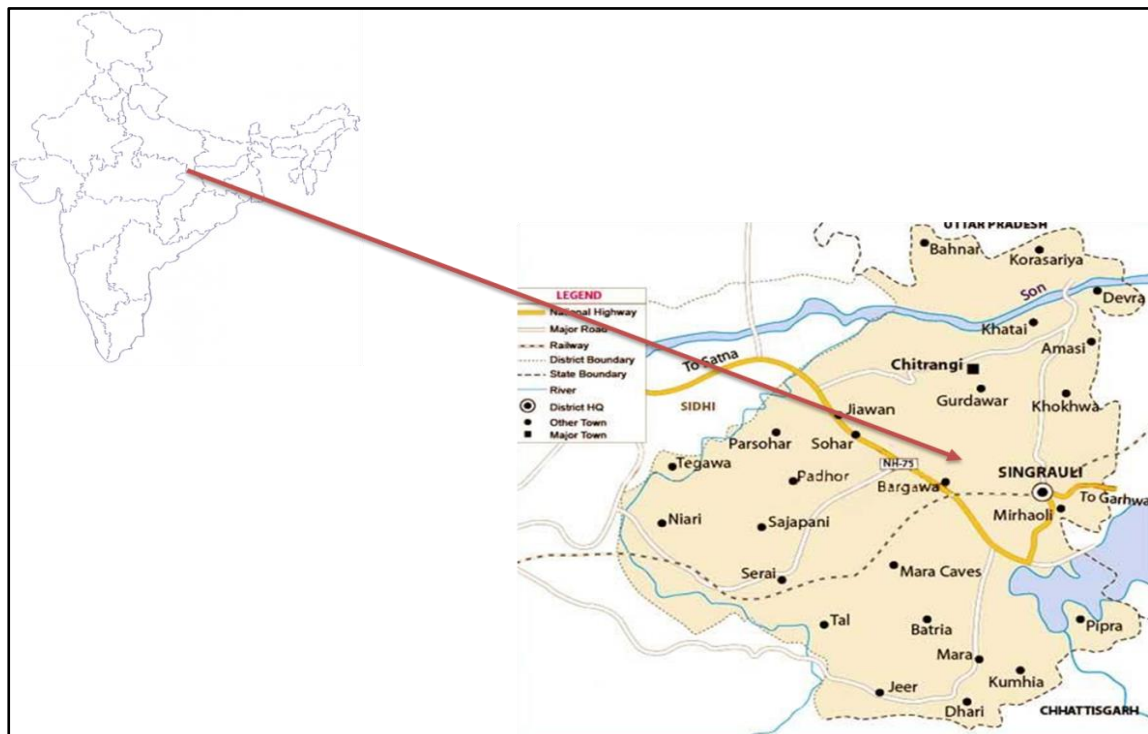forests of Kendu, Mahua, Bija. A Location map of drainage pattern in the study area are shown in **Fig. 3.4.**

