

**EXPERIMENTAL STUDIES ON SPONTANEOUS  
HEATING LIABILITIES OF COALS OF CENTRAL  
COALFIELD LIMITED (CCL)**

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR

THE DEGREE OF

**BACHELOR IN TECHNOLOGY**

IN

**MINING ENGINEERING  
BY**

**SUBHAM KUMAR BEHERA**

**111MN0584**



**DEPARTMENT OF MINING ENGINEERING**

**NATIONAL INSTITUTE OF TECHNOLOGY**

**ROURKELA-769008**

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UNDER THE GUIDANCE OF

**PROF.D.P.TRIPATHY**



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**2015**



## **NATIONAL INSTITUTE OF TECHNOLOGY**

**ROURKELA**

### **CERTIFICATE**

This is to certify that the thesis entitled “**EXPERIMENTAL STUDIES ON SPONTANEOUS HEATING LIABILITIES OF COALS OF CENTRAL COALFIELD LIMITED (CCL)**” submitted by **Sri Subham Kumar Behera, 111MN0584** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

**Date:**

**PROF. D.P. TRIPATHY**  
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Rourkela – 769008

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## **ABSTRACT**

Spontaneous heating of coal is a major problem worldwide and has been a great concern due to its potential to destroy precious coal resources and cause fire accidents in coal mines. Majority of fires occurring in different coalfields are started by spontaneous combustion of coal. The auto oxidation of coal gives rise to spontaneous combustion which is the major root cause for the disastrous fire in coal mines. So, extensive research work is being done to characterize the nature and causes of spontaneous heating to prevent its occurrence. Since, coal is a sedimentary rock its properties vary widely from place to place. So, coal samples from individual seams need to be tested discretely to determine liability towards spontaneous heating.

In this project, different properties of coal obtained from coal mines under Central Coalfields Limited (CCL) have been evaluated based on: proximate and ultimate analysis, calorific value, FT-IR spectroscopy and DTA. The data obtained was used to compare characteristics of different seams with respect to their tendency towards spontaneous heating susceptibility. From the experimental investigations, it was found that, in general the CCL coals have high ash content, low in moisture ( below 2 %), and have higher calorific value. The CHNS analysis showed that the sulphur content of the coals is less than 1% in all the cases. Among organic compounds alkane was found to be the most abundant functional group. The transition temperature was highest at 213.28 °C for seam 2 of Sarubera and was found to be 153.09 °C at its lowest value for seam 8 of Pindra open cast. So, seam 2 of Sarubera is least susceptible whereas seam 8 of Pindra open cast mine is most susceptible to spontaneous heating.

**KEY WORDS:** Coal, Spontaneous heating, Proximate Analysis, Ultimate Analysis, Calorific Value, DTA, FTIR

## ACRONYMS

|       |   |
|-------|---|
| %     | Percentage                              |
| °C    | Degree Celsius                          |
| C     | Carbon                                  |
| Cal/g | Calorie per gram                        |
| DTA   | Differential Thermal Analysis           |
| FC    | Fixed Carbon                            |
| FTIR  | Fourier Transform Infrared Spectroscopy |
| GCV   | Gross Calorific Value                   |
| H     | Hydrogen                                |
| M     | Moisture                                |
| N     | Nitrogen                                |
| S     | Sulfur                                  |
| Tr    | Transition Temperature                  |
| VM    | Volatile Matter                         |
| Wt.   | Weight                                  |

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**CHAPTER 1**  
**INTRODUCTION**

## 1.1 INTRODUCTION

Spontaneous combustion is an oxidation reaction that occurs without an external heat source. The process changes the internal heat profile of the material leading to a rise in temperature. This can eventually lead to open flame and burning of the material [16].

Spontaneous combustion of coal is a fire initiated by the oxidation of coal. Coal fires require Three basic elements to exist as shown in Figure 1.1.



**Fig. 1.1 Fire triangle**

(Source: <http://www.fire-training.com.au/wp-content/gallery/fire-training/firetriangle.jpg> )

The process leading to spontaneous combustion can be summarized as follows:

- ✓ Oxidation occurs when oxygen reacts with the fuel, in this case coal.
- ✓ The oxidation process produces heat.
- ✓ If the heat is dissipated, the temperature of the coal will not increase.
- ✓ If the heat is not dissipated then the temperature of the coal will increase.
- ✓ At higher temperatures the oxidation reaction proceeds at a higher rate.
- ✓ Eventually a temperature is reached at which ignition of coal occurs.

Heat dissipation depends on the thermal conductivity of coal and the surrounding rock, on convection processes caused by wind and barometric changes in the atmosphere and on minor and major fracture density in the rock mass [16].

## 1.2 MOTIVATION

Coal fires have economic, social and ecological impacts. They are a serious health and safety hazard, affecting the environment by releasing toxic fumes and causing subsidence of surface

infrastructure. According to several investigations spontaneous heating is the major reason behind mine fire.

Across the world, thousands of underground coal fires are burning at any given moment. Among them Jharia coalfield in Jharkhand, India is a notable example. As of 2010 68 occurrences of mine fire has been reported from this area.

The problem is most acute in industrializing, coal-rich nations such as China and India. Global coal fire emissions are estimated to cause 40 tons of mercury to enter the atmosphere annually, and to represent three percent of the world's annual CO<sub>2</sub> emissions [13].

To prevent occurrence of Fire due to spontaneous heating adequate steps must be taken to control spontaneous heating in early stages. This requires us to design suitable mining methods and establish guidelines for safe handling and storing practices suitable to the specific coal deposit. Thus the nature of the coal must be extensively studied and its tendency to self-heating must be determined which can be used as an aid in preventing spontaneous combustion.

### **1.3 OBJECTIVES**

The objectives of the project are enumerated below:

- To carryout experimental studies viz.: proximate analysis, ultimate analysis of coal.
- To determine the calorific value of coal using Digital bomb calorimeter
- To determine organic components by Fourier Transform Infra-red Spectroscopy (FT-IR).
- To evaluate spontaneous heat susceptibility of coals from thermograms obtained from by Differential Thermal Analysis (DTA).

### **1.4 METHODOLOGY**

Over the years several methods has been suggested by researchers for quantitative measurement of susceptibility of coal towards spontaneous heating. They help us to design coal panels and decide rate of extraction keeping in mind the incubation period of coal. If open panels are still not extracted after incubation period proper preventive measures should be taken to avoid spontaneous combustion like sealing off the area from ventilation. Several theories put forward to explain spontaneous combustion include Bacterium theory, Phenol theory, coal-oxygen complex theory, pyrite theory etc. But still these theories cannot explain spontaneous heating in every occasion because of complex chemical structure of coal. More than one method is available to assess the

spontaneous heating liability of coal. The standard methods followed in India include: Differential thermal Analysis, Crossing Point Temperature, Wet oxidation potential, Crossing point temperature, Russian U-index, Oplinski index, adiabatic calorimetry, Differential scanning calorimetry etc. None of the methods can determine spontaneous heating liability single-handedly. So, more than one method is followed for accurate results most of the times.

In this project, an attempt has been made to understand the effect coal constituents on its tendency to spontaneous heating. The constituents of coal was established by data obtained from proximate analysis, ultimate analysis, and Fourier transform infrared spectroscopy. The spontaneous heating tendency was derived from transition temperature obtained during differential thermal analysis. Later several plots were produced between different constituents and transition temperature to observe the trend of spontaneous heating susceptibility against the corresponding constituent.

### **1.5 LAYOUT OF CHAPTERS**

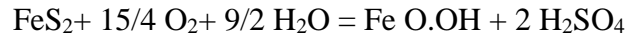
The thesis has been split into 4 different chapters.

- ✓ The first chapter introduces the concept of spontaneous heating and the objective of the project.
- ✓ The second chapter focuses on different theory of spontaneous combustion and the factors that influence it.
- ✓ The third chapter describes the experimental investigation done on different samples.
- ✓ The fourth chapters summarizes all the results obtained during the experimental work followed by discussion about their significance related to spontaneous heating of coal.
- ✓ The conclusions derived from experiment are mentioned at the end.

**CHAPTER 2**  
**LITERATURE REVIEW**

## 2.1 THEORIES OF SPONTANEOUS COMBUSTION

### 2.1.1 Pyrite Theory:-



In pyrite rich coals oxidation of pyrites as shown above generates high quantity of heat. This can initiate spontaneous heating of coal if iron pyrite is present in substantial amount in finely powdered and spread state in presence of moisture. Iron pyrites, oxygen and moisture react exothermically, and produce materials of greater volume compared to original pyrite thus making the coal more porous facilitating adsorption of oxygen. The equation shows that both oxygen and moisture, alter and weather pyrite producing sulfuric acid as by-product. Existence of moisture doubles the rate of oxidation and pyrite if present in finely divided state increases the rate of oxidation by 10 fold. Pyrite if below 5% does not have significant effect on oxidation [16].

### 2.1.2 Bacterial Theory:

It is supposed that bacteria are involved in accelerating spontaneous combustion. Later investigations showed the effect of bacteria on self-heating tendency of coal is negligible. But the effect of bacteria cannot be ruled out totally. Spontaneous heating of hay stack or wood is due to bacterial activity. The theory cannot be authenticated or overlooked [16].

### 2.1.3 Humidity Theory:

It states that the quantity of heat required in removing water from coal is much higher than the quantity of heat liberated by atmospheric oxidation of coal. The temperature of heating would definitely decrease if the evaporation is to be done at the cost of heating. But as a matter of fact water is one of the oxidation product formed during low temperature oxidation of coal along with CO and CO<sub>2</sub>. Higher humidity prevents vaporization, thus the heat produced during oxidation is not lost and utilized to aid oxidation [16].

#### **2.1.4 Phenol Theory:**

Experimental research and investigations have justified that poly phenols and phenolic hydroxyls oxidize faster than many other organic groups. This theory is convincing as well as interesting as it drives a way in determining liability of coal to spontaneous heating [16].

#### **2.1.5 Coal-Oxygen Complex Theory:-**

The native radical site is the point of initiation of oxidation of coal. The formation of peroxy radical and hydro peroxides justifies the fact that during the formation mechanism oxygen and moisture are initially incorporated into an organic matrix. In fact these species may react, decompose, reform, and alter to form a wide range of complex showing oxygen functionality in matrix or gaseous product form [16].

It has been suggested that that coal oxygen interaction takes place via formation of peroxy complex. The above process occurs in 3 stages:

1. The physical adsorption of oxygen which takes place at low temperatures and it requires low activation energy.
2. The Chemisorption step where motion of complex containing active form of oxygen called per oxygen occurred and it occurred between temperature ranges 70°C to 80°C. Much amount of heat is involved in this process.
3. Rapid chemical reaction resulting in decomposition of per-oxygen formed leading to release of CO, CO<sub>2</sub> and H<sub>2</sub>O and finally active combustion taking place.

#### **2.1.6 Heating due to earth movement:**

Movement in earth crust can crush the coals. This crushing of coal under pressure from super incumbent rock produces heat which can increase chances of spontaneous combustion. Events like sudden roof fall result in adiabatic compression producing heat and elevating ambient temperature. The crushing of coal also increases its surface area increasing rate of oxidation [16].



## 2.2 MECHANISM OF SPONTANEOUS HEATING

The oxidation of coal is an exothermic reaction. The interaction between in coal and oxygen is through adsorption process at low temperatures and turns in to chemisorption beyond ambient temperature. This oxidation produces oxides of carbon like CO, CO<sub>2</sub> and some resultant heat with traces of water vapor. If the heat is allowed to dissipate it does not cause any harm. But when the heat remains stagnant it raises the temperature around the coal and accelerates the oxidation process. As the thermal equilibrium is disturbed i.e. the rate of evolution of heat is greater than the rate of dissipation of heat spontaneous heating starts to occur. At ambient temperatures the degree of proneness of coal to spontaneous heating is greatly affected by the extent of oxidation of coal. The complex low temperature oxidation involves several instantaneous chemical processes which erode and change the organic structure of coal.

Investigators have found that the oxidation of coal like other chemical reactions follow Arrheius Law. The relationship between reaction chemistry, rate of reaction constant and temperature determines the Arrhenius equation [16].

$$\ln(k) = \ln(A) - EA/ (RT)$$

Where, k = Rate of reaction constant

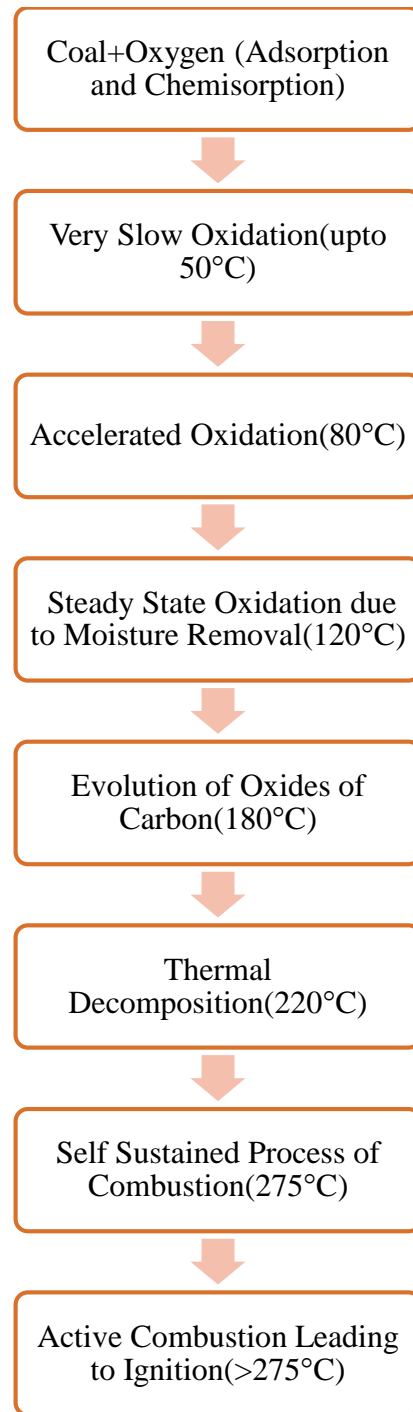
A = Frequency factor

EA = Activation energy

R = Gas constant

T = absolute temperature

The stages of spontaneous heating has been shown in the following scheme.



**Fig.2.1 Stages of spontaneous heating [14]**

## 2.3 FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

Several factors are known to affect the spontaneous heating susceptibility of coal. They are discussed below.

|   |                    |                              |
|---|--------------------|------------------------------|
| Factors Affecting Spontaneous Heating of Coal | Mining Factors     | Rate of Advance              |
|   |                    | Pillar Condition             |
|   |                    | Roof Condition               |
|   |                    | Mining Methods               |
|   |                    | Crushing                     |
|   |                    | Packing                      |
|   |                    | Effect of Timber             |
|   |                    | Roadways                     |
|   |                    | Leakage                      |
|   |                    | Multiseam Working            |
|   |                    | Coal Loss                    |
|   |                    | Worked out Areas             |
|   |                    | Heat From Machines           |
|   |                    | Stowing                      |
|   |                    | Ventilation Pressure         |
| Change in Humidity                            |                    |                              |
|   | Geological Factors | Seam Thickness               |
|   |                    | Seam Gradient                |
|   |                    | Caving Characteristics       |
|   |                    | Friability                   |
|   |                    | Depth of Cover               |
|   |                    | Geothermal Gradient          |
|   | Seam Factors       | Rank                         |
|   |                    | Ash Matter                   |
|   |                    | Petrographic Composition     |
|   |                    | Effect of Previous Oxidation |
|   |                    | Available Air                |
|   |                    | Temperature                  |
|   |                    | Particle Size                |
|   |                    | Moisture                     |
|   |                    | Sulphur                      |
|   |                    | Physical Properties          |
|   |                    | Heat Due to Earth Movement   |
|   |                    | Bacteria                     |
|   |                    | Pyrite Content               |

**Fig.2.2 Factors affecting spontaneous heating of coal [12] [16]**

**2.3.1 Rank of Coal:** The rank of coal is dependent upon the percentage of carbon, ash, moisture and volatile matter present in the coal. Experimental studies show that with increase in rank the spontaneous heating susceptibility of coal continues to fall. The high concentration of volatile matter and moisture in low rank coal are responsible for its higher tendency to undergo spontaneous heating. Other than volatile matter porosity of coal is also high in low rank coals. High porosity ensures a larger surface area for reaction causing rapid oxidation. The presence of unsaturated hydrocarbons also tends to increase the self-heating effect among low rank coals. Yet the spontaneous heating tendency among same rank coals may vary widely depending on other factors [16].

