

# **An Investigation into Spontaneous Heating Characteristics of Coal and its Correlation with Intrinsic Properties**

A THESIS SUBMITTED IN PARTIAL FULFILLMENT  
OF THE REQUIREMENTS FOR THE DEGREE OF

**BACHELOR OF TECHNOLOGY  
IN  
MINING ENGINEERING**

By  
**PRASOON SINGH**  
110MN0536



**DEPARTMENT OF MINING ENGINEERING  
NATIONAL INSTITUTE OF TECHNOLOGY  
ROURKELA - 769008  
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Under the guidance of

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NATIONAL INSTITUTE OF TECHNOLOGY  
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2014**



## **National Institute of Technology, Rourkela**

### **CERTIFICATE**

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This is to certify that the thesis entitled “**An Investigation into Spontaneous Heating Characteristics of Coal and its Correlation with Intrinsic Properties**” submitted by Sri Prasoon Singh (Roll No. 110MN0536) in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in this thesis has not formed the basis for the award of any Degree or Diploma or similar title of any University or Institution.

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## ACKNOWLEDGEMENT

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I would like to express my profound gratitude and indebtedness to Prof. H.B. Sahu, Associate Professor, Department of Mining Engineering, NIT, Rourkela for his guidance and valuable suggestions. This work would not have been possible without his encouragement and constructive criticism. I sincerely thank him for the time and patience he devoted for this work.

I am also thankful to all the faculty members for their valuable suggestions.

I am immensely thankful to Mr. B.K. Pradhan, Mr. B.N. Naik, Technical Assistants; and Mr. Soma Oram, Laboratory Attendant for their help in carrying out laboratory experiments.

I would like to acknowledge the authors of different research paper referred in the work, which were a valuable source for understanding the subject.

Lastly, I am thankful to all my friends who have encouraged and helped me in accomplishing this project.

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## ABSTRACT

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Spontaneous heating property of coal is a matter of prime concern in mining industry. Spontaneous combustion of coal has caused many mine fires leading to huge loss of coal and also loss of many human lives. Storage and transportation of coal requires knowledge about the spontaneous heating liability of coal, as it starts degrading on coming in contact with air. Numerous studies and research work has been done to understand the mechanism of spontaneous heating and its relation with coal's intrinsic properties. The present study presents the correlation of spontaneous heating susceptibility of coals belonging to different coalfields of India with their intrinsic properties.

The coal samples were collected from different Indian coalfields. Twelve coal samples were collected from Mahanadi Coalfield Ltd. (MCL), Singareni Collieries Company Ltd. (SCCL), Bharat Coking Coal Ltd. (BCCL) and North Eastern Coal Fields Ltd. (NECL). The Intrinsic properties were determined by carrying out – proximate analysis, Aliphaticity from Fourier transform infrared spectroscopy (FTIR) and Gross calorific value (GCV) using Bomb calorimetry. The susceptibility indices were found using Crossing point temperature (CPT) and Wet oxidation potential difference (WOPD). The correlation of intrinsic properties and susceptibility indices is done using both Simple linear regression and multiple linear regression. The results for intrinsic properties and the susceptibility indices are presented in table 1.

The simple linear regression analysis showed that the moisture, ash and aliphaticity as compared to other intrinsic properties are a better indicator of spontaneous heating as they have better correlation with CPT and WOPD. Therefore, aliphaticity is suggested as a good indicator of spontaneous heating susceptibility. On multiple linear regression analysis strong correlation was found between moisture, ash, aliphaticity and both the susceptibility indices. It is concluded that use of multiple parameter for predicting spontaneous heating might give better results. The results from this study is expected to help other researchers in further understanding of spontaneous heating of coal and help engineers in better production planning and safe operation.

**Table 1: Intrinsic properties and Susceptibility Indices of Coal Samples**

Sl. No.	Sample Name	Proximate			GCV (cal/gm)	Aliphaticity	CPT (° C)	WOPD after 30 minutes (mv)
		M (%)	V.M. (%)	A (%)				
1	MCL - 1	4.85	25.385	33.385	4370.11	1.515	145	56.7
2	MCL - 2	8.1	38.75	11.3	4821.012	1.41	148	64.4
3	MCL - 3	8.125	27.4	26.01	5570.72	1.057	156	41.8
4	MCL - 4	4.45	19.27	45.93	4521.633	1.1	153	41.7
5	MCL - 5	3.135	22.175	44.12	3756.361	0.918	149	46.3
6	MCL - 6	6.91	27.95	37.1	4968.26	0.93	143	60
7	CCL - 1	7.35	18.8	43.075	3484.759	1.109	145	62.1
8	CCL - 2	6.435	29.5	36.86	3123.503	0.97	142	56
9	BCCL - 1	10.8	22.9	17.9	6780.51	0.826	157	35.1
10	WCL - 1	8.9	38.8	15	5520.8	1.355	160	44.4
11	NECL - 1	2.73	42.85	8.4	3320.39	0.97	139	35
12	SCCL - 1	4.885	21.25	25.535	3145.26	1.678	130	68.3

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# **CHAPTER 1**

## **INTRODUCTION**

## 1. Introduction

### 1.1 GENERAL

Spontaneous combustion is a fairly common phenomenon in coal mining. Miners have faced this problem from the very start of coal mining. Several cases of mine fires started by spontaneous combustion of coal can be found since the beginning of coal mining history. Coal found in a lot of Indian coalfields are highly susceptible to spontaneous heating. Cases of mine fires started due to spontaneous heating and continued to burn for decades are numerous, and common all over the world. An example of most spontaneous combustion susceptible coalfield is Jharia coalfield in BCCL India which has many mine fires burning since 1925 (Ramlu, 2007). Cases of fire have also been reported during transportation of coal. Large cargo ships carrying coal have been reported to catch fire (Ramlu, 2007). Another problem associated with spontaneous heating is the deterioration of coal quality due to oxidation on coming in contact with air. This reduces the heating value of coal and affects its coking properties. Storage and transport of run-of-mine coal is an integral part of coal mining industry. Coal handling systems are required not only at mines but also at power plants and metallurgical coking plants. Apart from loss of coal, fire due to spontaneous heating also cause a lot of environmental pollution. Investigators have found that these incidents could have been prevented if proper planning and design for coal production and handling was adopted. This has led to number of researches to investigate and try to understand the process of spontaneous heating and find methods to prevent it.

Researchers have investigated the subject from both chemical and physical considerations and developed many theories trying to explain the process of spontaneous heating. Many methods have been suggested to measure the susceptibility of coal to spontaneous heating. These indices are important in design of coal panels. The incubation period of coal is decided using these indices which then in turn governs the panel size. It is important that the coal is extracted before the incubation period ends. Measures to inhibit spontaneous combustion may be required if extraction period is greater than incubation period of coal. Some of the theories of spontaneous combustion which have gained some popularity are Bacterium theory, phenol theory, coal-oxygen complex theory, pyrite theory etc. These theories though very advance, still do not hold under all conditions. This is because of the very less understanding of spontaneous combustion process due to the complex nature of coal.

There are many different methods to assess the spontaneous combustion liability. Different methods are followed in different countries, some of these are Differential thermal analysis, Wet oxidation potential, Crossing point temperature, Russian U-index, Oplinski index, Adiabatic calorimetry etc. There are many new technique emerging like Gas indices studies, Critical air blast rate, Differential scanning calorimetry etc. However, none of the methods have been successful in exactly predicting the spontaneous heating

susceptibility of coals. Use of more than one method to predict the spontaneous heating susceptibility may give a better result than using a single method.

## **1.2 OBJECTIVES**

To investigate the spontaneous heating susceptibility of coal and correlate its intrinsic properties with susceptibility indices. The work is divided into four parts. The objectives of project have been planned as follows:

- Sample collection and preparation according to standard procedures.
- Determination of the intrinsic properties of the collected coal samples using proximate analysis, bomb calorimetry and Fourier transform infrared spectroscopy.
- Determination of spontaneous heating susceptibilities by Crossing point temperature test and wet oxidation potential analysis.
- Correlation of spontaneous heating susceptibilities indices with that of the intrinsic properties.

## **CHAPTER 2**

### **LITERATURE REVIEW**

### 2.0 LITERATURE REVIEW

There has been a lot of research and studies on the spontaneous heating of coal. Some of the works relevant to this study are summarized below:

**Kaji et al. (1983)** studied the low temperature oxidation of various coals, ranging from sub-bituminous to anthracite. Different sizes of coal particles were used for the study. Apparatus used for measuring oxidation rates had a thermocouple to measure temperature and gas chromatography was used to analyze carbon di-oxide and carbon mono-oxide formation. Pore size distribution of coal samples were measured using mercury porosimeter. It was found that pore structure had significant effect on oxidation rate. They concluded that the oxidation rates were directly proportional to volume of pore of radius greater than 12 Å. However, it was found that pores which have opening greater than 100 Å radius were only responsible for reaction and that gaseous O<sub>2</sub> may not penetrate to smaller pores.

**Schmal et al. (1984)** developed a one-dimensional model to investigate spontaneous heating at low temperature (<100<sup>0</sup>). The process that were taken into account in developing the model included oxygen depletion, heat releases due to oxygen chemisorption, diffusion and convection of O<sub>2</sub>, heat transfer by convection and conduction and coal moisture. It was found that porosity had a great effect on process of spontaneous heating in absence of moisture. It directly as well as indirectly, via its influence on gas velocity between coal particles and on thermal conductivity effects the spontaneous heating.