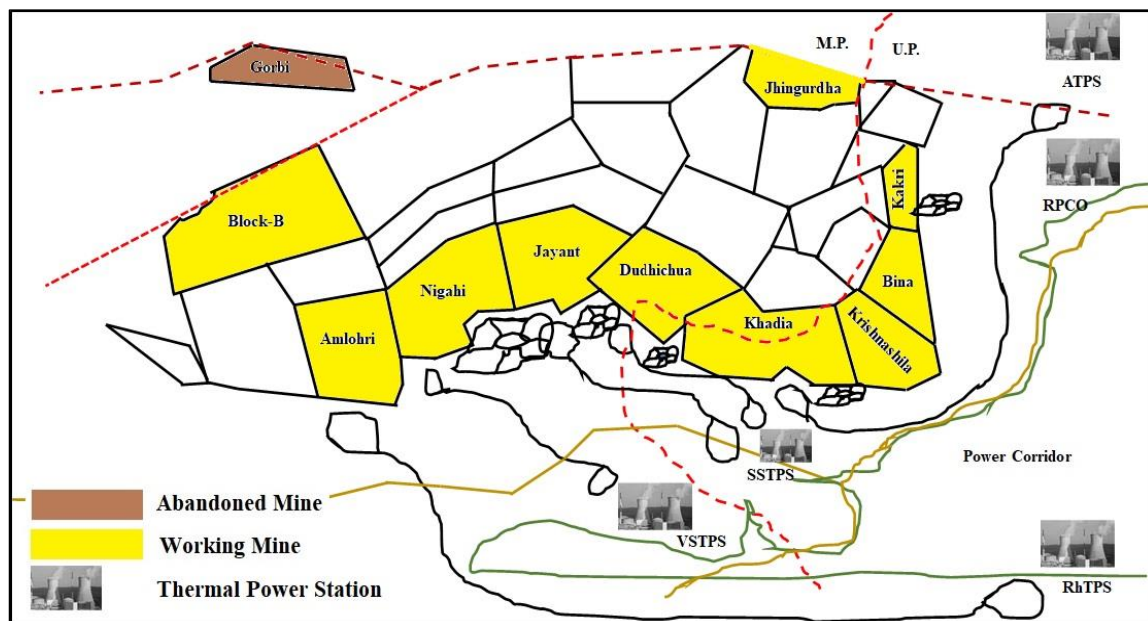**Fig. 3.1:** Location map of study area Singrauli, M.P
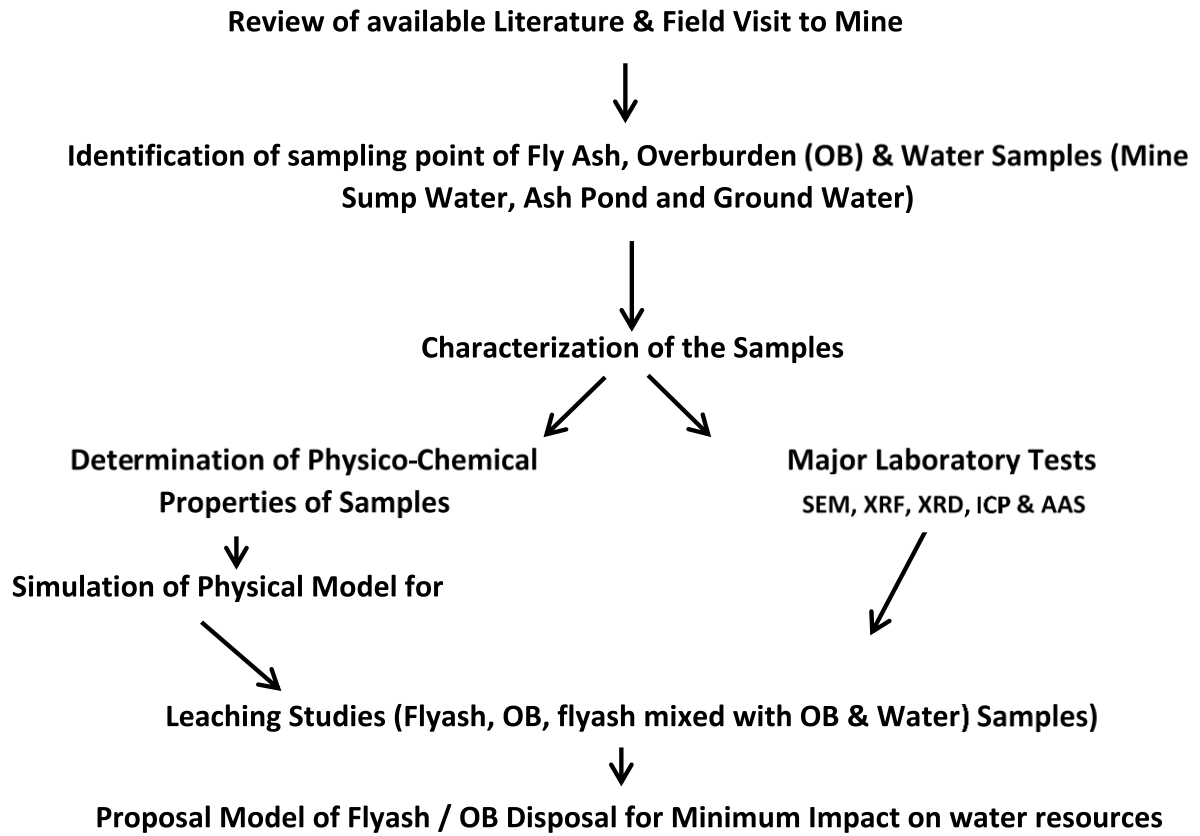

Fig. 3.2: Mine blocks of Singrauli coalfield

Review of available Literature & Field Visit to Mine

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Identification of sampling point of Fly Ash, Overburden (OB) & Water Samples (Mine Sump Water, Ash Pond and Ground Water)

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Characterization of the Samples

↙   ↘

Determination of Physico-Chemical Properties of Samples

Major Laboratory Tests
SEM, XRF, XRD, ICP & AAS

↓

Simulation of Physical Model for

↓   ↓

Leaching Studies (Flyash, OB, flyash mixed with OB & Water) Samples)

↓

Proposal Model of Flyash / OB Disposal for Minimum Impact on water resources

Fig. 3.3: Flow diagram of method and methodology
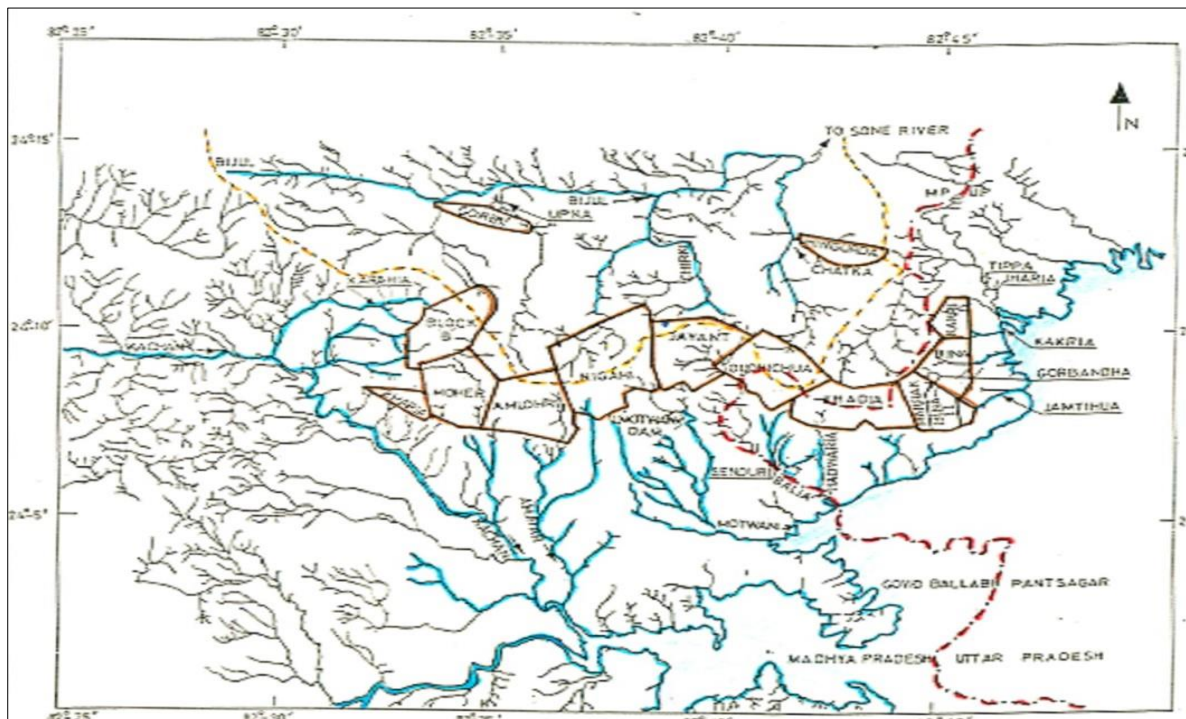