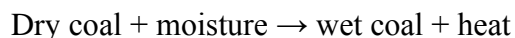
**2.3.2 Petrographic Characteristics:** The original plant materials that turn into coal define its petrological nature. The environment during the formation of coal is also a major factor in this matter. But the rank of coal which depends upon the degree of coalification have no significant effect on the petrology of coal. The micro-constituents are homogeneous and are termed as macerals. The macerals are further divided in to three groups known as vitrinite, liptinite and inertinite. All these macerals come from different tissues of plants. Vitrinite is formed from woody materials, liptinite comes from spores, resins and cuticles and lastly Inertinite has its origin in oxidized plant material. Among coals of same rank a higher inertinite content indicates higher tendency for spontaneous heating. Increase in liptinite content of coal is also seen to increase the proneness to spontaneous heating [16].

**2.3.3 Presence of Methane:** High concentration of methane around coal may inhibit the oxidation reaction by directly reducing oxygen concentration at low temperatures. But oxidation of methane is exothermic and may propel the self-heating of coal. The decrease in desorption rate of methane with time is found to increase the self-heating process by exposing more coal to oxygen. An emission rate lower than  $5\text{m}^3/\text{ton}$  fuels the oxidation process but an emission rate higher than  $8\text{m}^3$  reduces oxidation rate significantly [16].

**2.3.4 Minerals:** A high concentration of minerals effectively reduces the carbon content affecting heat value of the coal. Certain minerals like that of pyrite have their role in spontaneous heating as well. Minerals may accelerate or decelerate oxidation. Presence of silica and alumina reduces

oxidation rate. Pyrite ( $\text{FeS}_2$ ) undergoes oxidation itself and produces heat aiding the oxidation of coal. Not only heat but reaction product also affect oxidation by creating pressure causing fractures in coal, thus providing more surface area [16].

**2.3.5 Moisture:** Moisture may retard or aid the oxidation reaction. Vaporization causes cooling preventing spontaneous combustion. But wetting of coal generates heat which aids spontaneous heating.



On the other hand, moisture adsorbed on the surface prevents diffusion of oxygen reducing rate of oxidation [15].

**2.3.6 Particle size and surface area:** Several investigation shows smaller size of coal particles increases rate of oxidation by increasing the surface area of coal available for oxygen adsorption. The rate oxidation increase with reduction of particle size until acritical diameter is attained beyond which the rate of oxidation remains constant [16].

**2.3.7 Physical properties:** Certain physical properties like porosity, hardness, permeability, specific heat and thermal conductivity affect rate of oxidation. Low hardness and low value of thermal conductivity makes the coal more susceptible to spontaneous heating [16].

**2.3.8 Friability:** Coal oxidation rate varies linearly with available surface area and has a positive relationship. Highly friable coals expose more surface area accelerating rate of oxidation and producing more heat [16].

**2.3.9 Availability of Oxygen:** The concentration oxygen in the environment affect the degree of oxidation. Easy availability of oxygen through cracks and microscopic pores present in coal enhances the self-ignition process [16].

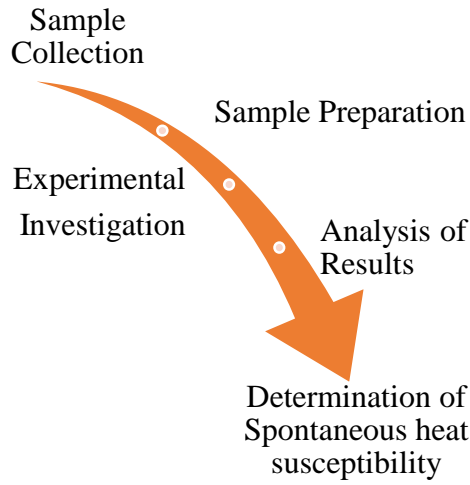
**2.3.10 Heat and mass transfer:** Heat if dissipated adequately can prevent spontaneous combustion whereas an easy movement of reactants aids the oxidation process. Heat can be transferred passively by temperature gradient between coal and its outside. If adequate airflow is

available convection plays a major role to dissipate the heat actively. Oxygen and water taking part in reaction move by diffusion and convection which is improved if the coal is porous [16].

**2.3.11 Volume to surface area ratio:** During handling of coal the volume to surface ratio of the coal stacks has a significant impact on ignition behavior. But same volume to surface area ratio does not ensure same ignition temperature as other factors come to effect [16].

**CHAPTER 3**  
**EXPERIMENTAL INVESTIGATION**

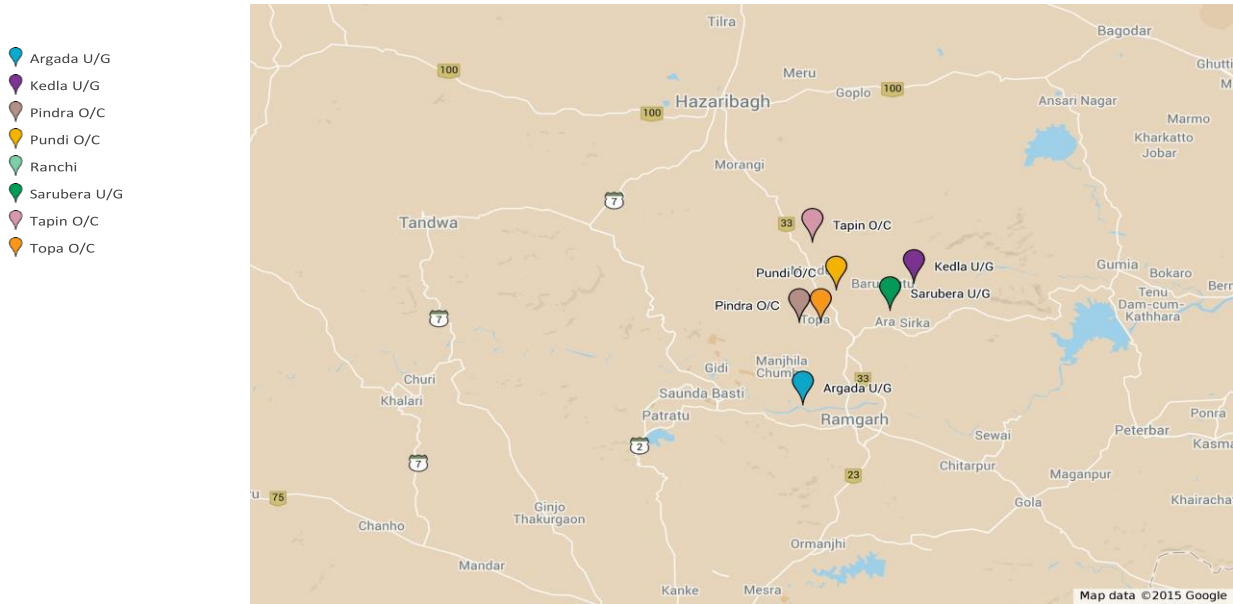
### 3.1 SEQUENCE OF WORK PROCEDURE



**Fig.3.1 Sequence of work procedure**

### 3.2 SAMPLE COLLECTION

#### 3.2.1 Site Description:-



**Fig.3.2 Sample collection sites of CCL [10] [11]**

The coal samples were collected from different seams of five mines. The mines (shown in table) are situated in RAMGARH District. The district is a part of Chotanagpur plateau. The regions with physiographic significance include Damodar valley. Damodar Valley is formed between



Hazaribag plateau in north and Ranchi plateau in south. In other words Ranchi and Hazaribag plateau are separated by east–west running Damodar Valley. The highest peak in the region is Barka Pahar (Marang Buru), about 1049 meters high above sea level located along the Ramgarh-Ranchi border. Damodar is the main river of the district and it also forms a major river basin, comprising a number of tributaries. Important Small Rivers are Hurhuri, Gomti, Barki, Kurum, Kochi, Sherbhuki, Dhobdhab etc. Suwarn Rekha River flow south eastern part of district. Its Tributaries include Kadamgara, Khatgara etc. The district holds a strong position on the mineral map of the country. The district is endowed with a large and rich deposit of coal & coal bed methane (CBM) and also possesses various other minerals like Limestone, Fire Clay, etc. The coalfields of the Ramgarh district lying in the Damodar Valley. The important geological formation of the district is Gondwana System and the rock of the Damuda Group of Lower Gondwana age comprises the most important coal seams. The coal deposit of the district mainly found in South Karanpura, West Bokaro and Ramgarh coalfields [1] [13].

**Table 3.1 Details of coal samples collected from seams of CCL [10] [11]**

| <b>MINE NAME</b>     | <b>LATITUDE</b>              | <b>LONGITUDE</b>             | <b>SEAM NAME</b> |
|----------------------|------------------------------|------------------------------|------------------|
| TOPA OPENCAST        | 23 <sup>0</sup> 44' 22.18" N | 85 <sup>0</sup> 28' 52.69" E | Seam v           |
|                      |                              |                              | Seam vi          |
|                      |                              |                              | Seam viii C      |
|                      |                              |                              | Seam vii C       |
|                      |                              |                              | Seam vii B       |
| PINDRA OPENCAST      | 23 <sup>0</sup> 44' 23.73" N | 85 <sup>0</sup> 27' 23.99" E | Seam vi          |
|                      |                              |                              | Seam v           |
|                      |                              |                              | Seam viii C      |
|                      |                              |                              | Seam vii C       |
|                      |                              |                              | Seam vii B       |
| PUNDI OPENCAST       | 23 <sup>0</sup> 46' 39.12" N | 85 <sup>0</sup> 29' 56.70" E | Seam vii         |
|                      |                              |                              | Seam iv          |
| TAPIN OPENCAST       | 23 <sup>0</sup> 49' 59.40" N | 85 <sup>0</sup> 28' 20.16" E | Seam viii A      |
|                      |                              |                              | Seam viii B      |
|                      |                              |                              | Seam viii C      |
| ARGADA UNDERGROUND   | 23 <sup>0</sup> 38' 38.74" N | 85 <sup>0</sup> 27' 42.31" E | Seam v           |
|                      |                              |                              | Seam vi          |
| KEDLA UNDERGROUND    | 23 <sup>0</sup> 47' 9.30" N  | 85 <sup>0</sup> 35' 18.29" E | Seam viii A      |
|                      |                              |                              | Seam viii B      |
| SARUBERA UNDERGROUND | 23 <sup>0</sup> 45' 12.81" N | 85 <sup>0</sup> 33' 40.68" E | Seam i           |
|                      |                              |                              | Seam ii          |

### 3.2.2 Grab Sampling:

Sample collection was done following guidelines mentioned in IS-436(Part-I), 1964 [4].

- ✓ A smooth, crack and dirt free representative freshly exposed face was chosen.
- ✓ Coal was cut using shovel.
- ✓ The freshly cut coal was collected in plastic bags and sealed immediately using rubber bands to avoid exposure to external dirt and moisture.
- ✓ Each sample was labelled immediately with the seam no. and mine name.

### 3.3 LABORATORY EXPERIMENTATION

- ✓ The coal sample was pulverized manually using mortar and pestle.
- ✓ The pulverized coal was sieved to obtain only particles of size -212 micron.
- ✓ About a 100g finely pulverized -212 micron coal specimen was prepared from each sample
- ✓ Each specimen was kept inside a zipper bag and sealed immediately to avoid exposure to moisture.
- ✓ Each bag was labelled with the mine name and seam no.
- ✓ Also the bags were labelled by integers from 1 to 21 for easy tracking during experiment.

**Table 3.2 List of experiments**

|               |                       |   |
|---------------|-----------------------|---|
| STUDY OF COAL | Proximate Studies     | Moisture  |
|               |                       | Ash   |
|               |                       | Volatile Matter   |
|               | Ultimate Studies      | Carbon  |
|               |                       | Hydrogen  |
|               |                       | Nitrogen  |
|               |                       | Sulphur   |
|               | Thermal Studies       | Differential Thermal Analysis                                 |
|               | Spectroscopic Studies | Fourier Transform Infrared Spectroscopy                       |
|               | Calorimetry           | Gross Calorific Value Measurement in Digital Bomb Calorimeter |

### **3.3.1 Proximate Analysis:-**

This is a quantitative study of amount of moisture, ash and volatile matter present in coal. While the amount of moisture, ash and volatile matter can be determined directly further calculation gives us the quantity of fixed carbon present in the coal. For proximate analysis the guidelines described in Indian Standard IS-1350(part-I), 1984 was followed [3].

#### **3.3.1.1 Moisture:-**

Moisture can be found intrinsically or extrinsically in coal. Intrinsic or inherent moisture is the moisture which resides inside microscopic fissures of the coal sometime freely and sometimes as water of hydration. Its origin in the coal can be dated back to the formation of the seam. On the other hand extrinsic or external moisture gets mixed with coal during mining activities. External moisture can be removed by air drying whereas internal moisture is removed by heating the coal at a steady rate at temperatures above 100°C. Moisture present in coal has following effects.

- Moisture has to be transported, handled and stored increasing the costs for these operations.
- It absorbs heat produced during oxidation, thus decreasing heat output per kg.
- Increases heat loss, due to evaporation and superheating of vapor.
- Helps, to a limit, in binding of fines.
- Aids radiation heat transfer.

The quantity of moisture was determined as per part-I of IS-1350, 1984 as follows [3].

#### **❖ Procedure**

- ✓ 21 watch glasses were labelled from 1 to 21 using a marker corresponding to the sample no.
- ✓ Each empty watch glass is pre-weighed and their weights were recorded
- ✓ 1g of -212 micron sized coal was weighed and was transferred to the watch glass of corresponding no.
- ✓ The watch glass with coal was weighed again and its weight recorded
- ✓ Then the watch glass was transferred to a desiccator and kept their until the above procedure is completed for all the samples to avoid absorption of external moisture

- ✓ The moisture oven was fired up and its temperature was set at a steady 108 C.
- ✓ The coal filled watch glasses were transferred to the oven and kept their for 1.5 hrs before being removed and kept in the desiccator for cooling
- ✓ Then each crucible was weighed again with the coal to determine the final weight post heating
- ✓ The amount of moisture was determined according to the following formula.

$$\% \text{ Moisture} = \frac{y-z}{y-x} \times 100 \quad (1)$$

Where, x=weight of empty watch glass, y=weight of watch glass + coal before heating, z=weight of watch glass + coal after heating.

### 3.3.1.2 Ash:

Ash is the incombustible residue left after the burning of coal in air. It originates from the mineral matter present in the coal. There are many minerals present in the coal. Quartz, kaolinite, illite, feldspars, Dolomite, Siderite, Pyrite, calcite, pyrite and gypsum make most of these minerals and called major minerals. A chemical composition of typical coal ash is Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, Na<sub>2</sub>O, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, TiO<sub>2</sub>, SO<sub>3</sub> etc.

Indian coals produce high quantities of ash due to their drift origin. Ash is quantitatively and qualitatively different from the mineral matter present in coal due to various chemical changes such as loss of water of hydration from silicate minerals, loss of carbon dioxide from carbonate minerals, oxidation of iron pyrite to iron oxide and fixation of oxides of Sulphur by bases such as calcium and magnesium. The circumstances during incineration decide the extent of change of weight, so standard practice must be followed to get accurate results.

Percentage of ash in coal affect it in following ways.