**Clemens et al. (1989)** analyzed the reaction of low rank coals (from New Zealand) with oxygen at temperatures of 30, 60, 90, 120, 150 and 180<sup>0</sup> C using differential thermal analysis. They found an immediate, sharp exothermic reaction between oxygen and coal. It was observed that exotherm increases with increasing temperature and is most pronounced for the coal most prone to spontaneous combustion. It was concluded that DTA techniques may be useful to gain more information about mechanisms involved and susceptibility of coals to spontaneous heating when applied to isothermal low temperature oxidations of coal.

**Tarafdar and Guha (1989)** presented the results of wet oxidation of coal using alkaline permanganate solution. They also measured the differential temperature at different base temperatures. Correlation of results obtained with that of CPT values was done and it was suggested that differential temperature and potential difference measurements during wet oxidation of coal may be used as alternative technique for the assessment of tendency to spontaneous heating.

**Jones and Vais (1990)** measured the activation energy of ignition and thermal diffusivity of three different Thai lignites and for a sample of an Australian lignite. They found the activation energy of the order  $10^2$  KJ/mol and thermal diffusivity of the order  $2 \times 10^{-7}$  square meter per second. They compared these values with existing values of similar materials and found that processes reflecting geological advance along coalification series (increase in carbon, decrease in oxygen) are accompanied by increase in value of product of thermal diffusivity and bulk density.

**Arisoy and Akgun (1994)** used a large scale experimental apparatus 3m in length and 0.3m in diameter to evaluate the effect of particle size on spontaneous combustion. It was found that overall rate of oxidation becomes less sensitive to temperature as particle size increases. They also concluded that as temperature increases effect of particle size on spontaneous heating becomes more significant.

**Krishnaswamy et al. (1995)** used a reaction-diffusion two-dimensional model for studying the role of bed porosity, side slope wind velocity, coal reactivity and bed particles size in spontaneous combustion of open coal stockpiles where the influence of moisture migration can be neglected. It was found that side slope, wind velocity and bed porosity exert the strongest influence on spontaneous heating of coal stock piles.

**Vance et al. (1996)** investigated the effect of moisture content of coal on its spontaneous heating susceptibility. They found that highest heating rate occurred when moisture content was 7 wt%. This was most pronounced at temperatures below  $80^{\circ}$  C. Furthermore they found that for high temperature like  $110^{\circ}$  C drying of coal gave less reproducible results. It was concluded that this was due to “thermal damage” to solid structure and un-controlled re-absorption of moisture by a very dry coal.

**Pis et al. (1996)** used differential thermal analyzer to study the self-heating behavior of fresh and oxidized coal. They found that the self-heating temperatures were between  $180^{\circ}$  C and  $270^{\circ}$  C for coals ranging from high A-bituminous to semi-Anthracite. The lowest temperatures compared to lower rank coals. It was found that oxidation resulted in change in both self-heating and end of combustion temperatures. They concluded that volatiles released up to  $425^{\circ}$  C were also responsible for this change. They also pointed out a relationship between the total heat flow and the ASTM heat value.

**Garcia et al. (1999)** measured the non-isothermal enthalpies for three Colombian coals. To reduce their susceptibility to spontaneous heating they were subjected to low temperature oxidation under ambient conditions over extended periods. Enthalpies of oxidation were determined using differential scanning calorimeter. It was found that DSC is useful in investigating early stages of oxidation of coal and that onset temperature of oxidation is a better indicator of propensity of coal to spontaneous combustion.

**Sujanti et al. (1999)** used wire mesh reactors of seven different sizes to study the low-temperature oxidation of Victorian brown coal using both steady-state and transient methods. A critical ambient temperature was found in steady-state above which self-heating occurs. This ambient temperature was



used to calculate oxidation kinetics constant. Transient state was also used to determine kinetics constant directly. It was found that result from steady-state and transient method were in agreement with each other. They suggested that transient method could be used to replace the steady-state method for a quicker assessment of the spontaneous combustion of coal.

**Nugroho et al. (2001)** used CPT to determine the effect of particle size in case of single type of coal on the rate of low temperature oxidation. They also studied spontaneous combustion tendencies of blended coal. It was concluded that the activation energies and reactivities of coal decreases with increasing particle size. They also found that the blended coals were more susceptible to self-heating due to higher reactivities and activation energies coupled with lower critical ambient temperatures of more reactive coals.

**Kusuk et al. (2002)** used crossing point temperature to determine the effect of gas flow rate, moisture of coal, humidity of air and particle size on spontaneous combustion characteristic of Akale lignite from Turkey. They also found the amount of three predominant functional groups (carbonyl, hydroxyl and carbonyl) using wet oxidation methods. It was found that the susceptibility increases with decrease in particle size. Susceptibility was reduced by increasing humidity of air or increasing flow rate of air.

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**Sahu et al. (2004)** presented the method of finding out the spontaneous heating tendencies of coal samples by using differential scanning calorimetry (DSC). 30 coal samples were collected from 7 different Indian coalfields and onsets temperature for each samples was determined. They also determined the crossing point temperature of all samples and compared the results with onset temperature obtained from differential scanning calorimetry and suggested that DSC can be a better method than CPT for assessing spontaneous heating tendency of coals.

**Panigrahy and Bhattacharjee (2004)** carried out extensive field studies to investigate pillar fire in a coal mine of Raniganj colliery. They used a thermal IR gun and a thermo vision camera to assess the state of heating in selected pillars. Sampling was done from specially designed sampling setups and a multi gas detector was used to analyze the composition of gas in situ. To predict spontaneous heating they proposed some modified gas ratios as established ones like Graham's ratio, Young's ratio, etc. gave negative values for high methane percentage. It was also suggested that these modified ratios could be used for assessing the condition of fires in sealed-off areas.

**Sahu et al. (2005)** described the spontaneous heating susceptibility of coal samples using crossing point temperature (CPT), differential thermal analysis and differential scanning calorimetry. It was suggested that all coals are not susceptible to spontaneous heating to same extent and therefore it was necessary to assess their degree of proneness in order to plan advance precautionary measures. It was also mentioned that acceptability of method in determining susceptibility of coal to spontaneous heating depends on how closely the method can predict the heating characteristics in field conditions. They concluded that the onset temperature obtained from differential scanning calorimetry may be a better method for predicting susceptibility of coals to self-heating as compared to crossing point temperature.

**Basil et al. (2008)** studied the effect of mineral matter on coal self-heating rate by adiabatic self-heating tests on coals from Callide Basin, Queensland. It was found that when mineral matter is admixed with coal then its effect as heat sink is evident. However, if mineral matter is naturally disseminated in the coal then along with heat sink effect, mineral matters also inhibits oxygen reaching carbon surfaces and probably acts on the chemical reaction mechanism.

**Nugroho et al. (2008)** evaluated the self-heating characteristics of a sub-bituminous coal under adiabatic conditions. They tested the self-heating susceptibility at different relative humidity of supplied gas. It was found that an increase in relative humidity of oxygen has a marked effect on self-heating rates. At 70% relative humidity oxidation rate was found to be highest. Effect of relative humidity decrease with increase in oxygen concentration.

**Beamish and Arisoy (2009)** tested a wide range of coals from Australia, New Zealand and Indonesia. They studied the effect of intrinsic properties on self-heating rates by adiabatic testing of coal. It was found that the relationship with coal rank could be expressed in the form of a third order polynomial for coals ranging in rank from sub-bituminous to low volatile bituminous coals. A coal ply sampling strategy was suggested to obtain site specific data regarding the effect of mineral-matter on self-heating rates. They also found that coking coals have lower self-heating rates than steaming coals due to high vitrinite percentage.

**De-ming et al. (2009)** suggested a new oxidation kinetics testing method for propensity of coal to spontaneous combustion. The concentration of oxygen in outlet of sample vessel at 70<sup>0</sup> C and crossing point temperature at temperature-programmed conditions, were selected as fundamental parameters. They used a comprehensive determination index to classify coals according to their susceptibility. It was found that comprehensive determination index is more accurate to identify propensity of coal to spontaneous heating than adiabatic oxidation time of coal.

**Geng et al. (2009)**, carried out the FTIR analysis of 35 Australian lignite coals. They studied the FTIR spectrum around the wavelength 1800–1500  $\text{cm}^{-1}$ , 3100 – 2990  $\text{cm}^{-1}$  and 2990–2750  $\text{cm}^{-1}$ . They calculated the content of COOH group in coal using both FTIR and conventional chemical analysis. They also calculated the aromaticity of coal using solid state NMR. They found that the COOH content found using FTIR was in good agreement with results from chemical analysis. They also found that the peak area ratio for aromatic to aliphatic bonds was in good correlation with aromaticity obtained from NMR.

**Xuyao et al. (2010)** investigated the CPT of different ranks of coal using self-designed CPT apparatus. They found that higher ranked coals show higher CPTs. Moisture inhibited oxidation due to evaporation and by forming a moisture screen over coal. Sulfur components of coal reacted and enhanced oxidation in moist  $\text{O}_2$  rich conditions. However, at low temperature these components form thin layer over coal inhibiting oxidation. Oxidation was also effected by air-flow rate. They also concluded that to evaluate and compare spontaneous heating susceptibility of different coals using CPT, test should be done at same or nearly same experimental conditions.