Fig. 3.4: Drainage pattern in the various mines of Singrauli

3.2 Field investigation

3.2.1 Sample collection and storage

3.2.1.1 Collection of overburden

A large quantity of overburden material was collected up to a depth of 60 cm after removing the topsoil from arbitrarily nominated points located on the surface of the overburden dumps of Singrauli coalfield. In this thesis, sixty overburden samples were taken from ten different opencast mines of NCL. The overburden rocks in the study area are friable, loose, and non-cohesive in natural and the size varies from boulder to clay. In the present study area, mine has been divided in to two sections the east and the west with a central exit ramp. Both the external and internal overburden dumping methods are adopted during the mining processes. During external overburden dumping, contour strip benches are prepared to stabilize overburden dumps. These benches improve dump stability against slope failure and reduce surface erosion by reducing slope length. The initial overburden dump of 97 million cubic meters has been removed as external overburden dumps on the east and the west side of excavations. These fill the southern boundary valley and extend over the plain to south. Samples for testing were selected and collected that those represent the average materials found in the mine.

3.2.1.2 Collection of flyash

The flyash used in the present study was collected in dry state from electrostatic precipitators. For this research work, 20 flyash samples were taken from four different Thermal Power Plant of Singrauli coalfield. Gunny bags made of strong poly-coated cotton with 50 kg capacity each were used to collect the dry flyash. During the combustion of pulverized coal in suspension-fired furnaces of thermal power unit, the volatile matter is vaporized and the majority of the carbon is burned off. The mineral matter associated with

the coal, such as clay, quartz and feldspar disintegrate or slag to varying degree. The finer particles that escape with flue gases are collected as fly ash using electrostatic precipitators in hoppers and stored. The hoppers have small outlets. The chute of hoppers was slowly opened and the bags were filled. The mouth of each bag was sealed immediately after collection and the same was again inserted in another poly-pack to prevent atmospheric influences. The bags were transported with utmost care from the plant to laboratory and kept in a secure and controlled environment. Samples of fly ash were taken out as per requirement of test.

Gunny bags were used to collect the loose soils sample leaving the gravels, boulders, etc. aside. The process followed for overburden collection was also repeated to collect flyash material. The details of the sampling location given in **Table 3.1**. Collection samples in the study are shown in the below given photographs (**Fig. 3.5**).

Table 3.1: Details of overburden samples with their sample code

Sl. No	Coalfields	Mine / Thermal Power Plant	Sample Id
1		Gorbi Mine	OBG1
2		Amlohri	OBAML2
3		Nigahi	OBNGH3
4		Jayant	OBJY4
5		Dudhichua	OBD5
6	Singrauli Coalfield	Khadia	OBK6
7		Bina	OBBA7
8		Kakri	OBK8
9		Jhingurdah	OBJH9
10		Block-B	OBBB10
11		Anpara	FAN1
12	Thermal Power Plants	Renusagar	FRS 2
13		Shaktinagar	FSK3
14		Vindhyachal	FVN4

OB samples namely OBG1, OBAML2, and OBNGH3 &……OBBB10 and Flyash such as FAN1, FRS2, FSK3, and FVN4 were subjected to geochemical analysis.

a. Nighai mine

b. Amlohri mine

c. Gorbi block 'B'

d. Dudhichua mine

e. Bina mine

f. Jhingudah mine

Fig 3.5: Sampling collection of different mines of NCL

31

3.2.1.3 Collection of water sample

The impact on the water environment in the inherent parts of opencast mine, area (NCL, Singrauli) was detected by the baseline data analysis of both surface and groundwater. In this work, a total of 60 water samples were collected during April-May (Pre-monsoon) and July (Monsoon) period in 2016, and 2017. These samples were collected from the mine sumps, inlet and outlet of effluent analysed **(Table 3.2)**. These samples were collected from the mine discharge treatment plant various mines of Singrauli Coalfield and nearby areas. Additionally, water samples were also collected, from the upstream, downstream and confluence points of nallah and river where mine water are discharged in order to study the pollution load discharged out by mines. The existing water resources, both surface and groundwater with respective significance are identified through reconnaissance within the study area (approx. 10 km radial distance). These locations were selected to assess the existing status of water quantity in the impact zone. For such study, the sample locations were very carefully chosen so that they be the representative samples of the whole, large water bodies Out of thirty water samples in pre-monsoon, six were nallah and twenty four were mine sumps. In monsoon, thirty water samples were collected also. The collection of water samples has been completed as per Indian standard guidelines (IS: 2488 (part I and V) and IS: 3025) in the year 2016 and year 2017**.** The water samples were collected in pre-treated sampling bottles Plastic gallons were used as a container, because they can be easily handled, inorganically inactive and durable. Manual sampling has been done during a water quality survey. Representative sampling has been carried out with all possible care. The water samples were collected to ensure that it must be both homogeneous and representative and Physico-chemical properties of the water must not be changed during collection and analysis. The time of sampling of mine water is also an important factor because the nature of water samples in mine varies due to washing schedule of mine

machinery, change in shift, etc. However, in this investigation, water samples were collected between 10 a.m. to 1 p.m. during the daytime. The sampling area photographs have been shown in **Fig. 3.6**. The details of the water sampling, codes, and coordinates are summarised **(Table 3.2)**. All the water samples were analysed by standard available techniques. The analysis result of water samples for both the season, has been given in the next chapter **(Chapter 4)**. The locations of water sampling sites of Singrauli Coalfield area have been shown previously in this chapter **(Fig. 3.1 and 3.2)**.