- Reduces handling and burning capacity
- Increase the handling costs
- It hampers combustion efficiency and boiler efficiency
- Causes clinkering and slagging in blast furnace operation
- Can corrode metal pipes in ash ponds if has high Sulphur content

The quantity of Ash was determined as per part-I of IS-1350, 1984 as follows [3]. Silica crucibles of standard dimensions are used in the process as they can withstand high temperatures of 850° C.

❖ **Procedure**

- ✓ The muffle furnace is heated to a temperature of 450° C.
- ✓ The crucible is preheated to discard any remaining mineral matter.
- ✓ The weight of empty crucible is taken and recorded.
- ✓ -212 micron Coal sample of approximately 1g is transferred to the crucible.
- ✓ The crucible containing coal is weighed and the weight is recorded.
- ✓ The crucible is covered with lid and transferred to the muffle furnace.
- ✓ The crucibles are kept inside the furnace in pre-decided order for easy tracking.
- ✓ The sample is held at 450°C for 30 minutes in the first stage and then at 850 C for additional 1 hour in the second stage.
- ✓ The crucible is taken out and labelled as per previous record.
- ✓ The crucibles are transferred to a desiccators for cooling.
- ✓ Then the lids are taken off and weight of the crucible and ash is taken and recorded.
- ✓ The percentage of ash is calculated in the following manner.

$$\% \text{Ash} = \frac{z-x}{y-x} \times 100 \quad (2)$$

Where, x=weight of empty crucible, y=weight of crucible + coal before heating, z=weight of crucible+ ash after heating.

**3.3.1.3 Volatile Matter:**

Volatile matters are the methane, hydrocarbons, hydrogen and carbon monoxide, and incombustible gases like carbon dioxide and nitrogen found in coal. Thus the volatile matter is an index of the gaseous fuels present. Typical range of volatile matter is 20 to 35%.

Mathematically, volatile matter is the loss of mass corrected for moisture, which results when coal is heated in a VM crucible under prescribed conditions. The observed loss in mass is due to the release of various volatile components present in coal or thermal decay of complex compounds.

The different volatile components include hydrogen, carbon monoxide, carbon dioxide, methane and other hydrocarbons, ammonia, tar vapor, organic Sulphur, oxides, water vapor etc. CO<sub>2</sub> and water vapor are released due to break down of carbonates and water of crystallization respectively.

The effect of VM on coal:

- Increases the flame length proportionally and eases the ignition of coal
- Affects minimum limit on the furnace height and volume
- Increases heat output of coal
- A little to moderate quantity of volatile matter actually help in cake formation

The quantity of Volatile Matter was determined as per part-I of IS-1350, 1964 as follows.

#### ❖ Procedure

A special cylindrical silica crucible of standard dimensions is used for this purpose.

- ✓ The Muffle Furnace is preheated to 800° C
- ✓ The crucible with its lid is heat treated to remove any impurities left over from previous use.
- ✓ Then the temperature of furnace is raised to a steady 925° C
- ✓ The empty crucible with its lid is weighed and its weight recorded
- ✓ Approximately 212 micron size coal is transferred to the crucible, covered with lead, weighed and recorded.
- ✓ The crucible with the sample and lid is kept inside furnace for exactly 7 minutes
- ✓ The crucible should be loaded on to the furnace in pre-decided order for easy tracking
- ✓ The crucible is taken out and kept in the desiccators for cooling.
- ✓ Then the crucible with its lead is weighed again and recorded
- ✓ The percentage of volatile matter is found in the following manner.

$$\% \text{VM} = \left( \frac{y-z}{y-x} \times 100 \right) - M \quad (3)$$

Where, x=weight of empty crucible +lid,

y=weight of crucible + coal+ lid before heating

z=weight of crucible+ V.M residue+ lid after heating,

M=% moisture.

### 3.3.1.4 Fixed Carbon:

Fixed carbon is the solid fuel left in the furnace after volatile matter is distilled off. It consists mostly of carbon but also contains some hydrogen, oxygen, Sulphur and nitrogen not driven off with the gases. Fixed carbon gives a rough estimate of heating value of coal.

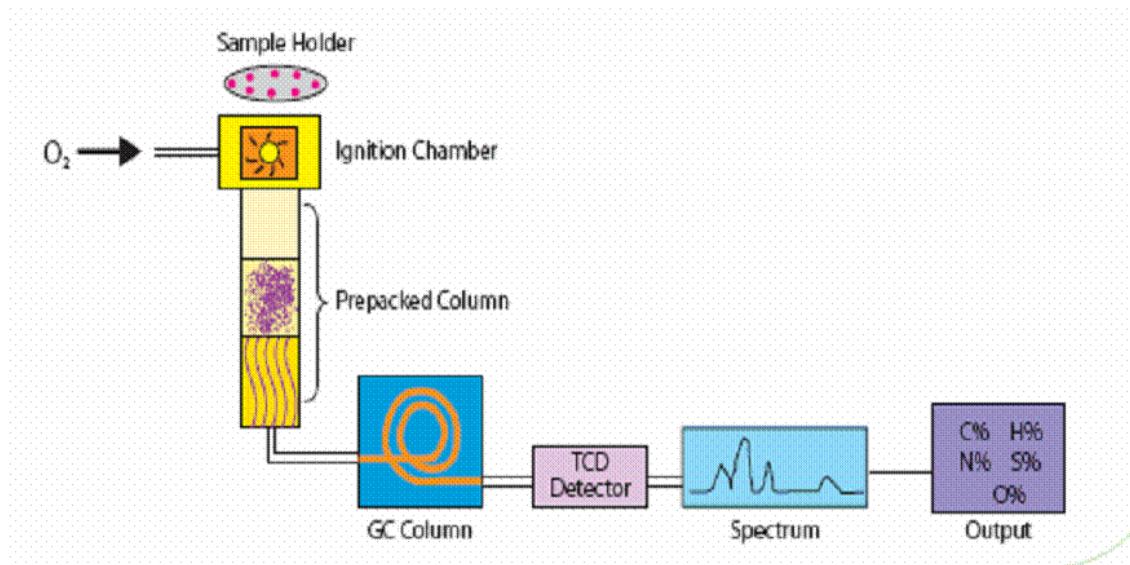
It is calculated as follows.

$$\% \text{ FC} = 100 - (\% \text{ VM} + \% \text{ M} + \% \text{ A}) \quad (4)$$

### 3.3.2 CHNS Analysis

In CHNS analysis the absolute percentage of carbon(C), hydrogen (H), nitrogen (N), Sulphur(S) is determined.

The CHNS analyzer used in this case was the “Vario EL cube” by Elementary. The principle of this equipment is described below.



**Fig.3.3 CHNS analysis principle**

(Source: <http://www.rsic.iitb.ac.in/Chn.html> )

CHNS analysis is based on the classical Pregl-Dumas method where samples are combusted in a pure oxygen environment, with the resultant combustion gases measured by Gas Chromatography using TCD detector [5].

The total nitrogen, carbon, and sulfur are determined using a CHNS analyzer. The following steps are followed.

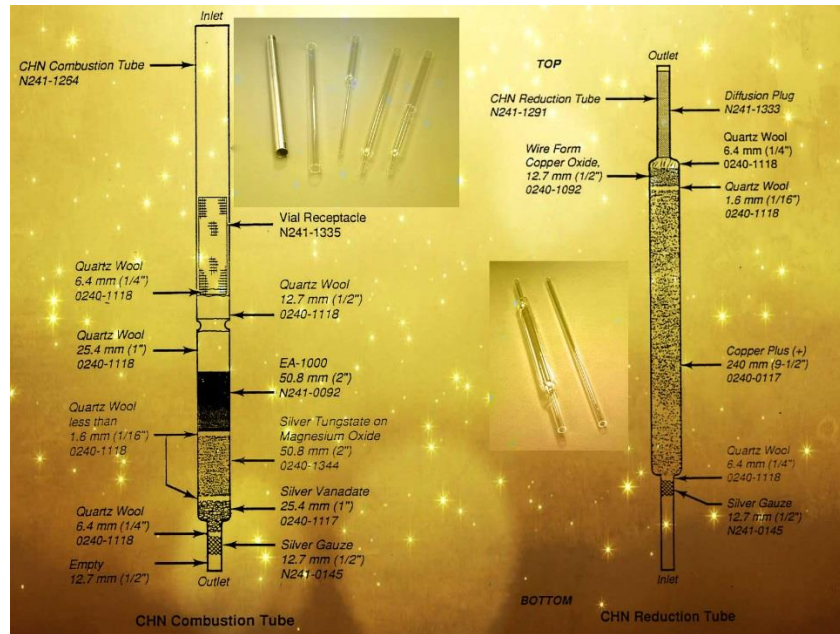


**Fig.3.4 CHNS analysis apparatus (Model: Vario EL)**

❖ **Procedure**

- ✓ Up to 5-10 mg air dried 212 micron size coal sample was weighed in an ultrasensitive balance.
- ✓ The weighed coal was kept inside a tin capsule.
- ✓ The capsule with the sample was allowed to burn at 1000° C
- ✓ The sample and container melt, and flash combustion promoted by tin is observed in a temporary oxygen rich atmosphere





**Fig.3.5 CHN combustion & reduction tube**

(Source: [http:// image.slidesharecdn.com/analizadorelemental-130221210957-phpapp02/95/analizador-elemental-60-638.jpg?cb=1422562987](http://image.slidesharecdn.com/analizadorelemental-130221210957-phpapp02/95/analizador-elemental-60-638.jpg?cb=1422562987) )

- ✓ A carrier gas (in this case helium) carries the product gases  $\text{CO}_2$ ,  $\text{SO}_2$  and  $\text{NO}$  through a glass column packed with an oxidation catalyst of tungsten trioxide ( $\text{WO}_3$ ) and a copper reducer, both kept at  $1000^\circ\text{C}$ .
- ✓ At  $1000^\circ\text{C}$ , nitrogen oxide is reduced to  $\text{N}_2$ . The  $\text{N}_2$ ,  $\text{CO}_2$ , and  $\text{SO}_2$  are then transported by the helium to, and separated by, a 2m long packed column) and quantified with a TCD (set at  $290^\circ\text{C}$ .)
- ✓ The chromatographic response was calibrated against pre-established standards, and the CHNS elemental contents were reported in weight percent.

### 3.3.3 Calorific Value:

The calorific value of the coal is defined as the heat released by burning a unit mass of coal in presence of oxygen. The calorific value is a very important parameter used to characterize a fuel. In case of coal calorific value combined with other parameters is used to classify the coal or determine its rank. Higher calorific value indicates higher concentration of carbon and volatile

matter. High quantity of ash or mineral matter and moisture reduce the calorific value of coal. The unit used is Cal/g.

The standard procedure to determine the calorific value of coal is to use a bomb calorimeter. Both manual and digital bomb calorimeters are available. In this case a digital bomb calorimeter manufactured by “PARR” was used. It has the following components.

#### The Bomb

- It is made from stainless steel alloy.
- It carries arrangements inside to hold a crucible of coal and a connection of nicrome wire.
- It has got an air tight cover on top with two connection for electrodes and one for filling of oxygen

#### Calorimeter Vessel

- It is made of stainless steel.
- It can has a capacity two hold 2 litre of water and holds the bomb submerged in water.
- It also accomodates the magnetic stirrer during calorimetry operation and a thermocouple to measure the temperature

#### Electrode

- Two electrodes one positive and one negative are used
- They connect the bomb to the digital controller present in the controlling unit of the Bomb calorimeter
- They carry electricity through the coal via nicrome wire to create sparks

#### Magnetic Stirrer

- It is sturdily fixed to the outer cover of the Calorimeter.
- It stirs the water when in operation to disperse the temperature evenly

#### Digital Interface

- It is a touch sensitive screen
- It displays all the controls available to the user.
- It is used to program, start and stop the operation.

#### Digital Balance

- It is a sensitive electronic balance.
- It can directly transfer the read weight to the calorimeter electronically.

**Fig.3.6 Components of digital bomb calorimeter**



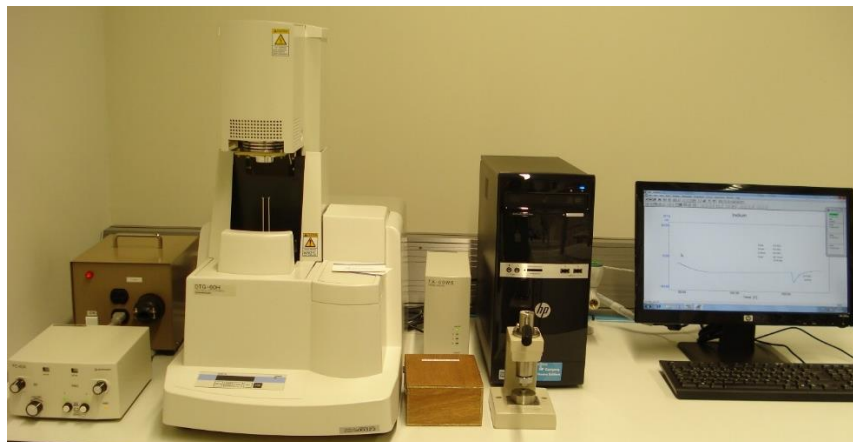
**Fig.3.6 Parr digital bomb calorimeter**

The following standardized procedure is followed to determine the calorific value of coal using the bomb calorimeter.

❖ **Procedure**

- ✓ The equipment is switched on.
- ✓ About 0.7 to 1g coal is weighed and transferred to the crucible or sample holder.
- ✓ A nicrome wire of proper length is cut and connected to the electrodes in the cover of the bomb.
- ✓ The wire is connected in such a way that it touches the surface of coal.
- ✓ The calorimeter vessel is filled with 2 liter cold water.
- ✓ 2 to 4ml of water is poured in the bomb before closing it by screwing the top.
- ✓ The bomb is filled with oxygen by connecting it to the gas filling valve.
- ✓ The flow is controlled from the interface.
- ✓ The water filled calorimeter vessel is placed inside the equipment.
- ✓ The Bomb is placed inside the calorimeter vessel using the holder.
- ✓ The external electrodes are connected to the bomb.
- ✓ The outer cover of the calorimeter is covered dipping the stirrer and thermocouple with it.
- ✓ The operation is started.
- ✓ The equipment sounds an alarm when the operation is completed.
- ✓ The gross calorific value is noted from the report in the interface.

### 3.3.4 Differential Thermal Analysis:-



**Fig.3.7 SHIMAZDU DTA equipment**

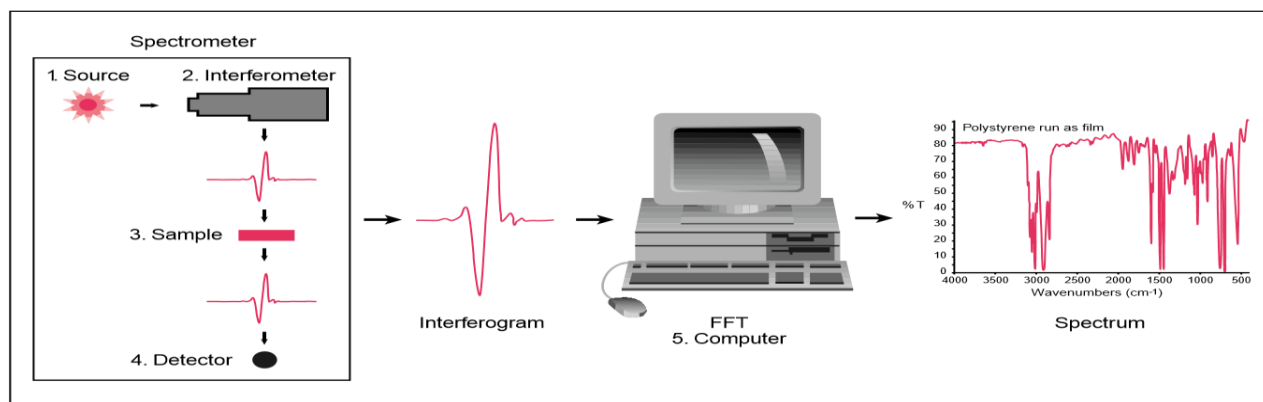
This is one of the thermal analysis methods to determine the susceptibility of coal of towards spontaneous heating. The method followed is as follows.