**Ejlali et al. (2011)** used a mathematical model to study the self-heating mechanisms of wet porous media (coal stockpile). Three parameters were used namely, porosity of the medium, moisture content and Darcy number. It was observed that the heating takes place in three phase. First temperature rises due to self-heating, then second phase consists of constant temperature due to liquid to vapor phase change. In third phase temperature again rises and ignition may occur at any time. It was found that the model can be used for estimation of time that a typical stockpile can be kept safe.

**Zhu et al. (2012)** predicted the self-ignition time and location of coarse coal piles using numerical model in comsolmultiphysics software. The kinetic data for low temperature oxidation was obtained from laboratory scale experiments with bituminous coal from Zindi coal mine of Shanxi province in China. They found the self-ignition temperature in coarse-stockpile is a function of wind velocity, height, porosity and side slope. It was found that wind-driven forced convections plays a predominant role in self-heating of coarse coal piles. It was also found that temperature rise decreases along with porosity solely. This showed that effects of porosity is more on oxygen availability than on heat transport.

**Xu et al. (2013)** carried out thermal analysis on coals of different ranks to find the effect of moisture content on spontaneous heating. They used differential scanning calorimetry to test heat release and thermo gravimetric analysis to detect to detect weight changes. It was found that moisture can act as an inhibitor or an accelerator depending upon moisture content and rank of coal. They also concluded that for each rank of coal there was a critical moisture content at which the coal has a high tendency for spontaneous heating.

**Choi et al. (2013)** used CPT and gas chromatography to relate spontaneous combustion susceptibility and rank of coal. They used three coals – two Indonesian Lignite and one Chinese bituminous coal. FTIR was used to analyze the hydrocarbon present in coals. It was concluded that coals of lower rank have more substance that can react with oxygen at low temperatures making them more susceptible to spontaneous combustion than higher ranking coals.

**Xia et al. (2014)** proposed a spontaneous combustion model of underground mine by establishing a set of partial differential equations, involving a coal deformation equation, an oxygen flow and transport equation and an energy conservation equation. Quantitative evaluation of extrinsic and intrinsic factors such as coal permeability, pressure difference, oxygen-consumption rate, and reaction heat of coal was done to assess their influence on spontaneous heating. It was found that during coal oxidation, the oxidation concentration has an “S-type” downward trend, whereas the heating temperature of coal and the gas velocity show “S-type” upward trends. They proposed that the simulated results can provide some suggestions for control of spontaneous heating.

## **CHAPTER 3**

# **SPONTANEOUS HEATING THEORIES AND MECHANISM**

### 3. Spontaneous Heating

Coal or other carbonaceous matter exhibit the property of self-heating. These matter undergo a rise in temperature on exposure to air and eventually ignition without the application of any external source of heat. In mining the phenomenon is mostly used for coal only as very few other minerals exhibit this property.

#### 3.1 Spontaneous Heating Theories

Coal on exposure to atmosphere oxidizes. The oxidation process of coal is highly exothermic in nature. The reactions occurring in initial phases of spontaneous heating are purely physical in nature. The oxygen is adsorbed at the coal surface resulting in release of large amount of heat. Rate of heat release in oxidation of coal varies from 2.0 to 4.0 Cal per mole of oxygen adsorbed. Initial rate of oxygen adsorption is very high which than gradually decreases. The released heat usually gets dissipated into environment but in some cases it accumulates and the reactions taking place change to chemical in nature (Banerjee, 1985).

There are many other theories regarding source of heat generation. Some of these are –

##### Pyrites theory

The pyrites present in coal oxidizes on coming in contact with air releasing heat. Coal contains pyrites in the form of iron pyrite and it was earlier thought that the heat released on oxidation of these pyrites is the reason behind spontaneous heating of coal. But later on researchers established that the heating due to oxidation of pyrites might aid the process of spontaneous heating but it cannot be the reason behind it as coal containing very less or no pyrites also exhibited spontaneous heating. It was concluded that in addition of supplying heat from its own oxidation, the oxidation of pyrites breaks the coal into smaller pieces and thus exposes a larger surface area for oxidation.

The pyrites need to be present in adequate concentration to provide any appreciable amount of heat energy. **Muzner (1975)** found that pyrite only if present in excess of 5% can contribute to self-heating of coal.

**Graham (1923-24)** concluded that in addition to a minimum concentration of pyrite necessary for any appreciable contribution to self-heating, the pyrite present in coal should be in the form of finely divided powder. The oxidation occurs due to the reaction with moist air.



The released heat and the products released from the reaction break open the coal thus exposing a large surface area for oxidation.

### **Bacterial Theory**

The bacteria present in coal can also contribute to the heat produced during self-heating. The phenomenon of self-heating found in some carbon containing materials like haystack and wood is due to this bacterial action.

The possibility of bacteria providing the heat for self-heating has been investigated by many researchers like **Winmill (1913), and Fuchs (1927)** etc. But no conclusive proof has been found which can either dispose the theory or confirm it (**Banerjee et al., 1995**).

It is concluded that the bacteria though provide heat but the heat produced by bacterial action can only cause slight heating and cannot be the reason for spontaneous heating (**Fuchs, 1927**).

### **Phenol theory**

The theory states that the phenolic hydroxyls and poly phenols present in coal have faster oxidation rates than other groups. This theory provides a method to access the spontaneous heating liability of coal (**Jain, 2009**).

### **Water sorption theory**

Coal absorbs liquids and vapours coming in contact with it. This leads to swelling of coal which require certain amount of energy. However, the energy released on interaction of liquid with the coal surface is much larger and leads to increase in temperature of coal. This heat is known as heat of wetting. Coal is hygroscopic in nature and therefore on absorbing water it releases a large amount of heat.

The heat of wetting is caused due to the absorption of water in liquid phase however, coal also absorbs water vapour, when water vapour is absorbed by coal the heat of vaporization of water gets added to the heat of wetting which results in a considerable rise in temperature of coal.

### **Heat released due to earth movement**

The heat released due to movement of strata can sometimes contribute enough heat to cause spontaneous combustion of coal. Roof falls lead to adiabatic compression of air causing temporary rise in temperatures. Although the frictional heat produced during crushing of coal can supplement the process of spontaneous combustion however, the oxidation of coal still plays a more significant role and crushing of coal exposes a large surface area available for oxidation.

## Coal-oxygen complex theory

The initiation of reaction of coal with oxygen occurs at negative radical sights. Peroxyl radical and hydro peroxides are considered to be the products formed from in the initial stages of interaction of coal with oxygen. The peroxy complex compounds formed are temporary products and these continuously decompose, rearrange or react to form different products either inside the coal matrix or some gaseous product. These peroxy complex compounds formed in the earlier stages of reaction later on decompose at higher temperatures.

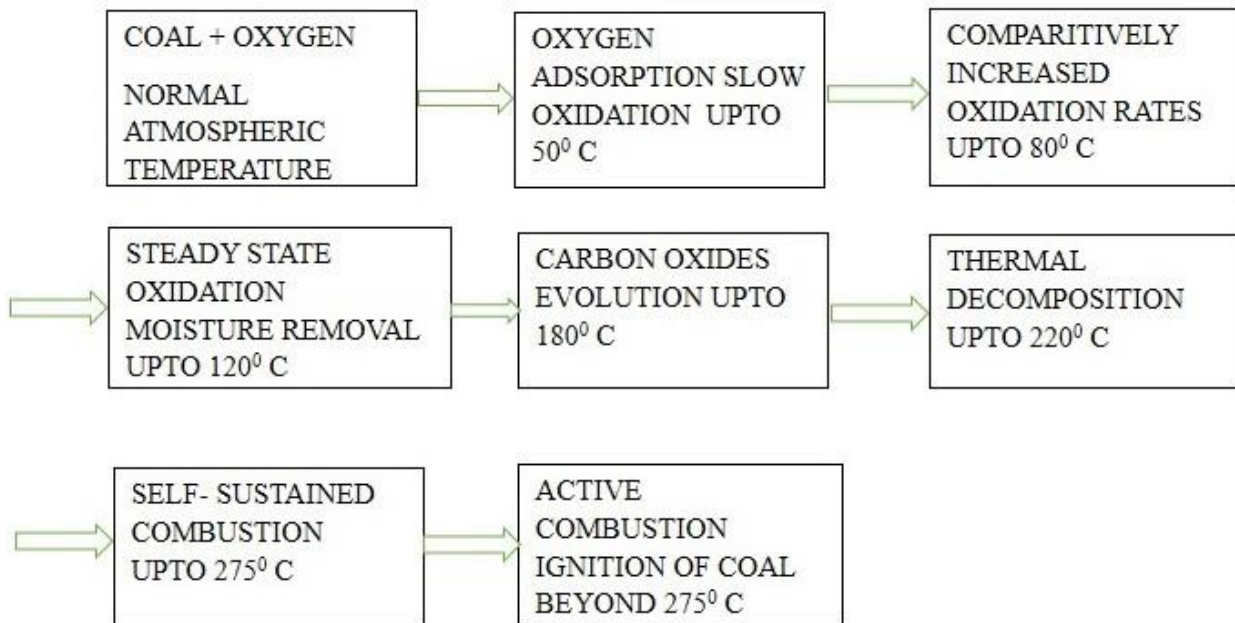
### 3.2 Spontaneous Combustion Mechanism

The oxidation of coal starts at low temperature. On coming in contact with oxygen present in air coal starts adsorbing oxygen. The process of adsorbing oxygen releases products like CO, CO<sub>2</sub> and water vapor along with a large amount of heat. The reactions occurring up to this point take place at normal temperature. The rate at which the heat is produced is slow and therefore can be easily carried away by air. However, if the heat gets confined due to absence of heat dissipation by air or the rate of heat dissipation is less than the rate of heat formation, the heat starts to build up. Up till now only certain parts of coal were taking up oxygen but as the temperature of coal gradually rises other parts also start participating in the process thus accelerating the coal oxidation process. As further increase in temperature occurs the reactions start changing from physical to chemical in nature. The peroxy-complexes formed in the earlier stages of oxidation start to decompose above 70<sup>0</sup> – 80<sup>0</sup> C producing CO, CO<sub>2</sub> and H<sub>2</sub>O. The reaction rates and type start changing rapidly after this point. The loss of moisture after 100<sup>0</sup> C results in some drastic changes. The increased temperature facilitates the formation of more stable oxygen-coal complexes. The temperature continues to rise until critical temperature is attained. At ignition point the coal catches fire and open fire breaks out. An effective circulation of air can keep the temperature from reaching critical limits. The temperature at which coal ignites is about 160<sup>0</sup> to 170<sup>0</sup> C for bituminous and 185<sup>0</sup> C for anthracite coal (Singh, 2013).