Table 3.2: Detailed sources of water sampling points

Sl. No.	Sample Code	Location	Type of sample	Latitude	Longitude
1	GB	Gorbi	Pit water	N 24.15759	E 82.72292
2	AML 1	Amlohri	Sump water New	N 24.13491	E 82.59043
3	AML 2	Amlohri	Sump water old	N 24.13557	E 82.59049
4	AML 3	Amlohri	Tap Water – Office	N 24.13575	E 82.59052
5	BN 1	Bina	Sump water New	N 24.15759	E 82.74292
6	BN 2	Bina	Sump water Old	N 24.15754	E 82.74293
7	BN 3	Bina	Sump water New	N 24.15746	E 82.74293
8	BN 4	Bina	Tap Water- Guest House	N 24.15737	E 82.74294
9	BN 5	Bina	Hand Pump	N 24.15727	E 82.74296
10	DD 1	Dudhichua	Sump water New	N 24.17028	E 82.67058
10	DD 2	Dudhichua	Workshop effluent	N 24.16995	E 82.67056
11	JY 1	Jayant	Sump water New	N 24.15562	E 82.64228
12	JY 2	Jayant	Sump water Old	N 24.15534	E 82.64239
13	JY 3	Jayant	Tape water	N 24.15516	E 82.64247
14	JY 4	Jayant	Mudwani nalla	N 24.15492	E 82.64253
15	KAK1	Kakri	Sump water Old	N 24.17427	E 82.75422
16	KAK 2	Kakri	Sump water new	N 24.17391	E 82.75426
17	KAK 3	Kakri	Hand Pump	N 24.17372	E 82.75429
18	KHA 1	Khadia	Sump water old	N 24.12088	E 82.71884
19	KHA 2	Khadia	Sump water new	N 24.12054	E 82.71876
20	KHA 3	Khadia	Tap water	N 24.12040	E 82.71875
21	KSL 1	Krishnashila	Sump water old	N 24.14047	E 82.77246
22	KSL 2	Krishnashila	Sump water new	N 24.14007	E 82.77247
23	KSL 3	Krishnashila	Hand Pump	N 24.13956	E 82.77253
24	KSL 4	Krishnashila	Tap Water	N 24.13928	E 82.77254
25	NGH 1	Nighai	Sump water old	N 24.13968	E 82.62831
26	NGH 2	Nighai	Sump water new	N 24.13936	E 82.62842
27	NGH 3	Nighai	Tap Water	N 24.13918	E 82.62842
28	NGH 4	Nighai	Hand Pump	N 24.13886	E 82.62845

a. Krishnashila mine

b. Amlohri mine

c. Bina mine

d. Gorbi abandoned mine

e. Jayant mine

f. Kakri mine

Fig. 3.6: Water bodies and sampling sites in various mines of Singrauli Coalfield

Table 3.3: Indian standards for drinking water (IS-10500, 2012)

Parameter	Ground Water (BIS (2012) Specified Limit)	
	Acceptable Limit	**Max. Permissible Limit**
pH	6.50	8.50
EC (μS cm^{-1})	-	-
TDS	500.00	2000.00
Turbidity	1.00	5.00
Hardness	200.00	600.00
F^-	1.00	1.50
Cl^-	200.00	1000.00
HCO_3^-	-	-
SO_4^{2-}	200.00	400.00
NO_3^-	45.00	No relaxation
PO_4^{3-}	-	-
Ca^{2+}	75.00	200.00
Mg^{2+}	30.00	100.00
Na^+	-	-
K^+	-	-
Fe	300.00	No relaxation
Mn	100.00	300.00
Zn	5000.00	15000.00
Pb	10.00	No relaxation
Cu	500.00	1500.00
Cr	50.00	No relaxation
Ni	20.00	No relaxation
As	10.00	50.00
Se	10.00	No relaxation
Co	-	-
Al	30.00	200.00
Ba	700.00	No relaxation
Ag	100.00	No relaxation
V	-	-

3.3 Laboratory investigation

The samples were then brought to the laboratory of Mining Engineering Department at IIT (BHU) in sealed bags. The material was explored in the laboratory after sieved it to discard gravels, pebbles, etc. Coning and quartering method was followed to collect representative samples from the field. The samples were air-dried for 2 to 3 days, mixed properly, and passed through 4.75 mm sieve before experimentation. Again, coning and quartering procedure was followed to obtain a representative sample for analysis (Karfakis et al., 1996). The sample was prepared in two-part, one part was completely overburden and the second part was mixed with 30% flyash +70% overburden samples. Hence, overburden and 30 overburden +30% flyash samples are prepared. The flyash, overburden and overburden + 30% flyash samples were acid digested with aqua region, filtered and diluted to 100ml. The chemical constituents of the sample were determined using through different instrumentation like, Atomic Absorption Spectrophotometer (AAS) Inductively Coupled Plasma Emission Spectrograph (ICP-ES), SEM, XRF, and XRD analysis also conducted. Apart from these, the pH, Temp, Conductivity and Organic carbon were determined. A brief of these methods followed are described in subsequent **(Table 3.4)**.