#### ❖ Procedure

- ✓ The equipment was turned on.
- ✓ The two crucibles were cleaned properly using a brush and forceps.
- ✓ About 67 to 100 mg of powdered alumina was weighed using a digital precision balance and poured in to one of the crucible.
- ✓ Approximately same amount of coal was weighed and transferred in to the other crucible
- ✓ The Top of the equipment was opened and the two crucibles were kept on the two ultrasensitive sample holder cum balance protruding from the bottom of the equipment
- ✓ The top was closed and the auto zero button was pressed to make the difference in weight between alumina and coal zero
- ✓ The top is opened again. The crucible containing coal is taken out using forceps
- ✓ An additional 10 mg of coal is weighed and added to the crucible.
- ✓ The coal crucible is kept in the equipment and the top is closed.
- ✓ The program is started and the sample details are entered.
- ✓ The upper limit of temperature is set at 450° C at a heating of 5° C/min.
- ✓ The weight reading is done clicking on 'read weight' and the experiment is started.
- ✓ After about 1.5 hour the plot between time, temperature and weight is obtained.
- ✓ The spontaneous heating propensity of coal is obtained by analyzing the data.

### 3.3.5 FT-IR:

FT-IR stands for Fourier Transform Infrared, the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. This makes infrared spectroscopy useful for several types of analysis. In case of coal FTIR spectroscopy can help us to determine its organic constituents [6].



**Fig.3.8 Principles of FTIR**

(Source: <http://mmrc.caltech.edu/FTIR/FTIRintro.pdf> )

The normal instrumental setup is as follows:

1. **The Source:** Infrared energy is emitted from a glowing black-body source. This beam passes through an aperture which controls the amount of energy presented to the sample (and, ultimately, to the detector).
2. **The Interferometer:** The beam enters the interferometer where the “spectral encoding” takes place. The resulting interferogram signal then exits the interferometer.
3. **The Sample:** The beam enters the sample compartment where it is transmitted through or reflected off of the surface of the sample, depending on the type of analysis being accomplished. This is where specific frequencies of energy, which are uniquely characteristic of the sample, are absorbed.

4. **The Detector:** The beam finally passes to the detector for final measurement. The detectors used are specially designed to measure the special interferogram signal.
5. **The Computer:** The measured signal is digitized and sent to the computer where the Fourier transformation takes place. The final infrared spectrum is then presented to the user for interpretation and any further manipulation.



**Fig.3.9 PerkinElmer FTIR spectrometer**

(Source: <http://www.nsc.gpi.ru/DLISP/DML/Perkinelmer.jpg> )

❖ **Procedure**

- ✓ About 50 g of KBr was kept in the moisture oven at 108 C for about 3 hours to remove moisture
- ✓ About 10 mg KBr was ground using mortar and pestle for 5 minutes.
- ✓ The KBr was made in to a pellet first by putting it in a pelletizer then keeping it under a force of 10 kN in the manual pneumatic press.
- ✓ The KBr pellet was carefully removed from the pelletizer on to a paper using a razor blade.
- ✓ About 1mg of 212 micron coal was taken and mixed with 10 mg of the pulverized KBr, keeping a ratio of 1:10, prepared earlier.


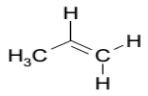
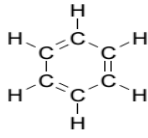
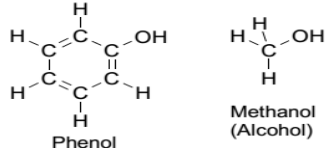
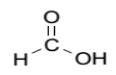
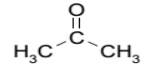
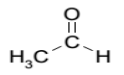


**Fig.3.10 Pellet preparation before FTIR**

- ✓ The mixture ground to even finer size for about 5 minutes
- ✓ The procedure followed to make the KBr pellet was followed for the Coal + KBr pellet
- ✓ The KBr pellet was scanned first as background followed by the Coal + KBr pellets in a PerkinElmer FT-IR spectrometer
- ✓ The peaks were obtained from the software and analyzed to determine organic constituents.

Table.3.3 was used to identify organic compounds present in coals from FTIR spectra.

**Table 3.3 FTIR absorption range for various organic compounds [2]**

| <b>Functional Group Names</b><br>&<br><b>Example compounds</b>   | <b>Absorption Ranges(<math>\text{cm}^{-1}</math>)</b><br>[Look for a single absorption<br>in these regions, unless stated otherwise.] | <b>Type of Vibration</b><br>causing IR absorption  |
|--|---|--|
| <b>Alkanes:</b><br> Methane   | <b>3000-2800</b><br>(Note: The absorptions can be seen as several distinct peaks in this region.)                                     | H-C-H Asymmetric & Symmetric Stretch   |
|  | <b>1500-1440</b>  | H-C-H Bend   |
| <b>Alkenes:</b><br> 1-Propene                                       | <b>3100-3000</b>  | C=C-H Asymmetric Stretch   |
|  | <b>1675-1600</b>  | C-C=C Symmetric Stretch  |
| <b>Alkynes:</b><br>$\text{HC}\equiv\text{C}-\text{CH}_3$ Propyne   | <b>3300-3200</b>  | $\equiv\text{C}-\text{H}$ Stretch  |
|  | <b>2200-2100</b>  | $\text{C}\equiv\text{C}$ Stretch   |
| <b>Aromatic Rings:</b><br> Benzene                                  | <b>3100-3000</b>  | C=C-H Asymmetric Stretch   |
|  | <b>1600-1580</b>  | C-C=C Symmetric Stretch  |
|  | <b>1500-1450</b>  | C-C=C Asymmetric Stretch   |
| <b>Phenols &amp; Alcohols:</b><br> Phenol      Methanol (Alcohol) | <b>3600-3100</b><br><br>(Note: Phenols <u>MUST</u> have Aromatic Ring Absorptions too.)   | Hydrogen-bonded O-H Stretch<br><br>(This peak usually appears much broader than the other IR absorptions.) |
| <b>Carboxylic Acids:</b><br> Formic Acid                          | <b>3400-2400</b><br>(This peak always covers the entire region with a <b>VERY BROAD</b> peak.)  | Hydrogen-bonded O-H Stretch<br>[Note: This peak can obscure other peaks in this region.]                   |
|  | <b>1730-1650</b>  | C=O Stretch  |
| <b>Ketones:</b><br> Acetone                                       | <b>1750-1625</b>  | C=O Stretch  |
| <b>Aldehydes:</b><br> Ethanal                                     | <b>1750-1625</b>  | C=O Stretch  |
|  | <b>2850-2800</b>  | C-H Stretch off C=O  |
|  | <b>2750-2700</b>  | C-H Stretch off C=O  |



CHAPTER 4  
RESULTS AND DISCUSSIONS

## 4.1 OBSERVATIONS:

### 4.1.1 Proximate Analysis

Table 4.1 Moisture, ash, volatile matter and fixed carbon content of CCL coals

| Sample No. | %M       | %ASH     | %VM      | %FC<br>(100-[%M+%ASH+%VM]) |
|------------|----------|----------|----------|----------------------------|
| Argada 1   | 9.488252 | 18.42501 | 21.13524 | 50.9515                    |
| Argada 2   | 9.60185  | 18.80291 | 20.07002 | 51.52522                   |
| Kedla 1    | 0.911915 | 45.04153 | 16.20878 | 37.83777                   |
| Kedla 2    | 1.612903 | 21.66869 | 29.53838 | 47.18002                   |
| Pindra 5   | 2.557187 | 20.72009 | 29.03124 | 47.69148                   |
| Pindra 6   | 2.915247 | 10.82    | 26.03028 | 60.23447                   |
| Pindra 7B  | 1.113431 | 11.73038 | 31.93318 | 55.22301                   |
| Pindra 7C  | 2.172164 | 26.68672 | 26.27922 | 44.8619                    |
| Pindra 8   | 2.082495 | 25.50837 | 29.11062 | 43.29851                   |
| Pundi 4    | 1.939719 | 34.40152 | 24.80493 | 38.85383                   |
| Pundi 7    | 2.111478 | 5.877061 | 38.75218 | 53.25928                   |
| Sarubera 1 | 6.767444 | 24.04255 | 10.79149 | 58.39852                   |
| Sarubera 2 | 6.876445 | 27.3656  | 25.77845 | 39.9795                    |
| Tapeen 8A  | 2.199458 | 21.79487 | 23.35342 | 52.65225                   |
| Tapeen 8B  | 2.047371 | 12.09368 | 25.73371 | 60.12524                   |
| Tapeen 8C  | 1.496183 | 10.69042 | 28.74662 | 59.06677                   |
| Topa 4     | 2.407833 | 50.6217  | 19.41685 | 27.55361                   |
| Topa 5     | 2.201226 | 19.70105 | 28.37844 | 49.71929                   |
| Topa 6     | 8.390701 | 14.22262 | 28.06344 | 49.32323                   |
| Topa 7     | 2.029043 | 20.668   | 28.04924 | 49.25372                   |
| Topa 8     | 4.585873 | 7.265774 | 30.93451 | 57.21384                   |

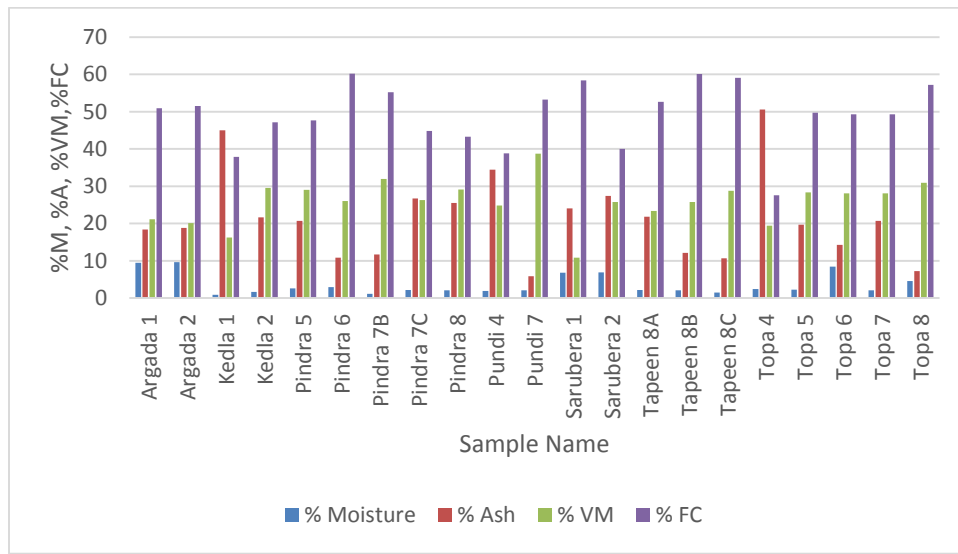
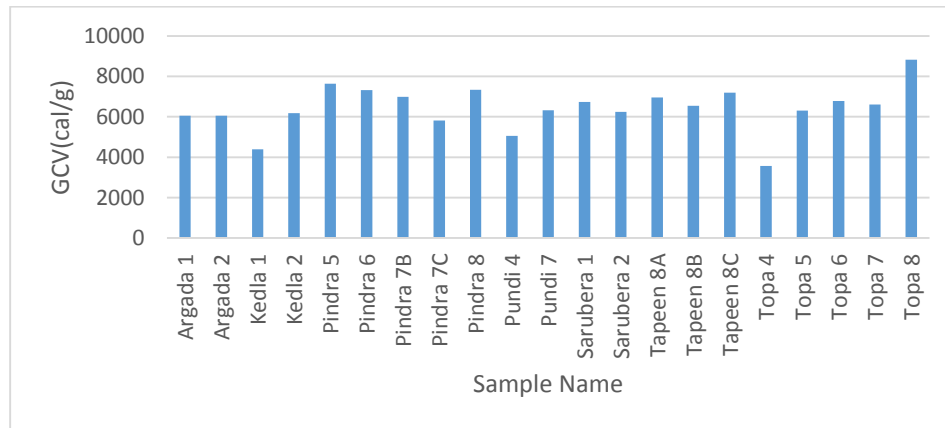


Fig. 4.1 Variation of moisture, ash, volatile matter and fixed carbon content of CCL coals

#### 4.1.2 Gross Calorific Value

**Table 4.2 Gross calorific value of CCL coals**

| Sample No. | GCV(in Cal/g) |
|------------|---------------|
| Argada 1   | 6052.428      |
| Argada 2   | 6057.646      |
| Kedla 1    | 4384.653      |
| Kedla 2    | 6185.189      |
| Pindra 5   | 7644.707      |
| Pindra 6   | 7322.785      |
| Pindra 7B  | 6985.542      |
| Pindra 7C  | 5820.249      |
| Pindra 8   | 7340.788      |
| Pundi 4    | 5061.968      |
| Pundi 7    | 6323.887      |
| Sarubera 1 | 6741.796      |
| Sarubera 2 | 6237.85       |
| Tapeen 8A  | 6951.634      |
| Tapeen 8B  | 6551.211      |
| Tapeen 8C  | 7203.419      |
| Topa 4     | 3571.334      |
| Topa 5     | 6312.924      |
| Topa 6     | 6787.24       |
| Topa 7     | 6613.645      |
| Topa 8     | 8821.638      |



**Fig. 4.2 Variation of gross calorific value of CCL coals**

### 4.1.3 CHNS Analysis

Table 4.3 Carbon, hydrogen, nitrogen and sulphur content of CCL coals

| SAMPLE NO. | C %    | H%    | N%    | S%    |
|------------|--------|-------|-------|-------|
| Argada 1   | 61.666 | 3.47  | 4.794 | 0.386 |
| Argada 2   | 61.049 | 3.537 | 4.346 | 0.408 |
| Kedla 1    | 42.867 | 2.367 | 3.43  | 0.461 |
| Kedla 2    | 59.966 | 3.626 | 4.365 | 0.767 |
| Pindra 5   | 60.429 | 3.687 | 3.577 | 0.608 |
| Pindra 6   | 70.559 | 3.475 | 4.034 | 0.543 |
| Pindra 7B  | 68.621 | 3.936 | 3.985 | 0.797 |
| Pindra 7C  | 58.289 | 3.408 | 4.319 | 0.691 |
| Pindra 8   | 71.842 | 4.131 | 4.269 | 0.841 |
| Pundi 4    | 49.287 | 2.669 | 4.443 | 0.582 |
| Pundi 7    | 58.741 | 3.864 | 4.062 | 0.432 |
| Sarubera 1 | 66.629 | 3.12  | 4.596 | 0.555 |
| Sarubera 2 | 61.217 | 2.911 | 3.566 | 0.471 |
| Tapeen 8A  | 62.737 | 3.285 | 3.698 | 0.569 |
| Tapeen 8B  | 69.75  | 3.683 | 3.799 | 0.774 |
| Tapeen 8C  | 70.537 | 3.864 | 4.048 | 0.687 |
| Topa 4     | 35.436 | 2.414 | 3.919 | 0.508 |
| Topa 5     | 61.221 | 3.621 | 3.789 | 0.693 |
| Topa 6     | 70.859 | 4.435 | 4.266 | 0.649 |
| Topa 7     | 58.054 | 3.287 | 3.44  | 0.587 |
| Topa 8     | 68.114 | 4.191 | 3.87  | 0.862 |