Once ignition temperature is reached and coal starts burning any increase in air circulation will only facilitates the burning of coal. The described process of self-heating of coal which ultimately results in the ignition is known as spontaneous combustion of coal.



The flowchart given below describes different stages in the process of spontaneous heating:-



**Fig. 3.1 Stages in the process of spontaneous heating**

### 3.3 Factors affecting spontaneous heating of coal

Spontaneous heating of coal has been a major source of hazard in mines leading to loss of life as well as money. It is the responsibility of an engineer to consider the factors related to prevention of spontaneous heating all the while keeping the process of mining coal profitable. Therefore he needs to consider a lot of factors to minimize the risk to life and maximize the profit.

Spontaneous heating might occur in a seam while the other adjoining seams and working will never experience any spontaneous combustion. It may also occur that only a certain part of the seam is susceptible to spontaneous heating and not the whole seam. Therefore each factor needs to be analyzed to predict the susceptibility of seam to spontaneous combustion. All the indications and causes of spontaneous combustion needs to be dealt with as early as possible because if not done so the results can be devastating.

Some of the important factors that affect the spontaneous heating of coals are:

#### **Moisture**

Effect of moisture on spontaneous heating is uncertain. Moisture in small quantity accelerates spontaneous heating, however in higher quantity it tends to retard the heating. Initial heating rates at

room temperature have been found to be high for coals with high moisture content. However, the time taken to reach thermal runaway increased with moisture. (Beamish et al., 2008).

### **Volatile Matter**

Volatile matter increases the susceptibility of coal to spontaneous combustion by lowering the ignition temperature of coal. Coal containing high volatile matter has a higher tendency of catching fire (due to low temperature exothermic oxidation) when stored in open space (**Gupta, 2007**)

### **Particle Size**

Particle size is a very important parameter affecting the spontaneous heating of coal. The smaller the size of particle larger is the surface area available for oxidation. The activation energy required for oxidation reactions decreases with decreasing particle size. Therefore, the oxidation rates will be high even at low temperature (**Akgun et al., 1994**). Concentration of oxygen functional group increases with decrease in particle size. Particle size plays an important role in oxidation at low temperature (**Kucuk et al., 2002**).

### **Petrographic Component**

The susceptibility of coal to self-heating decreases with increasing inertinite content (**Moxon et al., 1985**). Vitrinite contains more non-aromatic components than inertinite and therefore is easily oxidize at low temperatures making coal more susceptible to self-heating. Exinite is the most reactive maceral and therefore increases the spontaneous heating liability (**Falcon, 1986**).

### **Caving Characteristics**

In mines practicing partial extraction methods, pillars are left to support the superincumbent strata, the caving characteristics are of very less significance. The permeability of the waste should be kept to a minimum. The waste material should be friable enough so that it breaks into fine pieces and fill the voids to minimize air flow.

### **Mining method**

Mining method strongly affects the spontaneous heating characteristics of coal. An advancing long wall method leaves behind extracted area which lies between the entries serving the working places. The ventilating pressure difference between entries encourage the rate of air flow across the mined out area. This leads to increased rate of self-heating and a high risk of combustion. Where coal is highly susceptible to spontaneous-heating retreat system of long wall is practiced.

## **Rate of Advance**

When a waste area is located adjacent to working place there will be a lot of air entering the waste area. Due to bleed action or force of ventilation air enters the waste area. As the face advances a location in waste area experiences increased air flow due to increase in porosity and permeability. But as the face advances the air flow returns to its normal values. The time taken to complete this transition is important. Longer the coal remains in this transition phase higher is the spontaneous-heating risk. Therefore, by controlling the rate of advance of face spontaneous-heating can be reduced.

## **Leakage**

For spontaneous heating to take place there should be enough supply of oxygen and a situation where built-up of heat can take place. This situation is brought up by the air leaking through the fissures into the solid coal and results in oxidation of coal. Thus high leakage can create dangerous situation of high risk of spontaneous combustion. The leakage occurs at places where there is a high pressure gradient like around doors and regulators. The leakage depends upon the pressure difference between the two sides of stoppings. The material used for constructions of stoppings are usually permeable and some air always leaks through them. The leakage across porous materials such as stopping or well stowed goaf is in direct proportion to difference in pressure on two sides.

## **Ventilation**

Ventilation plays an important role in the spontaneous heating of coal in an underground coal mine. If the ventilation rate is too high, heat is carried away by convection. If the ventilation rate is too low, the reaction rate becomes oxygen-limited. The heat produced by oxidation of coal will not dissipate if ventilation rate is low but it will also restrict the oxygen availability for oxidation. There is an optimum ventilation flow to produce maximum rate of temperature rise at the critical ambient temperature (**Yuan et al., 2012**)

## **CHAPTER 4**

# **EXPERIMENTAL INVESTIGATIONS**

## **4. Experimental Investigations**

### **4.1 Sampling**

This chapter presents the methods of sample collection and experimental investigation to determine the intrinsic properties and spontaneous heating. Sampling is the process of obtaining a representative set of data from the original population. Coal samples are collected from different mines in small quantities to analyze the constituents of the whole seam. During the prospecting stage of the mine sampling is done for the first time. Samples are collected from the trenches dug at the time of prospecting and also from the cuttings from the churn drills and cores from diamond drills. The results obtained from the samples are used for feasibility studies and in deciding the future of mine.

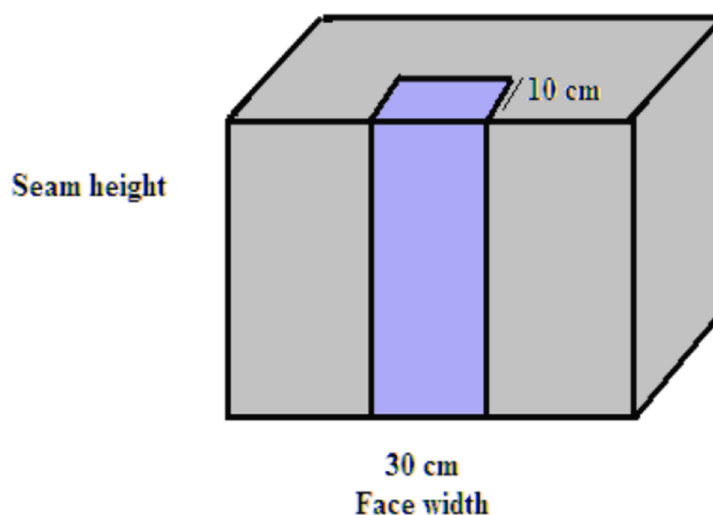
When the mine enters developmental stage the samples are then collected from the roadways. Samples might also be collected from further exploratory drilling done underground.

There are several methods employed in sample collection:

1. Channel or groove sampling.
2. Chip sampling.
3. Grab sampling.
4. Bulk sampling.

The samples were collected using the method of channel sampling. The method followed was that of IS 436 Part I/Section I - 1964. A section of seam is chosen which is exposed from roof to floor. The sample is taken from a channel dug in the seam across its whole cross-section. The channel to be dug has a width of 30cm and a depth of 10cm. (Fig. 4.1)

For the purpose of creating the channel two parallel lines are drawn on the exposed surface of seam. A clean cloth or tarpaulin was kept just below the marked channel. The material in between the marks is dug up to a depth of 10cm. The falling material is collected on the cloth or tarpaulin so that small chips or broken coal pieces do not scatter away. In selecting the place for sampling dirt bands exceeding have to be avoided. The name of the coalfields and subsidiaries from which samples have been collected are presented in table 4.1.



**Figure 4.1 Channel Sampling**

The samples collected from different mines were brought immediately to laboratory for sample preparation. Coning and quartering is done to get a representative sample. The sample was then crushed according to the experimental requirements.

For conducting tests for proximate, calorimetry, wet oxidation potential and CPT the sample was crushed and sieved to  $-212 \mu$  size. For the purpose of FTIR analysis the sample was sieved with 200 mesh size. The crushed samples were kept in air-tight plastic bags to isolate it from external factors which might result in some change of its properties.

**Table 4.1: Samples collected**

Sample No.	Sample Name
1	MCL - 1
2	MCL - 2
3	MCL - 3
4	MCL - 4
5	MCL - 5
6	MCL - 6
7	CCL - 1
8	CCL - 2
9	BCCL - 1
10	WCL - 1
11	NECL - 1
12	SCCL - 1

## 4.2 Determining Intrinsic Properties of coal

### 4.2.1 Proximate analysis (IS 1350 part I -1984)

Method used in performing the proximate analysis was taken from IS 1350 part I -1984. The procedures were used to obtain the moisture %, volatile matter % and ash %.