3.2.1 Geochemical and mineralogical analysis of flyash, overburden, and overburden +30% flyash

Soil contamination mostly occurs due to anthropogenic activities such as mining, smelting, agriculture, chemical and metallurgical industries. Natural phenomenon such as volcanic eruption, dust storm, forest fire etc. are also important sources of environmental pollution. Overburden soil has an important role in sustaining environmental pollution. The overburden soil helps in maintaining the environmental quality, improve plant and animal health; and regulates biological productivity (Behera et al., 2016; Shirin and Jamal, 2018a;

36

2018b). Mining activities after the soil quality and result in potentially toxic chemical (metals, metalloids and elements) contamination, which leads in various harmful effects. The metals are categorized as "heavy metals" only if their specific gravity is more than $5g/cm^2$.Heavy metals also reduce plant growth, cause accumulation and yield of dry matter. Heavy metals in plants or animals in minor concentrations are not toxic, rather useful. However, lead, cadmium and mercury are potentially toxic even in very small concentrations (Suciu et al, 2008). The mine waste dumps with increase metal concentration have the potential to discharge them to the nearby water bodies downstream may affect cropes on the river belt which affect the health of local inhabitants who consume these crops regularly.

The rock samples collected from exploration sites and active mines, were crushed by using crusher for experimentation **(Fig. 3.7 and 3.8)**. The crushing samples in powder form for the analysis by XRF, XRD, and SEM are shown in the **Fig. 3.9**.

Table 3.4: Analytical methods and instruments used for characterization of flyash and overburden associated rocks

Sl. No.	Instrument used	Analytical Methods	Parameters
1	Multi Parameter Analyzer	Electrode (Probe)	pH, Temp, TDS
		Major Cations	
2	Ion Chromatograph	Ion Chromatography	Chloride, Sulphate, Fluoride, Nitrate and Phosphate
		Major Anions	
3	Ion Chromatography	Ion Chromatography	Calcium, Magnesium, Sodium, Potassium,
		Metals / Heavy Metals	
4	Inductive coupled plasma Optical Emission Spectrometer	Inductive coupled plasma	Aluminum, Arsenic, Iron, Copper, Chromium, Lead, Manganese, Zinc
5	Scanning Electron Microscope (SEM)	Scanning Electron Microscopy (SEM)	Soil Micromorphology, Mineral Identification and Mineral grain Morphology.
6	X-ray fluorescence	Multi-Element Analysis	Elemental Identification
7	X-Ray Diffraction	Multi-Element Analysis	Mineral Identification

Fig. 3.7: Preparation of samples for different laboratory experiment and analysis

Fig. 3.8: Preparation of samples for different laboratory experiment and analysis

Fig. 3.9: Flyash and overburden sample for analysis of elemental and minerals by SEM, XRD and XRF

38

3.3.1.1 Mineralogical analysis

Mineral identification is performed with optical microscopy, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) with micro- chemical analysis by wavelength dispersive X-ray spectroscopy (WDS) or Energy dispersive X-Ray and electron probe Micro-analysis (EPMA) and X-ray fluorescence (XRF). In this thesis, the flyash, overburden and overburden +30% flyash materials were subjected to XRF, XRD and SEM analysis for various purposes as explained in subsequent paragraphs.

3.3.1.2 X-ray diffraction analysis

X-Ray diffraction (XRD) experiment of oven dried flyash, overburden and overburden +30% overburden samples of less 75µ was carried out in order to detect the mineral composition of various mineral phases present in them. Mineral phase's recognition by X-ray diffraction method depends upon the diffraction peaks at different 2ϴ values relating to the d-spacing value of the mineral. As each mineral has a highly ordered crystal structure, its d-spacing values are characteristics to a set of diffraction peaks according to Bragg's law. In the current work, Ultima IV (Make: Rigaku, Japan) with a Cu Ka Central Instrumentation Facility, IIT (BHU). XRD instrument Using for Sample Analysis in Central Instrumentation Facility **(Fig. 3.10)**. Scatting angle 2θ ranged from 5º to 85º at a scanning rate of 5º/min with a step size of 0.05º and the target used was a Cu- Ka (ג = 1.5418Aº) target. One of the standard software namely X pert High Score was used to identify the mineral phases identified in each representative sample is mentioned on the right side of its XRD pattern graph as an index. The XRD pattern graphs of the representative samples of flyash, overburden and overburden + 30% flyash are presented in the next chapter **(Chapter 4)**.

3.3.1.3 X-ray fluorescence analysis

An X-ray fluorescence (XRF) spectrometer is an x-ray instrument used for routine, relatively non-destructive chemical analyses of rocks, minerals, sediments, and fluids. It works on wavelength-dispersive spectroscopic principles that are similar to an electron microprobe (EPMA). However, an XRF cannot generally make analyse at the small spot sizes typical of EPMA work (2-5 microns), so it is typically used for bulk analyses of larger fractions of geological materials. The relative ease and low cost of sample preparation, and the stability and ease of use of x-ray spectrometers make this one of the most widely used methods for the analysis of major and trace elements in rocks, minerals, and sediments.

Fig. 3.10: XRD instrument using for sample analysis

3.3.1.4 Scanning electron microscopy

The scanning electron microscopy (SEM) technique helps in understanding rock and soil micromorphology, pore size distribution, mineral identification, and mineral grain morphology. Microscopy is an analytical technique of adapting microscopes to slight objects and areas of objects, which are not inside the resolution range of the naked human eye. The Flyash, overburden and overburden +30% flyash samples were studied under a high-resolution scanning electron microscopy at Central Instrumentation Facility, IIT (BHU) (JOEL model JSM-6480 LV, Japan) to understand their surface micro-morphology. Since overburden and overburden+30% flyash are nonconductive in nature, their coating was done with a layer of platinum for 180 seconds as needed for the current of 50 mA at Vaccum before acquiring the SEM micrograph. The representative SEM images of Flyash, overburden and overburden +30% flyash mixture samples are shown and discuses in next chapter **(Chapter 4)**.