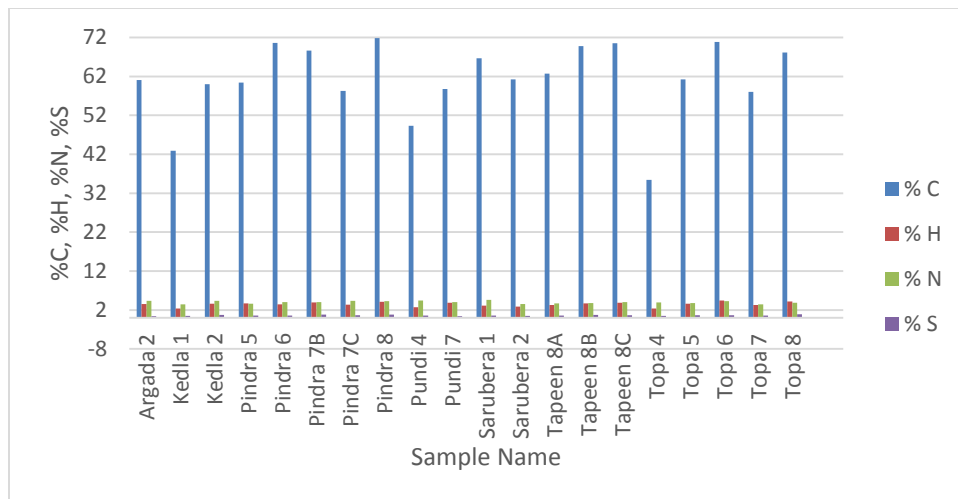


Fig. 4.3 Variation of carbon, hydrogen, nitrogen and sulphur content of CCL coals

#### 4.1.4 FTIR

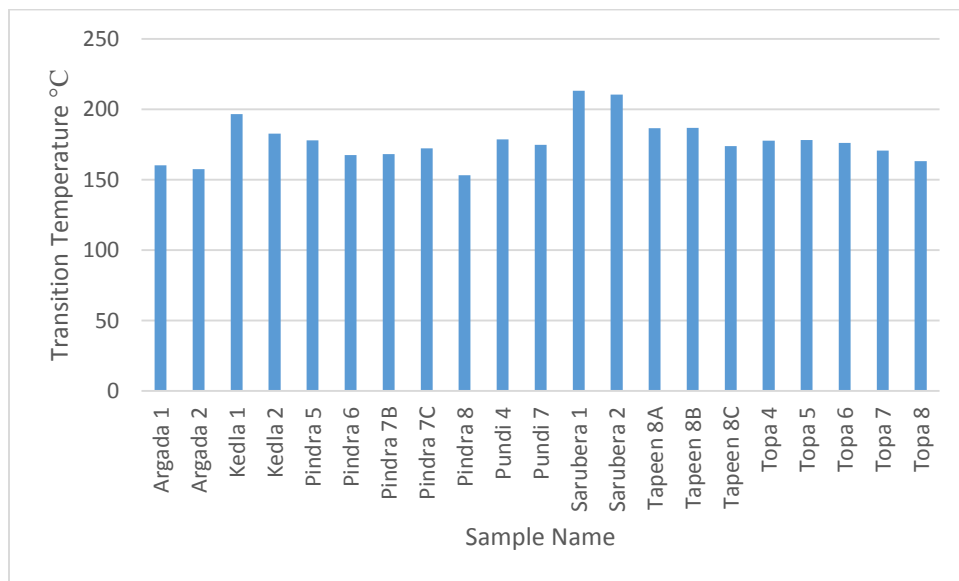
**Table 4.4 Organic compounds present in CCL coals [2] [21]**

| SAMPLE NAME | ALKANE | ALKENE | ALKYNE | AROMATIC RINGS | PHENOL AND ALCOHOL | CARBOXYLIC ACIDS | KETONES | ALDEHYDES |
|-------------|--------|--------|--------|----------------|--------------------|------------------|---------|-----------|
| Argada 1    | Yes    | Yes    | No     | No             | No                 | No               | No      | No        |
| Argada 2    | Yes    | Yes    | No     | No             | No                 | Yes              | Yes     | No        |
| Kedla 1     | Yes    | No     | No     | No             | No                 | No               | No      | No        |
| Kedla 2     | Yes    | Yes    | No     | No             | Yes                | No               | No      | No        |
| Pindra 5    | Yes    | No     | No     | No             | No                 | No               | No      | No        |
| Pindra 6    | Yes    | No     | No     | No             | No                 | Yes              | Yes     | No        |
| Pindra 7B   | Yes    | Yes    | No     | No             | No                 | No               | No      | No        |
| Pindra 7C   | Yes    | No     | No     | No             | No                 | Yes              | Yes     | No        |
| Pindra 8    | Yes    | No     | No     | No             | No                 | Yes              | Yes     | No        |
| Pundi 4     | Yes    | No     | No     | No             | No                 | No               | No      | No        |
| Pundi 7     | Yes    | No     | No     | Yes            | No                 | No               | No      | No        |
| Sarubera 1  | Yes    | No     | No     | No             | No                 | No               | No      | No        |
| Sarubera 2  | Yes    | Yes    | No     | No             | No                 | No               | No      | No        |
| Tapeen 8A   | Yes    | Yes    | No     | No             | No                 | Yes              | Yes     | No        |
| Tapeen 8B   | Yes    | Yes    | No     | No             | No                 | Yes              | Yes     | No        |
| Tapeen 8C   | Yes    | Yes    | No     | No             | No                 | No               | No      | No        |
| Topa 4      | Yes    | No     | No     | No             | No                 | No               | No      | No        |
| Topa 5      | Yes    | No     | No     | No             | No                 | No               | No      | No        |
| Topa 6      | Yes    | Yes    | No     | No             | No                 | No               | No      | No        |
| Topa 7      | Yes    | No     | No     | No             | No                 | No               | Yes     | No        |
| Topa 8      | Yes    | Yes    | No     | No             | No                 | No               | No      | No        |

#### 4.1.5 Differential Thermal Analysis

**Table 4.5 Transition temperature of CCL coals**

| Sample No.        | Transition Temperature(DTA) ° C |
|-------------------|---------------------------------|
| <b>Argada 1</b>   | 160.18                          |
| <b>Argada 2</b>   | 157.43                          |
| <b>Kedla 1</b>    | 196.66                          |
| <b>Kedla 2</b>    | 182.65                          |
| <b>Pindra 5</b>   | 178.02                          |
| <b>Pindra 6</b>   | 167.51                          |
| <b>Pindra 7B</b>  | 168.1                           |
| <b>Pindra 7C</b>  | 172.36                          |
| <b>Pindra 8</b>   | 153.09                          |
| <b>Pundi 4</b>    | 178.63                          |
| <b>Pundi 7</b>    | 174.76                          |
| <b>Sarubera 1</b> | 213.28                          |
| <b>Sarubera 2</b> | 210.36                          |
| <b>Tapeen 8A</b>  | 186.67                          |
| <b>Tapeen 8B</b>  | 186.81                          |
| <b>Tapeen 8C</b>  | 173.85                          |
| <b>Topa 4</b>     | 177.62                          |
| <b>Topa 5</b>     | 178.23                          |
| <b>Topa 6</b>     | 176.06                          |
| <b>Topa 7</b>     | 170.59                          |
| <b>Topa 8</b>     | 163.12                          |



**Fig. 4.4 Variation of transition temperature (DTA) of CCL coals**

## 4.2 DISCUSSIONS:


The transition temperature obtained during DTA can be used as an indicator of spontaneous heating susceptibility of coal. According to previous investigations the spontaneous heating susceptibility of coal has an inverse relationship with transition temperature.

$$\text{Spontaneous Heating Susceptibility} \propto \frac{1}{T}$$

Where, T= Transition Temperature obtained from DTA

So, the lower the transition temperature more susceptible the coal to spontaneous heating [20]. Table. 4.9 ranks the coals according to their spontaneous heating susceptibility.

**Table 4.6 Ranking of CCL coals according to spontaneous heating liability [20]**

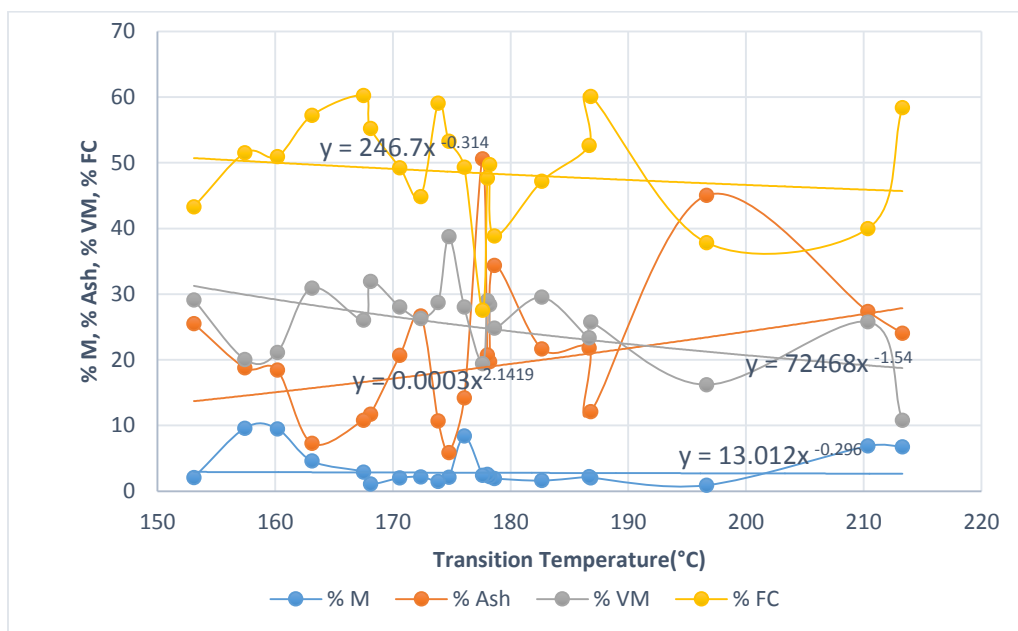
| Sample Name | Transition Temperature (C) | Highest Susceptibility to Spontaneous Heating  |  |
|-------------|----------------------------|--|--|
| Pindra 8    | 153.09                     |  |  |
| Argada 2    | 157.43                     |  |  |
| Argada 1    | 160.18                     |  |  |
| Topa 8      | 163.12                     |  |  |
| Pindra 6    | 167.51                     |  |  |
| Pindra 7B   | 168.1                      |  |  |
| Topa 7      | 170.59                     |  |  |
| Pindra 7C   | 172.36                     |  |  |
| Tapeen 8C   | 173.85                     |  |  |
| Pundi 7     | 174.76                     |  |  |
| Topa 6      | 176.06                     |  |  |
| Topa 4      | 177.62                     |  |  |
| Pindra 5    | 178.02                     |  |  |
| Topa 5      | 178.23                     |  |  |
| Pundi 4     | 178.63                     |  |  |
| Kedla 2     | 182.65                     |  |  |
| Tapeen 8A   | 186.67                     |  |  |
| Tapeen 8B   | 186.81                     |  |  |
| Kedla 1     | 196.66                     |  | Lowest Susceptibility to Spontaneous Heating |
| Sarubera 2  | 210.36                     |  |  |
| Sarubera 1  | 213.28                     |  |  |

Different organic functional groups has been identified from the FTIR spectra.

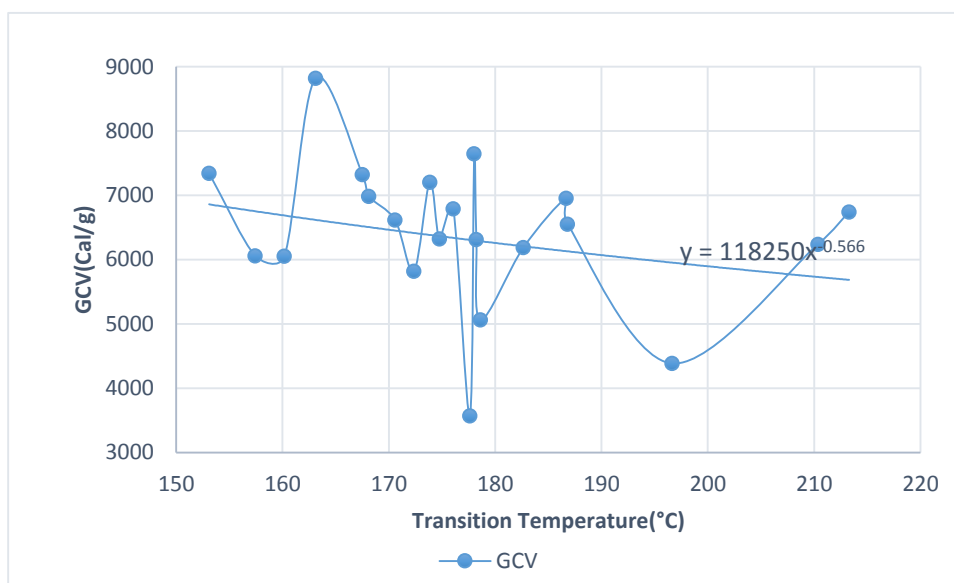
- ✓ Alkanes were found in all the coal samples.
- ✓ Alkenes were present in all samples except in some samples of Kedla, Pindra and Topa mines

- ✓ No alkynes were observed
- ✓ There was one occurrence of each of aromatic ring and alcohol in Pundi 7 and Kedla 2 samples respectively.
- ✓ Six of the samples contained carboxylic acid and ketone groups from which 3 samples had the lowest values of transition temperature.

The plots between transition temperature and different intrinsic parameters of coal were obtained using Excel 2013.

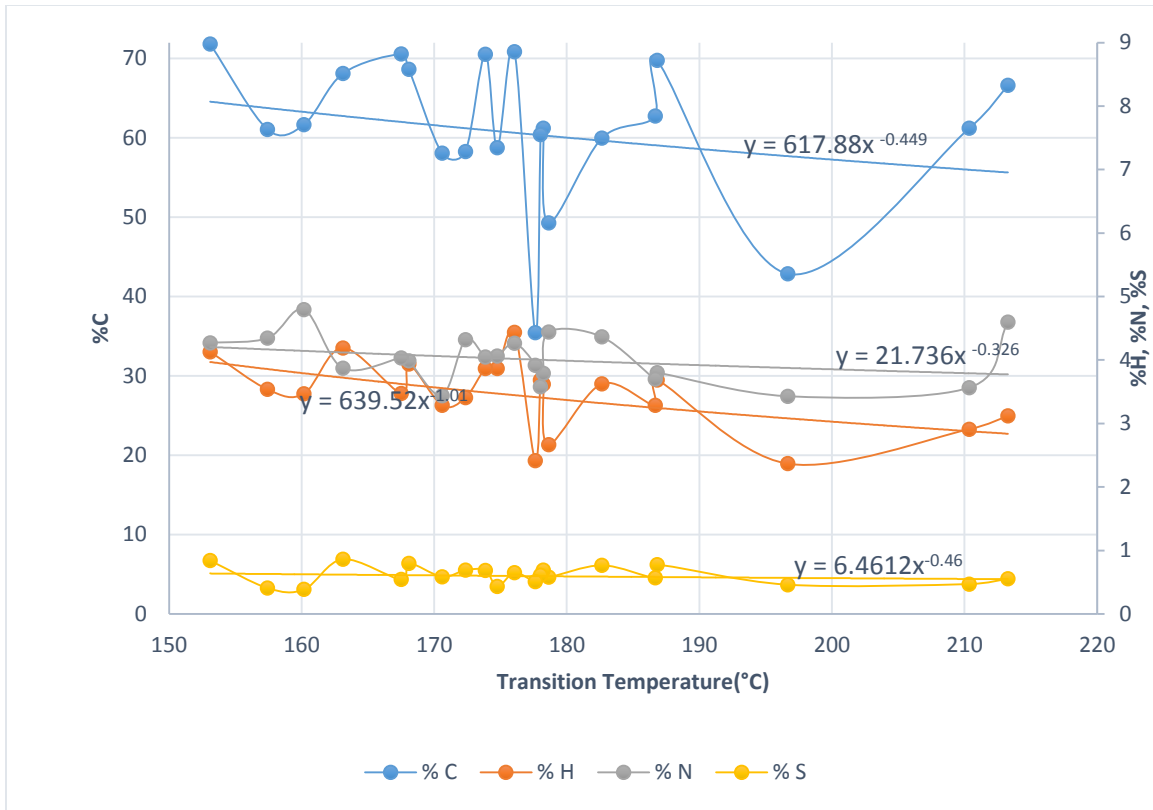


**Fig. 4.5 Transition temperature & M, A, VM, FC**



**Fig. 4.6 Transition temperature vs GCV**





**Fig. 4.7 Transition temperature & C, H, N, S**

The trend line equations obtained from these plots are summarized in Table 4.10.