#### Moisture

Some amount of moisture is always present in coal, it can be either physically bounded to coal or chemically bounded depending upon its origin and nature. On air drying that is drying the coal at normal atmospheric temperature the coal loses some of its moisture. This dry coal still contains some moisture which can only be removed by heating the coal at 100<sup>0</sup> C. This moisture is called inherent moisture as it is present due to the inherent hygroscopic nature of coal. The former moisture is called accidental or free as it occurs due to handling of coal. In this work, moisture content of the coal samples was found out by taking about 1g of finely powdered (-212 μ) air-dried coal sample in a watch glass and then placing it inside a moisture oven maintained at 108±2 °C. It was allowed to remain in the oven for 1.5 hours and was then taken out with a pair of heat resistant gloves, cooled in a desiccator for about 15 minutes and then weighed. The loss in weight was reported as moisture (on percentage basis).

The calculation was done as per the following..

$$\% \text{ Moisture} = \frac{Y - Z}{Y - X}$$

Total moisture content of the original sample,

Where, X = weight of empty crucible, g

Y = weight of crucible + coal sample before heating, g

Z = weight of crucible + coal sample after heating, g

Y - X = weight of coal sample, g

Y - Z = weight of moisture, g

#### Volatile matter

Volatile Matter is the loss in mass of coal when it is heated in anaerobic conditions in a closed crucible. The loss in mass has to be corrected for moisture to get the volatile matter content. Thermal decomposition of the various constituents of coal leads to this loss in mass. Some of these constituents of coal volatile matter are, methane, other hydrocarbons, tar vapours, oxygen containing compounds and some incombustible gases, such as carbon dioxide and water vapour.

In this study, a special silica crucible (38mm height, 25mm external diameter and 22mm internal diameter) was used. The empty volatile matter crucible was weighed. Approximately 1g of coal sample

of -212 $\mu$  (BSS) size was weighed in the volatile matter crucible and it was placed inside a muffle furnace maintained at 925 $\pm$ 5 °C with the lid covering the crucible. The heating was carried the muffle furnace out exactly for seven minutes, after which the crucible was removed, cooled in air, then in a desiccator and weighed again. The calculation was done as per the following.

$$V.M \% = \frac{Y - Z}{Y - X} \times 100 - M$$

Where X = weight of empty crucible, g

Y = weight of crucible + coal sample before heating, g

Z = weight of crucible + coal sample after heating, g

Y-X = weight of coal sample, g

Y- Z = weight of volatile matter + moisture, g

### **Ash content**

Coal ash is the residue that remains after the coal combustion in presence of air. Ash is not a part of coal composition as such. It is produced due to the chemical reactions of coal constituents on combustion. It is the mineral matter which is often confused with ash but it is not so.

There are two types of Mineral matter from which ash is formed in coal: extraneous and inherent mineral matters. The extraneous mineral matter consists of materials such as calcium, magnesium and ferrous carbonates, pyrite, marcasite, clays, shales, sand and gypsum. The extraneous mineral matter owes its origin to:

- i) During conversion of vegetative matter to coal the external matters that gets associated in the process, this type of mineral matter is difficult to remove by washing coal
- ii) The material such as dirt and clay that gets mixed during production of coal, this type of mineral matter can be easily removed by coal washing.

Indian coals have drift origin and therefore have a large concentration of first type of mineral matter. In this work, the procedure followed for ash content determination is described as follows. The empty silica crucible was cleaned by heating in a muffle furnace for one hour at 800°C so that other mineral matters if present get burnt. It was taken out, cooled to room temperature and the weight is taken. Approximately 1gm of coal sample of -212 $\mu$  (BSS) size was weighed in the crucible and placed in a muffle furnace at 450°C for 30 minutes and the temperature of the furnace was raised to 850°C for 1 hour. The crucible was taken out and placed in a desiccator and weighed.

$$\% \text{ ASH} = \frac{Z - X}{Y - X}$$

Where X = weight of empty crucible in grams



Y = weight of coal sample + crucible in grams (Before heating)

Z = weight of coal sample + crucible in grams (After heating)

Y - X = weight of coal sample, g

Z - X = weight of ash, g

### Fixed carbon

It is determined by subtracting the sum of all the above three parameters from 100% and is given as Fixed Carbon,

$$FC = 100 - (M + V + A)$$

Where,

M: Moisture content

V: Volatile matter content

A: Ash content

The results of proximate analysis is as presented in table 4.2

**Table 4.2: Results of Proximate Analysis**

Sample No.	Sample Name	Moisture (%)	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)
1	MCL - 1	4.85	25.385	33.845	35.92
2	MCL - 2	8.1	38.75	11.3	41.85
3	MCL - 3	8.125	27.4	26.01	38.465
4	SCCI - 1	4.885	21.25	25.535	48.33
5	MCL - 4	4.45	19.27	45.93	30.35
6	MCL - 5	3.135	22.175	44.12	30.57
7	CCL - 1	7.35	18.8	43.075	30.775
8	CCL - 2	6.435	29.5	36.86	27.205
9	MCL - 6	6.91	27.95	37.1	28.04
10	NECL - 1	2.73	42.85	8.4	46.02
11	BCCL - 1	10.8	22.9	17.9	48.4
12	WCL - 1	8.9	38.8	15	37.3

## 4.2.2 Determination of Gross Calorific Value

The calorific value of coal represents the total potential energy stored inside coal. The calorific value of coal can be used to compare the grade of different coals. Different materials have different amount of calorific value. Bomb calorimeter is used to measure the calorific value of coal.

### Bomb calorimeter

Bomb calorimeter contains a hollow cylindrical vessel called bomb made from stainless steel. The cylinder is provided with an air tight cover which is screwed over the cylinder. On the cover there are three terminals provided for the purpose of filling oxygen into the bomb. The other two terminals are provided for passing electric current to produce spark. After the oxygen is forced into the bomb the terminal is closed.

The whole arrangement is then kept inside a water jacket containing a known amount of water. Arrangements are made to minimize heat loss due to radiation. A mechanical stirrer is fixed to mix the water inside jacket to maintain an even temperature distribution in water. A thermocouple is provided for recording the temperature of water.

### Experimental Procedure

Around 1gm coal was measured in the digital balance and the reading imported to the bomb calorimeter using start pre-weight button and stored adjacent to the name of sample. Coal was then placed in the small crucible provided and fixed in the arrangement provided in the cover of bomb. A piece of nichrome wire was cut and attached to the two rods below the cover such that the wire is in contact with coal. The photographic view of the bomb calorimeter has been presented in fig. 4.2.



Figure 4.2: Photographic view of Digital Bomb Calorimeter

The whole arrangement was carefully screwed over the bomb and oxygen line was attached over the valve on the cover. Oxygen fill button was pressed and oxygen was filled in the bomb. After this the bomb was carefully placed inside the water jacket. The bomb is to be placed so that it does not come in contact with the stirrer. The leads are attached to the two terminals provided on the cover. The lid of machine is closed and the Start button is pressed. After sometimes the machine will ignite the coal and display the Gross Calorific Value on screen. The values of GCV obtained is presented in the table below:

**Table 4.3: Gross Calorific Value of coal samples**

Sl. No.	Sample Name	GCV (cal/gm)
1	MCL - 1	4370.11
2	MCL - 2	4821.012
3	MCL - 3	5570.72
4	MCL - 4	4521.633
5	MCL - 5	3756.361
6	MCL - 6	4968.26
7	CCL - 1	3484.759
8	CCL - 2	3123.503
9	BCCL - 1	6780.51
10	WCL - 1	5520.8
11	NECL - 1	3320.39
12	SCCL - 1	3145.26

### 4.2.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a technique used to obtain an infrared absorption and transmission spectrum of a solid, liquid or gas sample. In FTIR the sample is analyzed for a wide range of wavelength at once. The result obtained is then transformed to actual spectrum using Fourier transform (a mathematical process).

When a sample is exposed to infrared radiation containing a wide range spectrum of frequencies, the chemicals present inside the sample will absorb only certain wavelength and transmits the rest.

The light from a broadband source shines into a Michelson interferometer which is a configuration of mirrors controlled by a set of motors. The motors move the mirrors in such a way that a particular wavelength of light gets blocked and then transmitted and the blocked periodically. This is repeated with every wavelength so that each time a different spectrum is obtained. The detector receives and record the intensities of each wavelength reaching it. This raw data is the fed into the computer which uses a n algorithm known as Fourier transform to convert the raw data “Interferogram” into the light absorption or transmission spectrum for each wavelength.