3.3.2 Geo-environmental characteristics of coal and associated rocks

The following Geo-environmental characteristics of associated rocks were determined:

- Rock- pH (R-pH) of coal and associated rocks,

- Occurrence and percentage distribution of sulfide sulfur in associated rocks,

- Acid production potential (APP) of associated rocks, and

- Acid neutralization potential (ANP) of overburden sandstone

- Acid neutralization potential (ANP) of flyash

A brief discussion about the various geo-environmental parameters is summarized below for proper understanding of the terms used along with the procedure for the determination of various geochemical parameters.

3.3.2.1 Rock-pH

Rock-pH is a modified form of abrasion pH. It may be defined as the pH of the decanted water obtained after 24 hours of the interaction of the finally ground rock powder of 200 mesh with the water of 7.0 pH value at 25^0 C in a solid: liquid ratio of 1:10 by volume (Jamal et al., 2008). The R-pH value of rocks is a function of:

- Constituent ion of the minerals,

- Ease with which these ions pass into aqueous solution,

- Resultant effects that these ions have in the solution phase of the hydrogen and hydroxyl balance of the solution, and

- Type and nature of binding materials.

The determination of R-pH of different rock types and coal were done by standard available technique.

3.3.2.2 Determination of sulfide sulfur in coal and associated rocks

The rate of acid generation is influenced by type of sulfide mineral, available surface area and particle size, type and amount of acid neutralizing minerals, water and oxygen as well as presence of bacteria. The diverse rates of different mineral weathering responses inside the overburden in addition with local climate pattern might be the reason for temporal changes in the drainage chemistry. Shows acid neutralizing oxides in sandstone (**Table 3.5**).

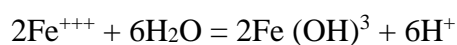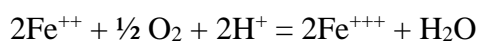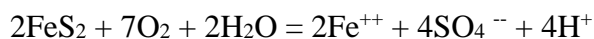These reactions take place for AMD generation

$$2FeS_2 + 7O_2 + 2H_2O = 2Fe^{++} + 4SO_4^{--} + 4H^+$$

$$2Fe^{++} + \frac{1}{2} O_2 + 2H^+ = 2Fe^{+++} + H_2O$$

$$2Fe^{+++} + 6H_2O = 2Fe (OH)^3 + 6H^+$$

$$FeS_2 + 14Fe^{+++} + 8H_2O - 2SO_4^{--} + 15Fe^{+++} 16H^+$$

Table 3.5: Acid neutralizing oxides in sandstone (Overburden)

Sl. No.	Acid Neutralizing Components	Acid Producing Components	Amphoteric Components
1	Na_2O		Al_2O_3
2	K_2O	SO_3	
3	CaO		Fe_2O_3
4	MgO		

3.3.2.3 Acid production potential

It is defined as the amount of acid that would be produced if all pyrites in the sample reacted according to the following stoichiometry:

$$FeS_2 + 15/4\ O_2 + 7/2\ H_2O = Fe\,(OH)_3 + 2SO_4^{--} + 4H^+$$
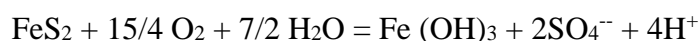
Acid production potential (APP) is determined in kg of $CaCO_3$ equivalent per ton of associated rock (expressed as Kg of $CaCO_3$/ton).

3.3.2.4 Acid neutralization potential

It is defined as the ability of the stratum to neutralize strong acid. Acid neutralization potential (ANP) is determined in kg of $CaCO_3$ equivalent per ton of associated rock (expressed as kg of $CaCO_3$/ton), (Grube et al., 1973; Skousen et al., 1997; White et al., 1997; Page et al., 1982; Brady et al., 2004). Neutralization potential was determined by reacting 2g of samples of rock with a known quantity and strength of hydrochloric acid. The solution was then back – titrated with a base (NaOH) of known strength to pH 7.0 to determine neutralizing content of the sample.

3.3.2.5 Physico-chemical composition of overburden

3.3.3.5.a Bulk density

Bulk density of overburden materials is the mass of the overburden materials per unit volume (**Table 3.6**). High bulk density value cannot be used as vegetation as well as plantation growth. Bulk density was determined by Gravimetric Method.

Table 3.6: Chemical composition of overburden

Sl. No	Parameter	Range
1	Bulk Density (gm/cc)	1.3-2.3
2	pH	6.25-8.98
3	Electrical Conductivity dS/m-1	4.0-7.0
4	Organic Carbon (%)	8.0-9.0
5	Nitrogen(kg/ha)	64.0-85.0
6	Phosphorus(kg/ha)	9.4-11.2

3.3.3.5.b Grain size

Grain size distribution plays an important role in plant establishment of the overburden dump materials. It plays an important role for the maintenance of bulk density of the overburden dump materials. The Grain size distribution was determined by gravimetric method by taking the weight of the fraction passed through the following sieve, 4.75 mm, 2.00 mm, 1mm, 0.425-micron, 0.212 micron, 150 micron, and 75 micron, divided by the total weight of the sample (Ranjan and Rao, 2016).

3.3.3.5.c Texture analysis of overburden

The texture analysis of overburden are shown in the **Table 3.7**.