**Table 4.7 Trend line equations**

| Variable        | x                      | y   | Trend line Equation               |
|-----------------|------------------------|-----|-----------------------------------|
| Proximate       | Transition Temperature | M   | $y = 13.012x^{-0.296}$            |
|                 |                        | A   | $y = 0.0003x^{2.1419}$            |
|                 |                        | VM  | $y = 72468x^{-1.54}$              |
|                 |                        | FC  | $y = 246.7x^{-0.314}$             |
| Calorific Value | Transition Temperature | GCV | $y = 11.83x^{-0.566} \times 10^4$ |
| CHNS            | Transition Temperature | C   | $y = 617.88x^{-0.449}$            |
|                 |                        | H   | $y = 639.52x^{-1.01}$             |
|                 |                        | N   | $y = 21.736x^{-0.326}$            |
|                 |                        | S   | $y = 6.4612x^{-0.46}$             |

### 4.2.1 Multiple Regression Analysis [7] [8] [9]

Multiple regression analysis was done with transition temperature as dependent variable and the others as independent variable as shown in Table 4.11.

Regression analysis is used to produce an equation that will predict a dependent variable using one or more independent variables. This equation has the form

$$Y = b_1X_1 + b_2X_2 + \dots + a$$

Where, Y is the dependent variable you are trying to predict, X<sub>1</sub>, X<sub>2</sub> and so on are the independent variables you are using to predict it, b<sub>1</sub>, b<sub>2</sub> and so on are the coefficients or multipliers that describe the size of the effect the independent variables are having on your dependent variable Y, and a is the value Y is predicted to have when all the independent variables are equal to zero.

**Table 4.8 Variables used in multiple regression**

| Dependent Variable                            |    | Independent Variable |                                     |
|---|----|----------------------|-------------------------------------|
| Transition Temperature (°C) obtained from DTA | Vs | Proximate            | % Moisture                          |
|   |    |                      | % Ash                               |
|   |    |                      | % Volatile Matter                   |
|   |    |                      | % Fixed Carbon                      |
|   |    | Calorific Value      | Gross Calorific Value (GCV) (cal/g) |
|   |    | CHNS                 | % Carbon                            |
|   |    |                      | % Hydrogen                          |
|   |    |                      | % Nitrogen                          |
|   |    |                      | % Sulphur                           |

The various components of the result is explained below in brief.

#### 4.2.1.1 Regression Statistics:

These are the “Goodness of Fit” measures. They tell you how well the calculated linear equation fits your data.

- ✓ **Multiple R:** This is the correlation coefficient. It tells you how strong the linear relationship is. For example, a value of 1 means a perfect positive relationship and a value of zero means no relationship at all. It is the square root of R squared
- ✓ **R square:** This is R, the Coefficient of Determination. It tells you how many points fall on the regression line. For example, 80% means that 80% of the variation of y-values around the mean are explained by the x-values. In other words, 80% of the values fit the model.
- ✓ **Adjusted R square:** The adjusted R-squared adjust for the number of terms in a model. You'll want to use this if you have more than one x variable.
- ✓ **Standard Error of the regression:** An estimate of the standard deviation of the error  $\mu$ . This is not the same as the standard error in descriptive statistics! The standard error of the regression is the precision that the regression coefficient is measured; if the coefficient is large compared to the standard error, then the coefficient is probably different from 0.
- ✓ **Observations:** Number of observations in the sample.

**Table 4.9 Regression statistics**

| Statistical Parameters | Proximate | GCV     | CHNS    |
|------------------------|-----------|---------|---------|
| Multiple R             | 0.9973    | 0.9786  | 0.9918  |
| R Square               | 0.9946    | 0.9576  | 0.9838  |
| Adjusted R Square      | 0.9348    | 0.9076  | 0.9221  |
| Standard Error         | 14.6      | 37.5486 | 25.2250 |
| Observations           | 21        | 21      | 21      |

#### 4.2.1.2 Regression Coefficients

This section of the table gives you very specific information about the components you chose to put into your data analysis.

The columns are:

- ✓ Coefficient: Gives you the least squares estimate.
- ✓ Standard Error: the least squares estimate of the standard error.

- ✓ T Statistic: The T Statistic for the null hypothesis vs. the alternate hypothesis.
- ✓ P Value: Gives you the p-value for the hypothesis test.

**Table 4.10 Regression coefficients**

| Variable           | Empirical equation coefficients |         | Standard Error | t Stat | P-value  |
|--------------------|---------------------------------|---------|----------------|--------|----------|
| Proximate analysis | %M                              | 1.022   | 1.225          | 0.835  | 0.416    |
|                    | %ASH                            | 2.149   | 0.229          | 9.394  | 3.84E-08 |
|                    | %VM                             | 0.817   | 0.528          | 1.548  | 0.14     |
|                    | %FC                             | 2.169   | 0.314          | 6.913  | 2.51E-06 |
| Calorific value    | GCV                             | 0.026   | 0.001          | 21.266 | 3.34E-15 |
| CHNS analysis      | % C                             | 1.548   | 1.048          | 1.477  | 0.158    |
|                    | % H                             | -27.824 | 19.155         | -1.453 | 0.165    |
|                    | % N                             | 37.634  | 8.921          | 4.219  | 0.0006   |
|                    | % S                             | 42.338  | 47.177         | 0.897  | 0.382    |

Empirical equations were derived to predict the value of transition temperature by using the coefficients from Table 4.13. These equations are listed in Table 4.14.

**Table 4.11 Empirical equations**

| Variables              |                    | Equation                                   |
|------------------------|--------------------|--|
| Independent Variable   | Dependent Variable |  |
| Transition Temperature | M, A, VM, FC       | $Tr = 1.02 M + 2.15 A + 0.82 VM + 2.17 FC$ |
|                        | GCV                | $Tr = 0.03GCV$                             |
|                        | C, H, N, S         | $Tr = 1.55 C - 27.82 H + 37.63 N + 42.34S$ |

## SUMMARY & CONCLUSIONS

- ❖ From the proximate analysis done on CCL coal samples the following conclusions were derived:
  - ✓ The minimum percentage of moisture content was found in sample 'Kedla 1' with a value of 0.91 %. The maximum percentage was recorded in case of sample 'Argada 2' with a value of 9.6 %.
  - ✓ The lowest quantity of ash content was 5.9 % obtained in case of sample 'Pundi 7'. The highest percentage of ash was present in the sample 'Topa 4' with a value of 50.6 %.
  - ✓ The minimum percentage of volatile matter was recorded in case of sample 'Sarubera 1' with a value of 10.8 % and the maximum value was found in case of 'Pundi 7' at 38.8 %.
  - ✓ Percentage of fixed carbon was found to be lowest at 27.6 % in case of sample 'Topa 4' and the highest value was obtained to be 60.2% for sample 'Pindra 6'.
- ❖ Calorific testing on CCL coal samples provide following conclusions:
  - ✓ The smallest figure for gross calorific value was 3571.3 Cal/g for sample 'Topa 4'. The highest figure for gross calorific value happened to be 8821.6 Cal/g in case of sample 'Topa 8'.
  - ✓ These figures are supported by proximate analysis as well. Because, at 50.6 % 'Topa 4' has highest percentage of ash content among the samples with 19.4 % volatile matter content and the lowest percentage of fixed carbon with a value of only 27.6 %. Similarly, 'Topa 8' has only 7.3% ash content, 30.9 % volatile matter and 57.2 % fixed carbon content.
  - ✓ So, the maximum gross calorific value in case of 'Topa 8' can be attributed to higher volatile matter, fixed carbon content and lower ash content. The opposite is true in case of 'Topa 4'.
- ❖ CHNS analysis of coal gave following conclusions:
  - ✓ The lowest and highest percentage of carbon content were found to be 35.4% in case of 'Topa 4' and 71.8 % in case of 'Pindra 8'.
  - ✓ It was observed that in all the coal samples the percentage of carbon content is higher than the percentage of fixed carbon content. This is because the carbon content obtained from CHNS analysis also includes carbon which is not available for oxidation and does not take part in combustion e.g. carbon present in carbonate minerals.
  - ✓ The minimum and maximum percentage of hydrogen content were 2.4 % in case of 'Kedla 1' and 4.4 % in case of 'Topa 6'.

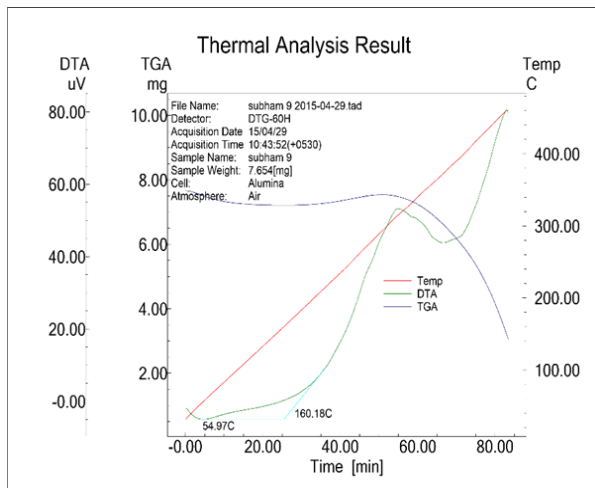
- ✓ The lowest value for nitrogen was 3.4 % in case of 'Kedla 1' and the highest value was 4.8 % in case of 'Argada 1'.
- ✓ Percentage of sulphur content was found to be minimum at 0.4 % in case of 'Argada 1' and maximum at 0.9 % in case of 'Topa 8'.
- ❖ The following conclusions were inferred from FTIR study of CCL coals.
  - ✓ Alkanes were the most prevalent organic compounds found to be present in all of the coal samples.
  - ✓ Only 10 samples indicated presence of alkanes.
  - ✓ Alcohol was observed in 'Kedla 2' and aromatic ring was found in case of 'Pundi 7' samples.
  - ✓ Six samples indicated presence of carboxylic acid and ketone groups including 'Argada 2', 'Pindra 6', 'Pindra 7C', 'Pindra 8', 'Tapeen 8A' and 'Tapeen 8B'.
- ❖ The following conclusions were adopted from the observation of differential thermal analysis:
  - ✓ The lowest value of transition temperature was 153.09° C in case of sample 'Pindra 8' and the highest value of transition temperature 213.28° C in case of sample 'Sarubera 1'.
  - ✓ The transition temperature value suggests that among the CCL coal samples 'Pindra 8' is most liable to spontaneous heating and 'Sarubera 1' is least liable to spontaneous heating.
  - ✓ The observation made during proximate and ultimate analysis supports this claim.
  - ✓ The sample 'Pindra 8' has 25.5 % ash content, 29.1 % volatile matter content and 43.3 % fixed carbon content. Additionally from CHNS analysis 'Pindra 8' has the maximum percentage of sulphur content with a value of 0.84 %.
  - ✓ Similarly, the sample 'Sarubera 1' has 24.04 % ash content, the lowest percentage of volatile matter content with a value of 10.8 % and fixed carbon content of 58.4 %. The sulphur content in this case was 0.55 %.
  - ✓ The ash content of both the samples are nearly equal, so the lower value of transition temperature in case of 'Pindra 8' can be attributed to a relatively higher volatile matter and sulphur content with a relatively lower fixed carbon content which also happen to be the conditions which make coal more liable to spontaneous heating. So, the observation confirms with the established theory.
  - ✓ It was observed that among the six samples that contained carboxylic acid and ketones 'Pindra 8', 'Argada 2', 'Pindra 7C' had relatively lower transition temperature 153.09° C, 157.43° C and 172.36° C respectively. Carboxylic acids and ketones contain active '-C=O' functional groups which react more readily. Thus their presence may make the coal more liable to spontaneous heating.

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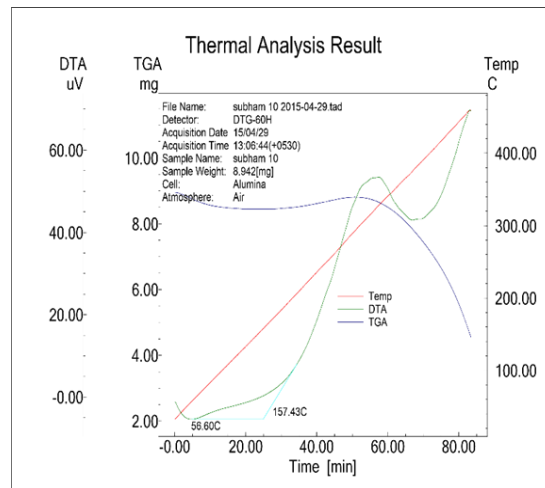
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**APPENDIX-1**  
**DTA THERMOGRAM & FT-IR SPECTRA**