The photographic view of the FTIR machine is given in fig. 4.3

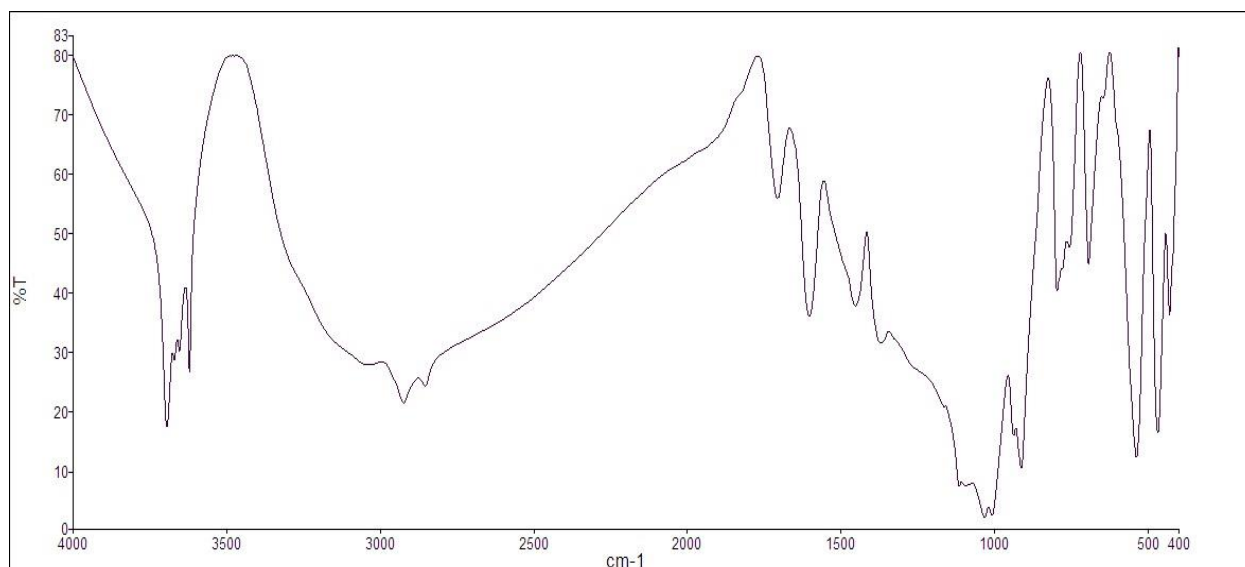


**Fig. 4.3: Photographic view of FTIR Machine**

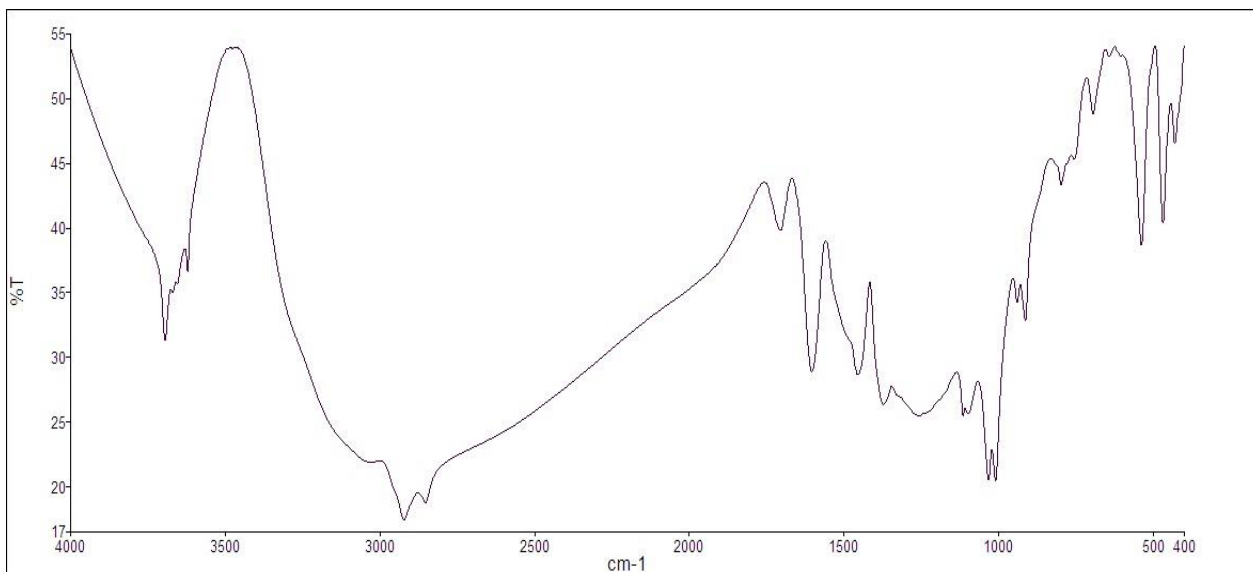
**Procedure:**

The coal samples used were air dried at 40<sup>0</sup> C. Sample was sieved with 200 size mesh. KBr was used to make pellets and the spectra range was 400 – 4000 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup>.

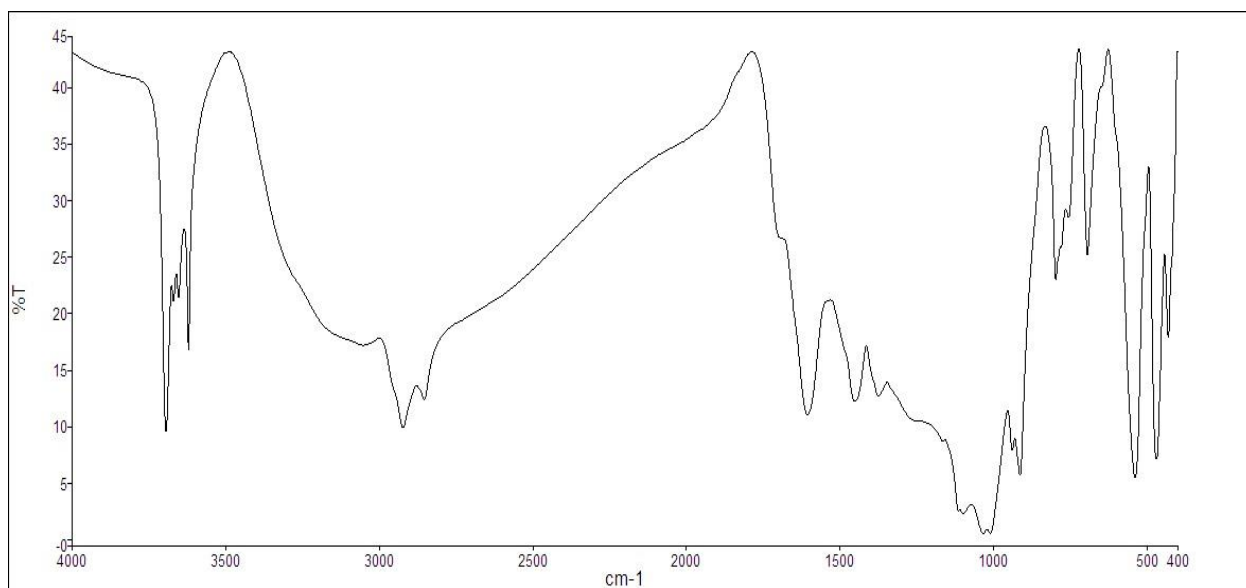
Approximately 1gm of coal was taken and mixed with 300 mg of KBr. After thorough mixing the homogenous mixture was pressed into pellets using a pressing machine applying a pressure of 10 tonnes. A background reading was taken using only KBr pellet before taking the reading for coal samples. All pellets were kept in a dessicator for 2 hours prior to testing. The Transmittance (T) plot for all coal samples obtained from FTIR is presented in figures 4.4 to 4.15.



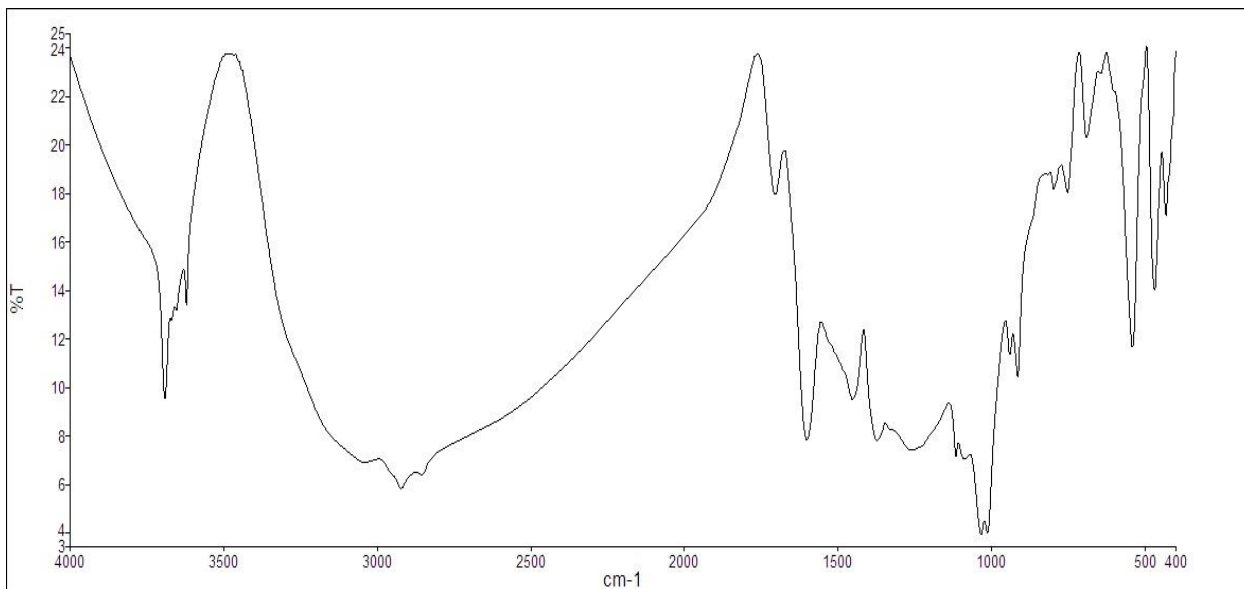
**Figure 4.4: FTIR Transmittance spectra for MCL - 1**