Table 3.7: Grain size analysis of overburden sample

Sieve Size(mm)
4.75
2
1
0.425
0.212
0.15
0.075
0.01

3.3.3.5.d Determination of pH

The pH of soil or more precisely the pH of the soil solution is very important because soil solution carries in its nutrients such as nitrogen, potassium, and phosphorus that plants need in specific amounts to grow, thrive, and fight off diseases. pH and Electrical conductivity were determined in (soil/water1:2.5) suspension with a pH meter and Conductivity meter respectively. The samples having pH less than 7 were found to be weakly acidic in nature in nature (Brady et al., 2002) and found that a pH range of 6.5 to 7.5 is optimal for plant nutrient availability. If the soil solution is too acidic plants cannot utilize N, P, K and other nutrients they need. In acidic soils, plants are more likely to take up toxic metals and some plants eventually die of toxicity. The measurement of hydrogen ions (or hydroxyl ions) activity of the soil water system is known as soil pH. This indicates the nature of soil reaction.

The pH meter was calibrated with pH 2.54, 4 and 7 buffer solutions prior to the measurement. 10g of OB sample was taken in 100 ml beaker. 50 ml distilled water was added to it. The sample was mixed properly and occasionally stirred for around 30 minutes and pH of the solution was measured using a standard pH meter.

3.3.3.5.e Determination of electrical conductivity

Electrical conductivity (EC) is the common measure of dump materials salinity and is indicative of the ability of an aqueous solution to carry an electric current. The rock composition determines the chemistry of the dump materials and ultimately affects electrical conductivity. For example, limestone leads to higher EC because of the dissolution of carbonate minerals in the dump particles. For mine soil, the researchers proposed that while EC < 4 dS/m may be considered to be good for plant growth (Wayment, 1978; Zeng et al., 2005). EC values within the range of 7 to 8 dS/m may be accepted as fair

and soil/spoil with an EC value 8dS/m should be considered to be of poor quality. Conductivity is a measurement of current carrying capacity, which gives an idea about the soluble salts present in soil samples.

10gm of OB sample was taken in a 100 ml beaker and 50 ml of distilled water was added to it. The solution was stirred for about 1 hour with intervals. The conductivity of the solution was measured using a standardized conductivity meter.

3.3.3.5.f Determination of organic carbon

Organic carbon is an index of dump materials productivity and the amount of carbon broken down from plants and animals that stored in soil. Organic carbon levels greater than 0.8% is rated as good quality of soil or dump and less than 0.4% is rated as low quality of dump. Organic matter of the overburden dump materials was determined by using the Walkley Blake method after first determining the organic carbon by using a conversion factor of 1.724.

The optimum quantity of organic carbon is essential for soil fertility and growth of plant species. Soil organic carbon (matter) in overburden soil samples is determined by digesting sample potassium dichromate $K_2Cr_2O_7$ solution in presence of sulfuric acid and excess of dichromate is measured by titrating the sample against ferrous ammonium sulfate $(NH_4)_2Fe(SO_4)_2.6H_2O$ (FAS) solution.

Soil organic carbon was measured by titration methods of walkley and black (Misra, 1968). In a 500ml flask, 5g of oven dried OB sample was taken. 10ml of $1N K_2Cr_2O_7$ solution was added followed by additional of 20 ml of concentrated sulphuric acid. The sample was mixed thoroughly for about 5 minutes and was allowed to cool for 30 minutes. The mixture was then diluted with 200ml of double distilled water. 10 ml of 85% phosphoric acid was

added followed by mixing of 1ml of diphenylamine indicator. The content was titrated against 1 N $(NH_4)_2Fe(SO4)_2.6H_2O$ solution until the colour of the solution appeared green.

3.3.3.5.g Determination of nitrogen

Nitrogen is a major soil limiting nutrient elements and influence plant productivity. The nitrogen used by plants on dump materials comes from organic matter, fertilizer application and legumes plants (Maiti et al., 2004). Available nitrogen was determined by the alkaline potassium per magnate method.

3.3.3.5.h Determination of phosphorus

Phosphorus of overburden samples was determined in the form of phosphorus pentoxide. Available phosphorus content was determined by Bray method.

3.3.4 Determination of trace/heavy metals of water samples, flyash, overburden and overburden+30% flyash

3.3.4.1 Heavy metal analysis

Inductively coupled plasma optical emission spectrometer (ICP-ES) of Perkin Elmer (ElanDRCe) was used for analysis of heavy metals in water samples, ICP-ES is an analytical technique used for elemental determination in geological and environmental samples at very low concentration. In this technique high temperature plasma (10000K) is coupled with an optical emission spectrometer. Plasma is used to ionize the metal atoms and the emission spectrometer determines the concentration of the elemental from the elemental charge to mass ratio.

About 5g of finely powder Flyash, OB and OB+30%FA sample was taken in a 250 ml in different beaker. 21 ml concentrated hydrochloric acid was added and stirred thoroughly. 7 ml concentrated nitric acid was added and stirred well again. The content was digested

on a hot plate for about 1-2 hours with covering in a fume cupboard. The content was then cooled and filtered through a Whatman 40 filter paper. The filtrate was collected and the volume was made up to 100ml in a volumetric flask with distilled water. The final content was used for determination of trace/ heavy element using AAS, ICP-ES etc. **(Fig. 3.11)**.


Fig. 3.11: Analysis of trace metals in water quality by ICP-ES instrument

3.3.4.2 Flyash characteristics

The chemical reactions and changes expected from fly ash after mixing it with the acidic water can be given as below:

(i) Silica (SiO_2): SiO_2 is commonly found as silica and it is stable below 870^0C. It will least react with any Base or acid except hydrofluoric acid. Hence, silica portion of fly ash may not have any impact on the AMD.