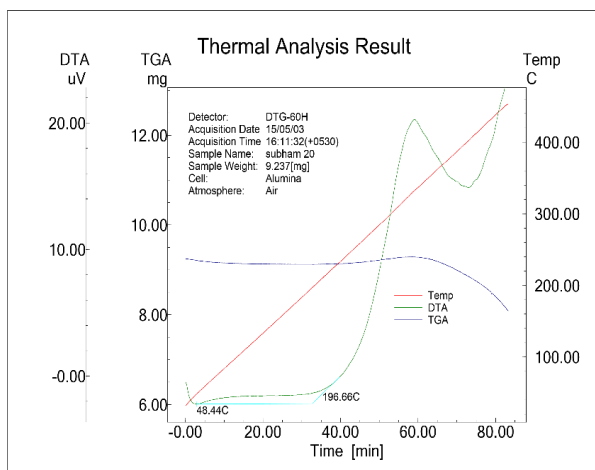




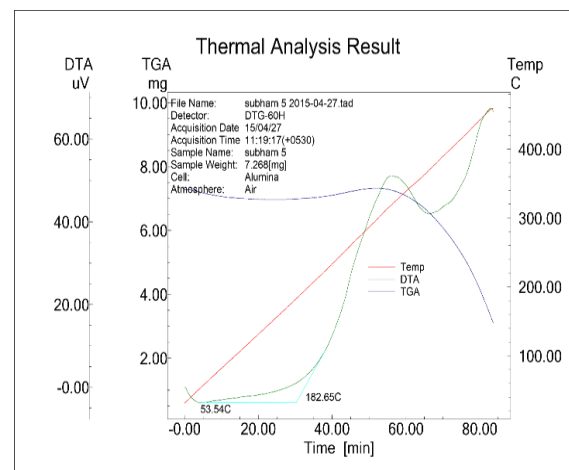
**Fig.5.1. DTA thermogram Argada 1**



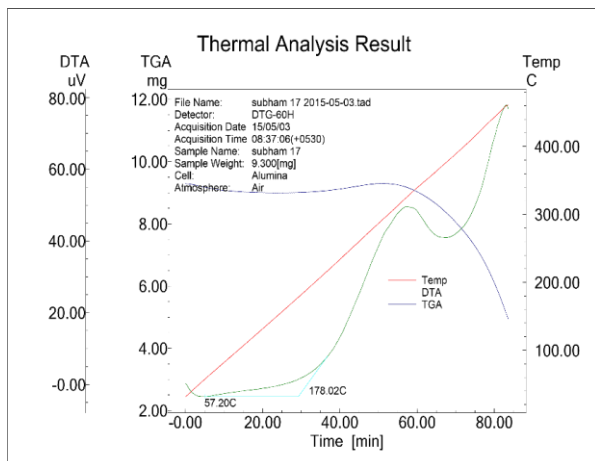
**Fig.5.2. DTA thermogram Argada 2**



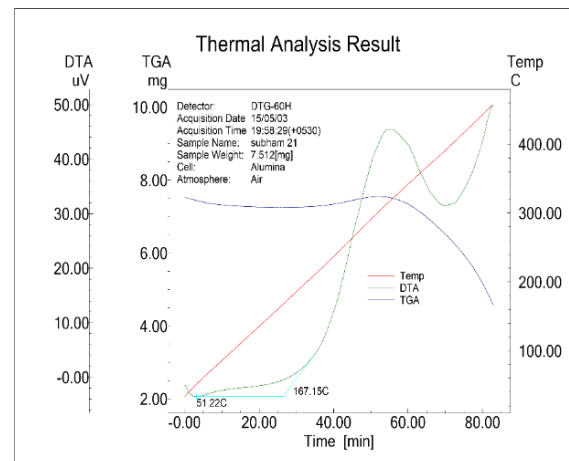
**Fig.5.3 DTA thermogram Kedla 1**



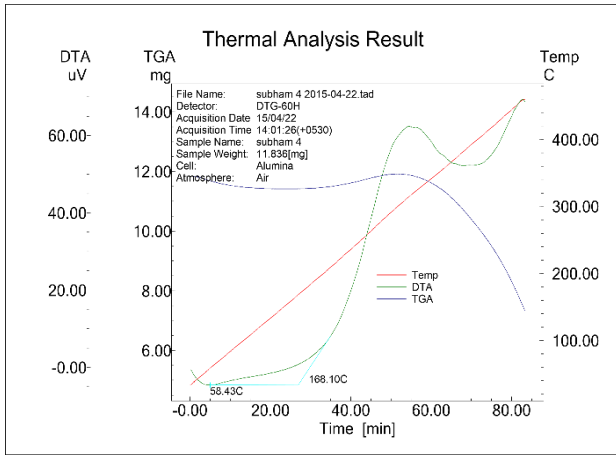
**Fig.5.4 DTA thermogram Kedla 2**



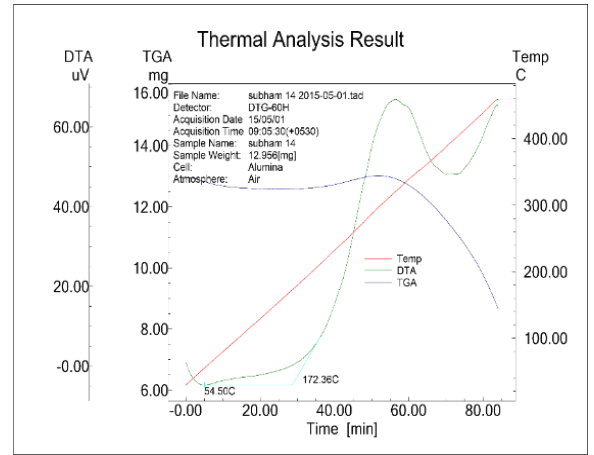
**Fig.5.5 DTA thermogram Pindra 5**



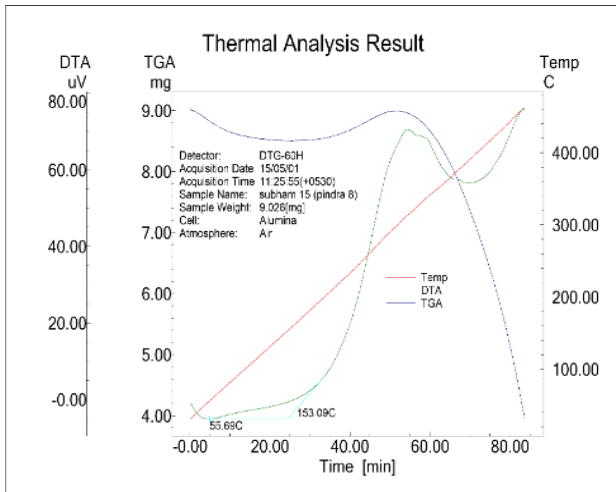
**Fig.5.6 DTA thermogram Pindra 6**



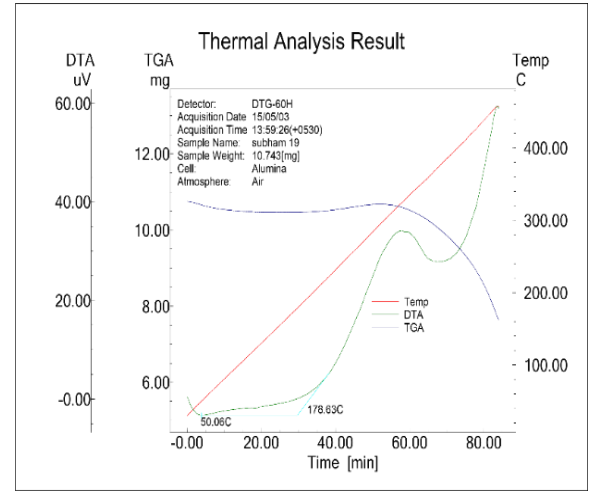
**Fig.5.7 DTA thermogram Pindra 7B**



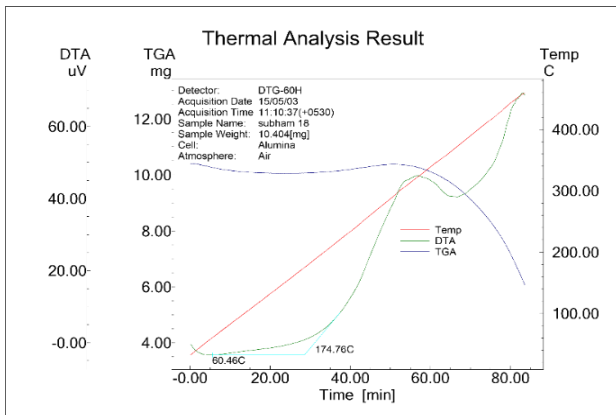
**Fig.5.8. DTA thermogram Pindra 7C**



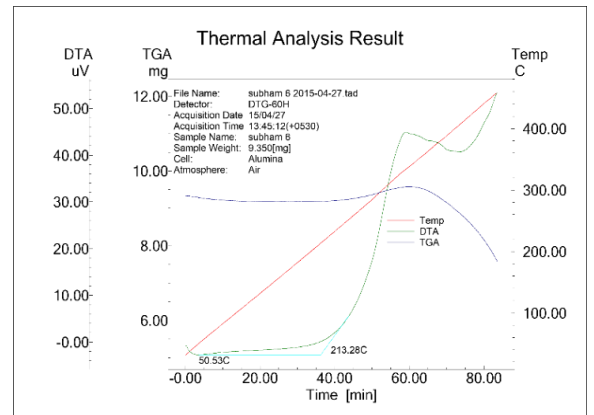
**Fig.5.9 DTA thermogram Pindra 8**



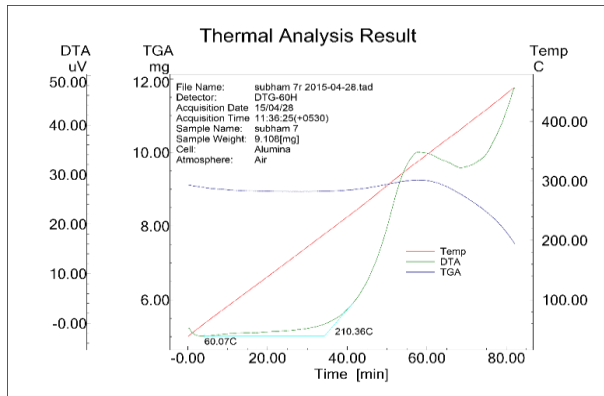
**Fig.5.10 DTA thermogram Pundi 4**



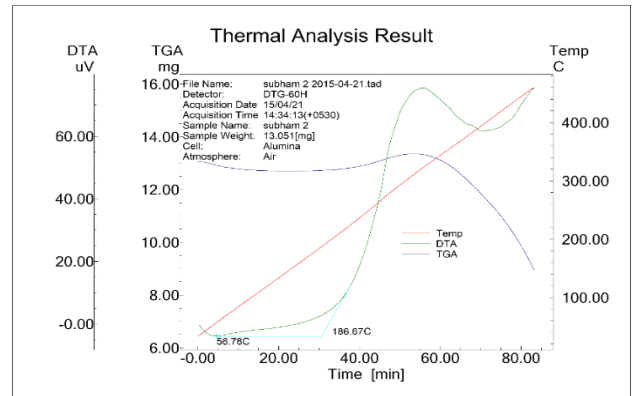
**Fig.5.11 DTA thermogram Pundi 7**



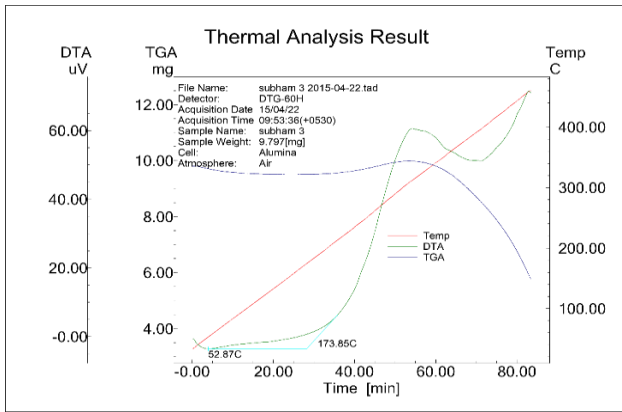
**Fig.5.12 DTA thermogram Sarubera 1**



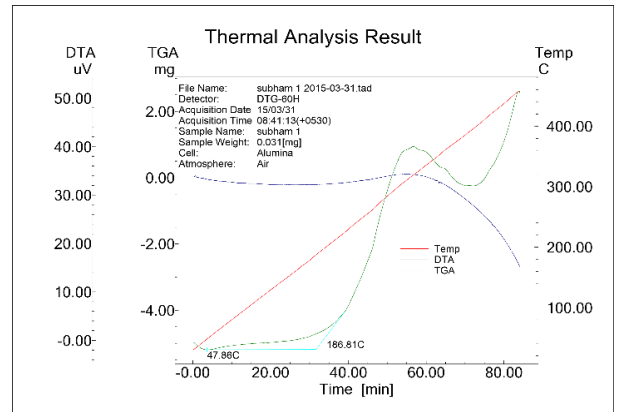
**Fig.5.13 DTA thermogram Sarubera 2**



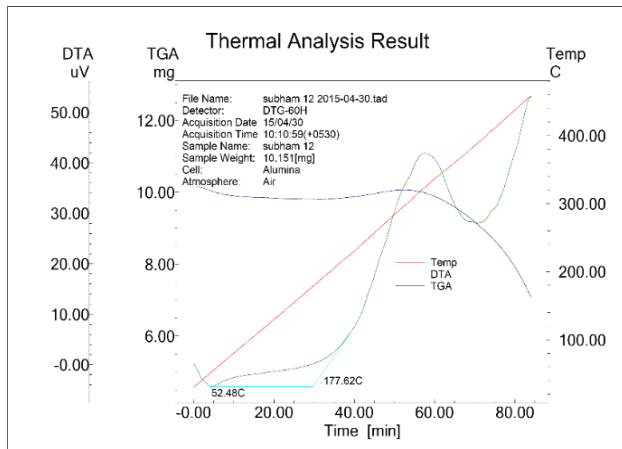
**Fig.5.14 DTA thermogram Tapeen 8A**



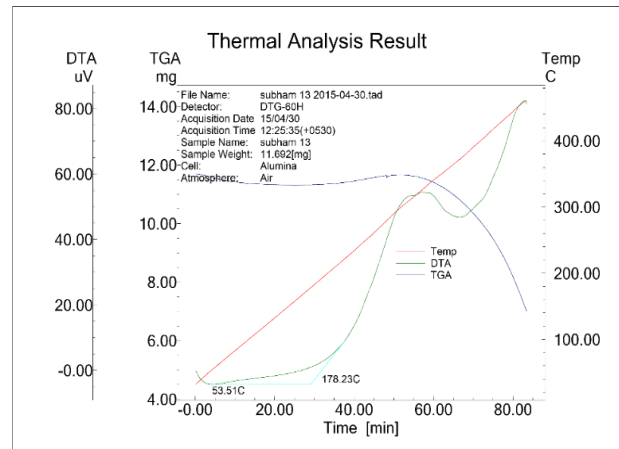
**Fig.5.15 DTA thermogram Tapeen 8B**



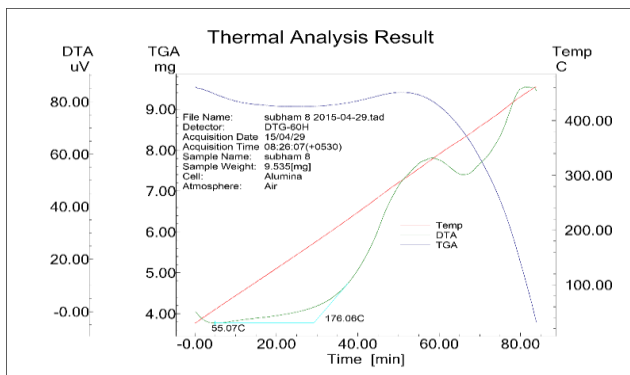
**Fig.5.16 DTA thermogram Tapeen 8C**



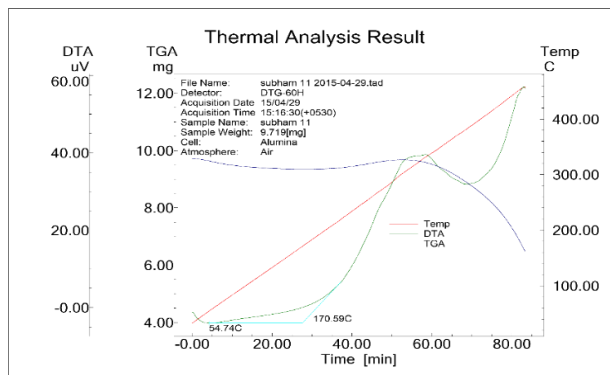
**Fig.5.17 DTA thermogram Topa 4**



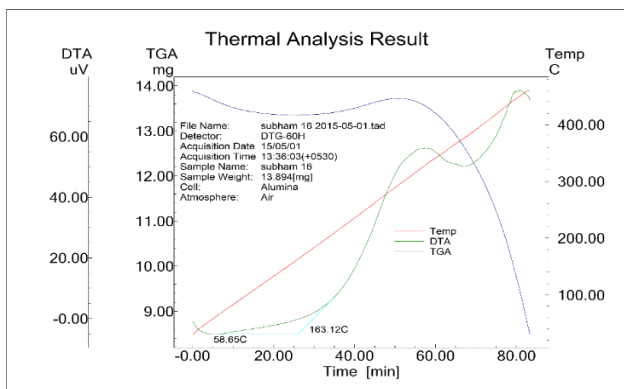
**Fig.5.18 DTA thermogram Topa 5**



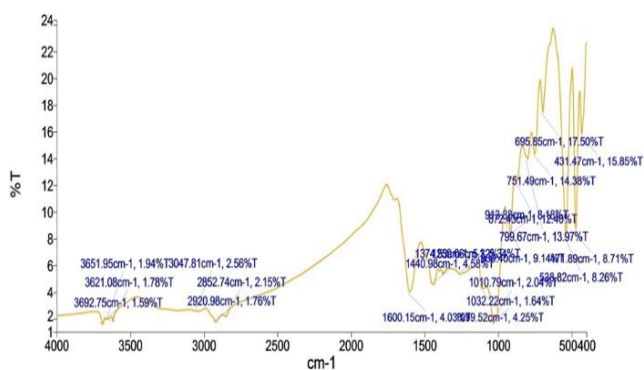
**Fig.5.19 DTA thermogram Topa 6**



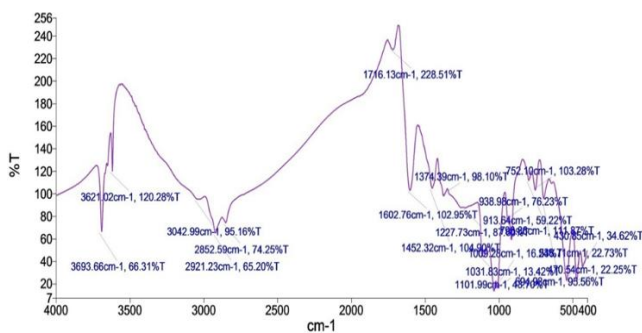