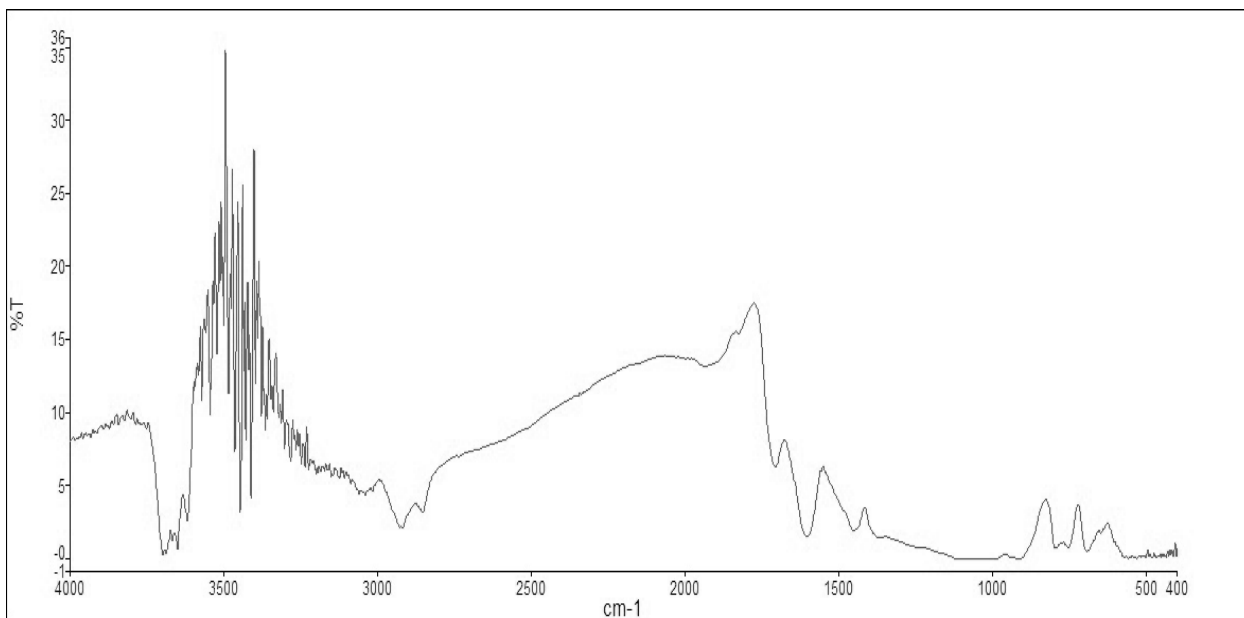
**Figure 4.5: FTIR Transmittance spectra for MCL – 2**



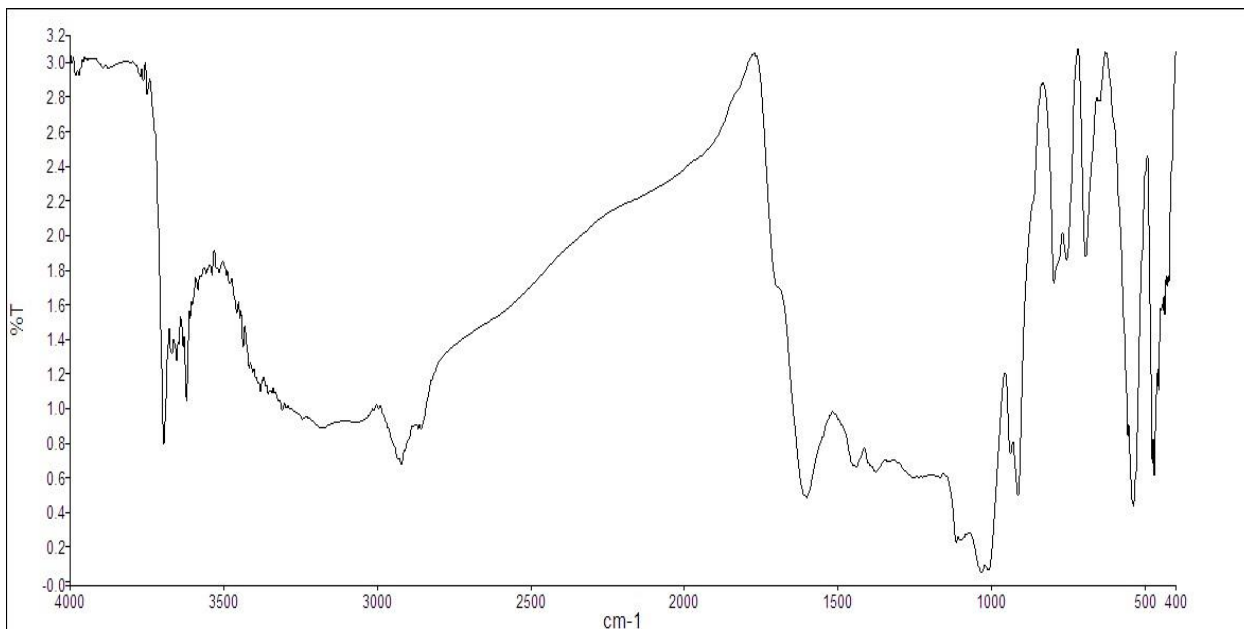
**Figure 4.6: FTIR Transmittance spectra for MCL - 3**



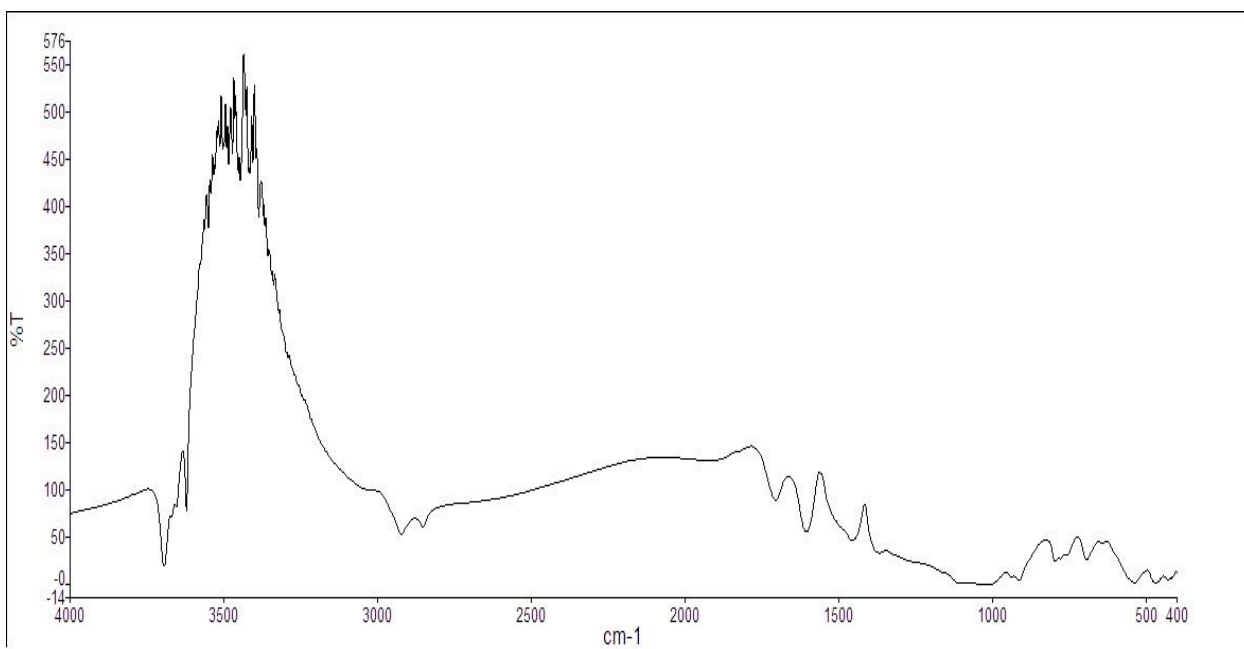
**Figure 4.7: FTIR Transmittance spectra for MCL – 4**



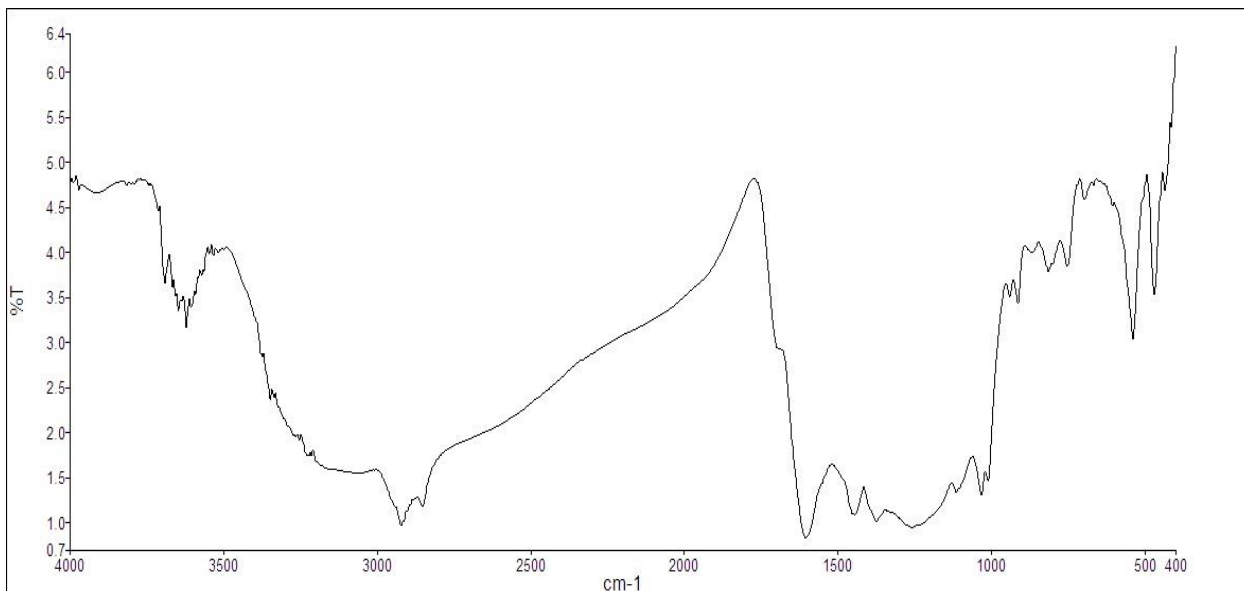
**Figure 4.8: FTIR Transmittance spectra for MCL – 5**