(ii) Alumina (Al_2O_3): Alumina in fly ash is normally not found as γ - Al_2O_3. Hence, alumina portion of fly ash will also not have any impact on acidic water at least at room temperature and pressure condition.

(iii)   Hematite (Fe₂O₃): Fe₂O₃ will also will have least impact on acidic water at room temperature and pressure condition and also upto the most extreme temperature and pressure condition found in the field.

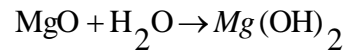(iv)   Lime (CaO): CaO present in fly ash will react with H₂O to give Ca(OH)₂.

$$CaO + H_2O \rightarrow Ca(OH)_2$$
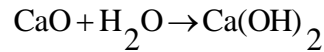
After ionization, Ca (OH)₂ will give Ca⁺⁺ and OH⁻ ion,

$$Ca(OH)_2 \rightarrow Ca^{++} + 2OH$$
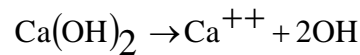
$$\Rightarrow 1\,mole\,of\,CaO\,will\,give\,2\,equivalents\,of\,OH^-\,ion.$$

(v)   Magnesia (MgO) : MgO of fly ash will react with H₂O to give Mg (OH)₂,

$$MgO + H_2O \rightarrow Mg(OH)_2$$

After ionization, Mg (OH)₂ will give Mg⁺⁺ and OH⁻ ion,

$$Mg(OH)_2 \rightarrow Mg^{++} + 2OH^-$$
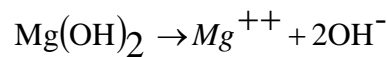
$$\Rightarrow 1\,mole\,of\,MgO\,will\,give\,2\,equivalents\,of\,OH^-\,ion.$$

Thus, it can be said that the effect of SO₃ will change with temperature as changes in its state in normal temperature range (i.e. below 50⁰C). However, the amount of SO₃ in the flyash is 0.18% only. For the sake of simplicity its effect it may be neglected.

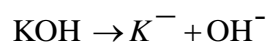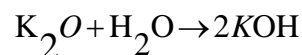(vi)   Na₂O : Na₂O will react with H₂O to give NaOH as below :

$$Na_2O + H_2O \rightarrow 2Na(OH)_2,$$
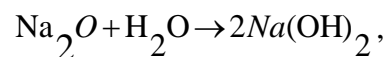
after ionization NaOH will give Na⁺ and OH⁻ ion,

$$NaO \rightarrow Na^+ + OH^-$$
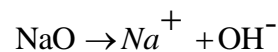
$$\Rightarrow 1\,mole\,of\,Na_2O\,will\,give\,2\,moles\,of\,OH^-\,ion.$$

(vii)   K₂O : K₂O will react with H₂O to give KOH :

$$K_2O + H_2O \rightarrow 2KOH$$

$$KOH \rightarrow K^- + OH^-$$

$$Thus,\,one\,mole\,of\,K_2O\,will\,give\,2\,moles\,of\,OH^-\,ion.$$

(viii) Sulphur trioxide (SO_3): The presence of SO_3 is very low however it decreases the neutralization potential of fly ash by increasing the acidity. It can be found in following three forms:

(a) α - sulphur trioxide: This forms a prismatic needle like crystals. It melts at 17^0C and boils at 45^0C.

(b) β - Sulphur trioxide: The α form, if allowed to stand at 16^0 for some time changes gradually into the β -form. It founds in form of S_2O_6 (chemical formula). This form solidified to give asbestos like needles at 32.25^0C.

(c) γ - Sulphur trioxide: It obtained from β - on complete drying.

Thus, considering CaO, MgO, Na_2O and K_2O for neutralizing acidic water, the acid neutralization potential can be calculated.

3.3.5 Water quality analysis

A Multi-Parameter Analyzer has an Electrode probe (**Fig 3.12**) which was used to measure simultaneously up to 10 parameters such as pH, Conductivity, TDS, Turbidity, DO, ORP, Salinity, Temperature & pH (mv). This meter has a unique designed control unit and sensor technology for analysis of water.

The sensor were checked thoroughly and rinsed with distilled water. The single measurement option was selected. The sensor was then immersed in water sample such that no air bubbles present around the sensor. After the readings stabilized, results were recorded and tabulated.

Fig. 3.12: Water sample analysis by Multi parameter

3.3.6 Graphical representation of water quality data

3.3.6.1 Contour mapping

Contour mapping is one of the ideal approaches to visualize spatial differences in water quality. The geographical information system (GIS) based analysis of the concentration contour maps of the study area was made QGIS Software within the inverse distance weighted (IDW) interpolation technique. IDW interpolation estimates cell values by means of a linear weighted combination of a set of samples points. The weight is a function of inverse distance. The concentration contour maps for some of the important parameters viz. pH, Cd, Cr, Cu, Mn, Pb and Fe have been presented in next chapter.

3.3.6.2 Water type and hydro-geochemical facies

The piper trilinear plot is very useful in determining relationships of different dissolved constituents and classification of water on the basis of its chemical characters. Although there are other plot types that can show an abundance of ions in water, this plot type is especially useful because it allows plotting multiple samples on the same plot, thus allowing for grouping water samples by groundwater facies and other criteria. The piper plot makes easy to determine which type of water samples present, the top quadrant is calcium sulfate waters (gypsum groundwater and mine drainage), the left quadrant is calcium bicarbonate waters (shallow fresh groundwater), the right quadrant is sodium chloride waters (marine and deep ancient groundwater), and the bottom quadrant is sodium bicarbonate waters (deep groundwater influenced by ion exchange).whether it is suitable for human use. Determine the major cations and anions of analyzed water samples discussed in next chapter.