**Fig.5.20 DTA thermogram Topa 7**



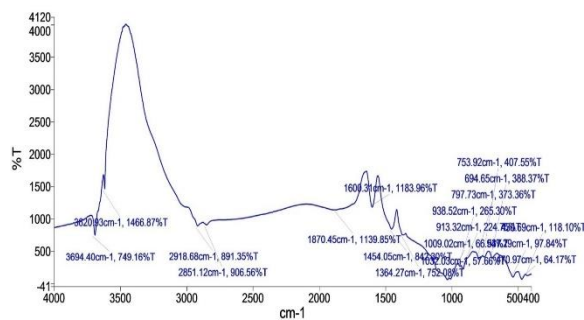
**Fig.5.21 DTA thermogram Topa 8**



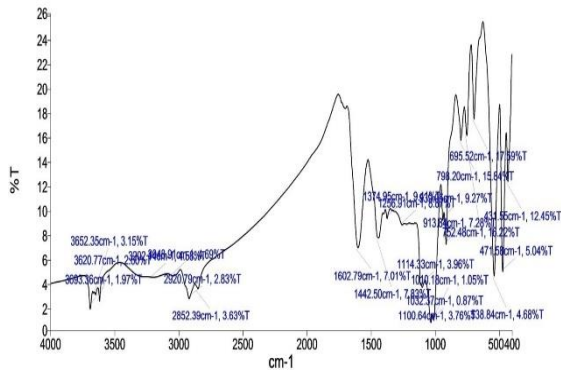
**Fig.5.22 FTIR spectra Argada 1**



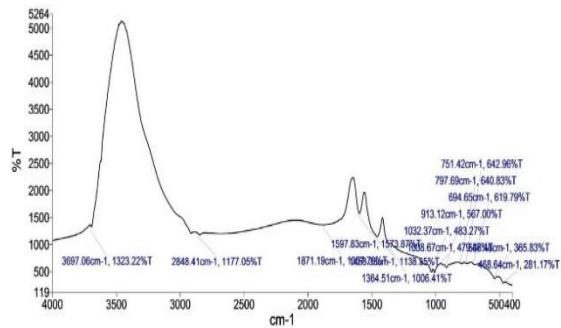
**Fig.5.23 FTIR spectra Argada 2**



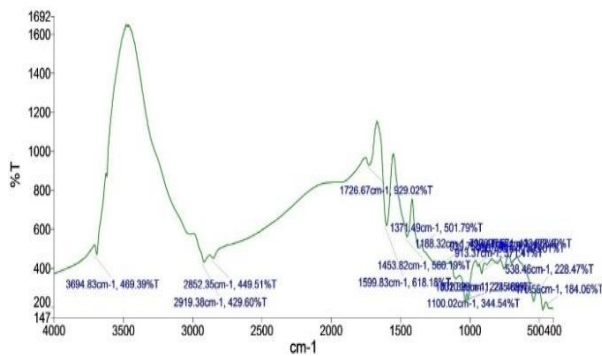
**Fig.5.24 FTIR spectra Kedla 1**



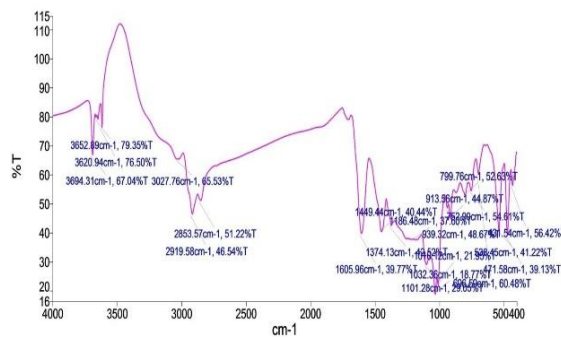
**Fig.5.25 FTIR spectra Kedla 2**



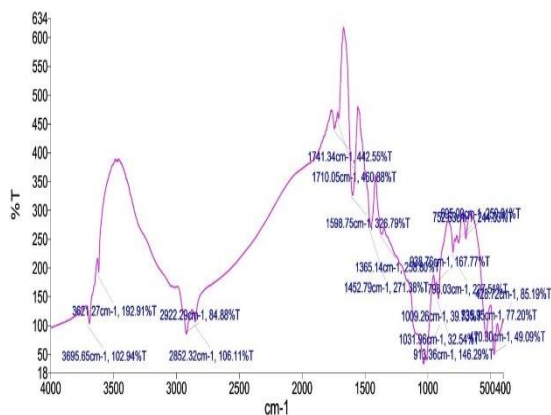
**Fig.5.26 FTIR spectra Pindra 5**



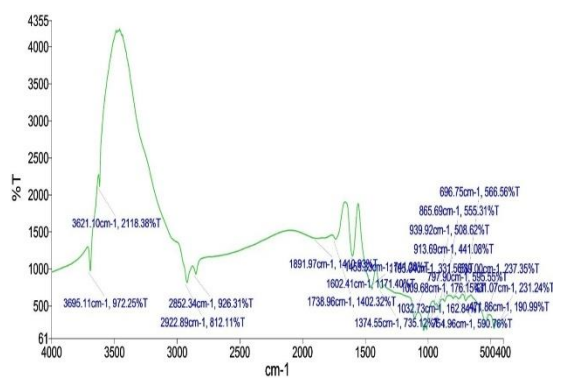
**Fig.5.27 FTIR spectra Pindra 6**



**Fig.5.28 FTIR spectra Pindra 7B**



**Fig.5.29 FTIR spectra Pindra 7C**



**Fig.5.30 FTIR spectra Pindra 8**

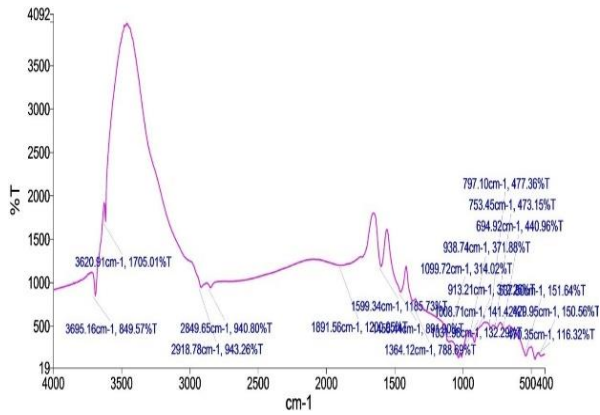


Fig.5.31 FTIR spectra Pundi 4

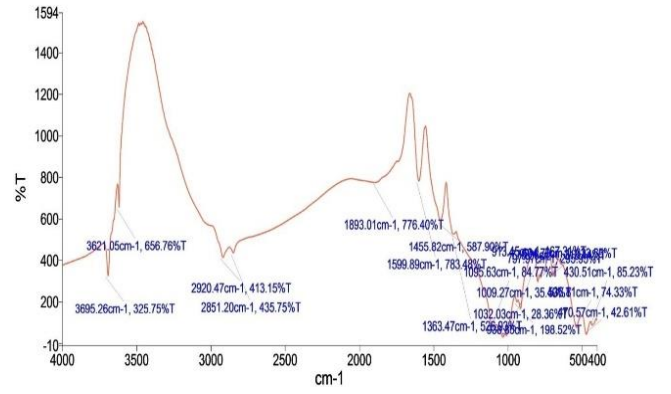


Fig.5.32 FTIR spectra Pundi 7

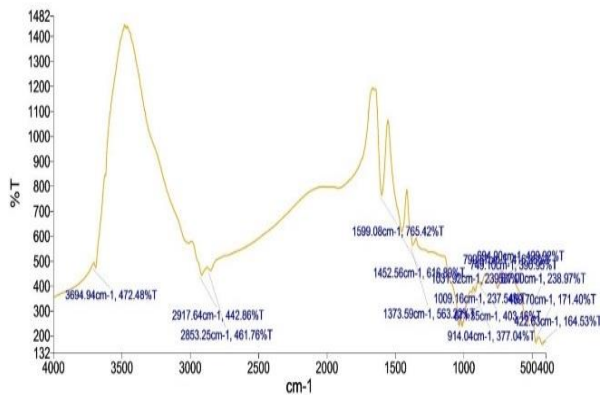


Fig.5.33 FTIR spectra Sarubera 1

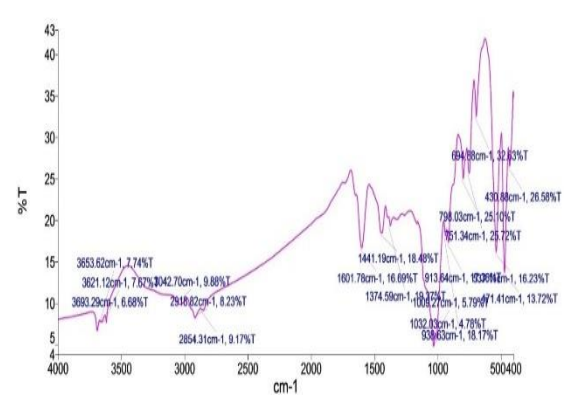


Fig.5.34 FTIR spectra Sarubera 2

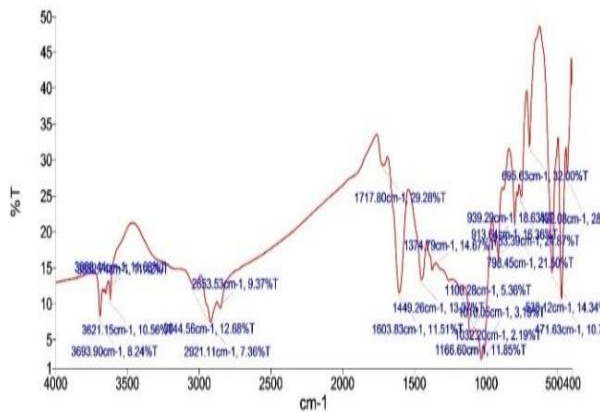


Fig.5.35 FTIR spectra Tapeen 8A

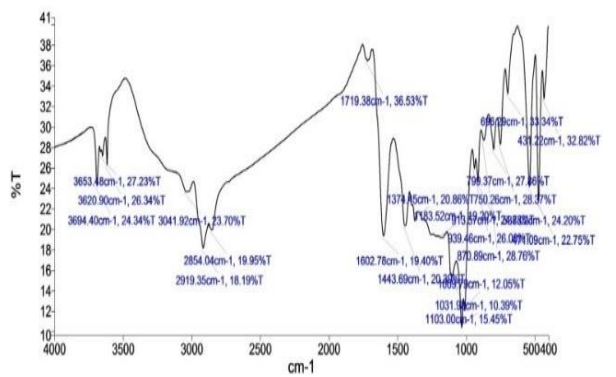


Fig.5.36 FTIR spectra Tapeen 8B

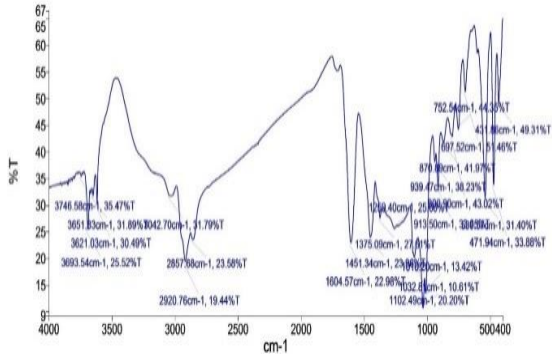


Fig.5.37 FTIR spectra Tapeen 8C

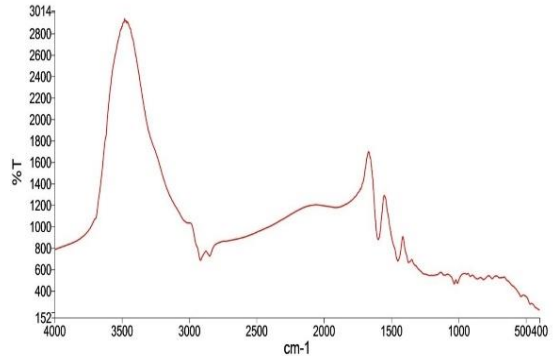


Fig.5.38 FTIR spectra Topa 4

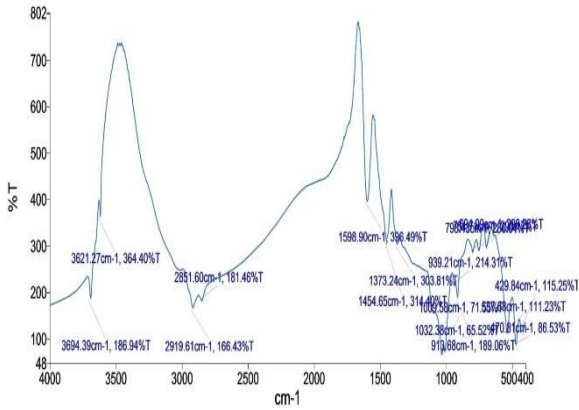


Fig.5.39 FTIR spectra Topa 5

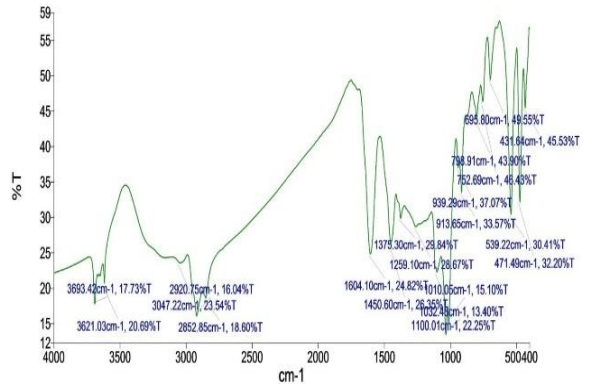


Fig.5.40 FTIR spectra Topa 6

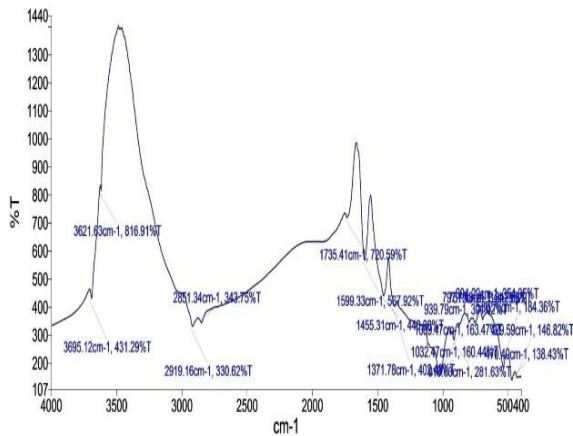


Fig.5.41 FTIR spectra Topa 7

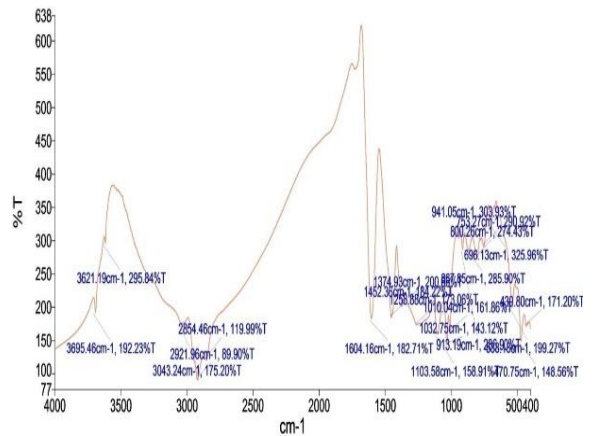


Fig.5.42 FTIR spectra Topa 8