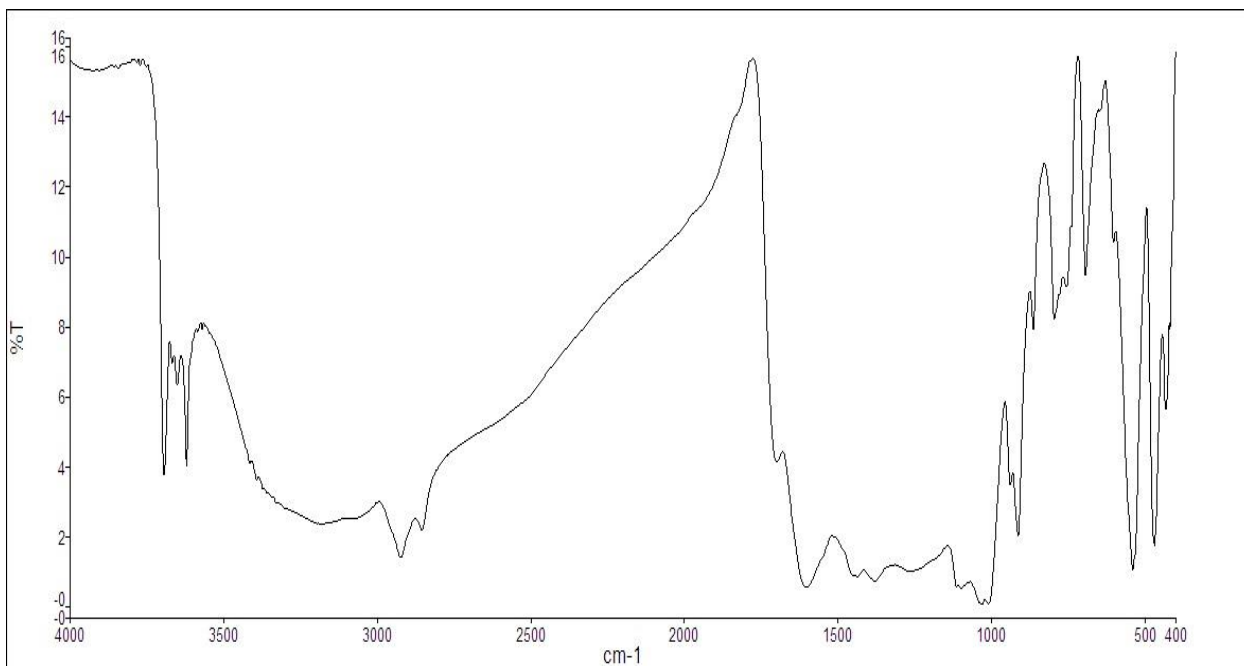
**Figure 4.9: FTIR Transmittance spectra for MCL - 6**



**Figure 4.10: FTIR Transmittance spectra for CCL - 1**

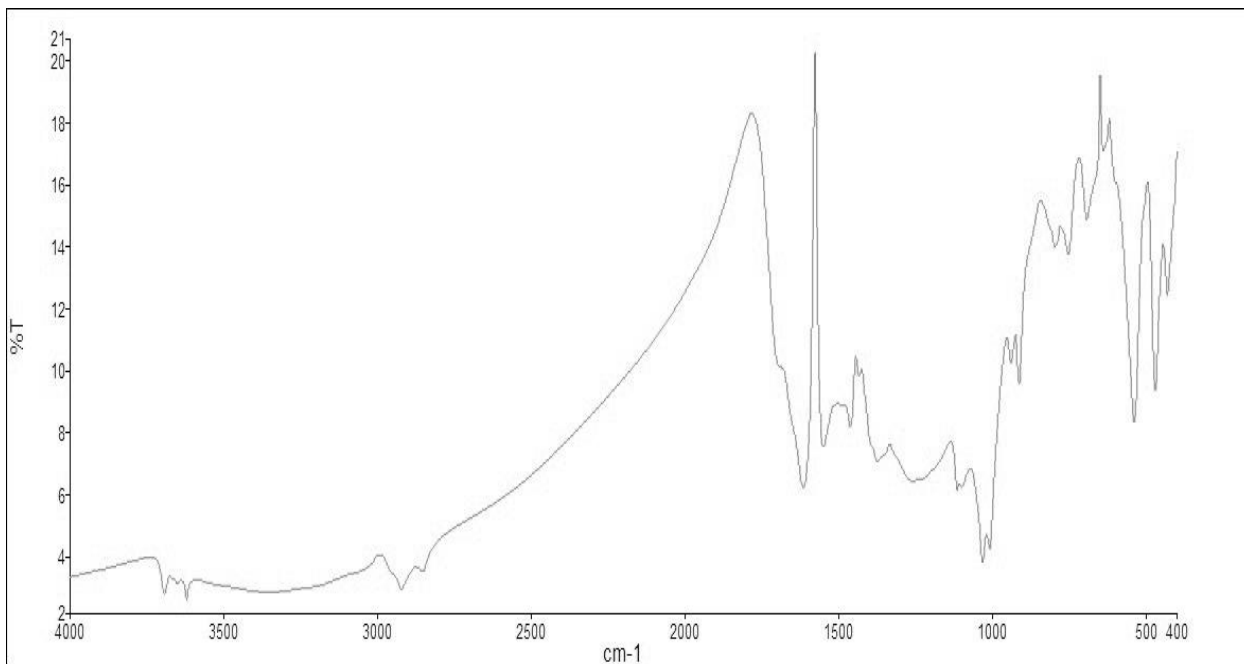


**Figure 4.11: FTIR Transmittance spectra for CCL – 2**

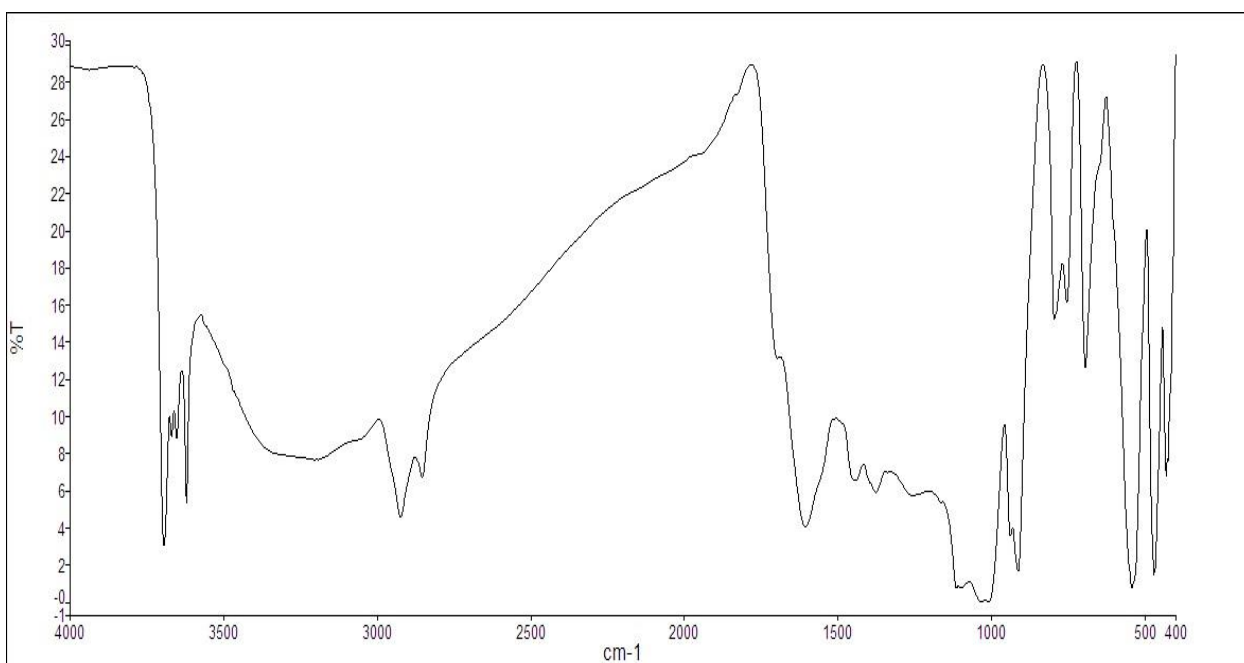


**Figure 4.12: FTIR Transmittance spectra for BCCL – 1**

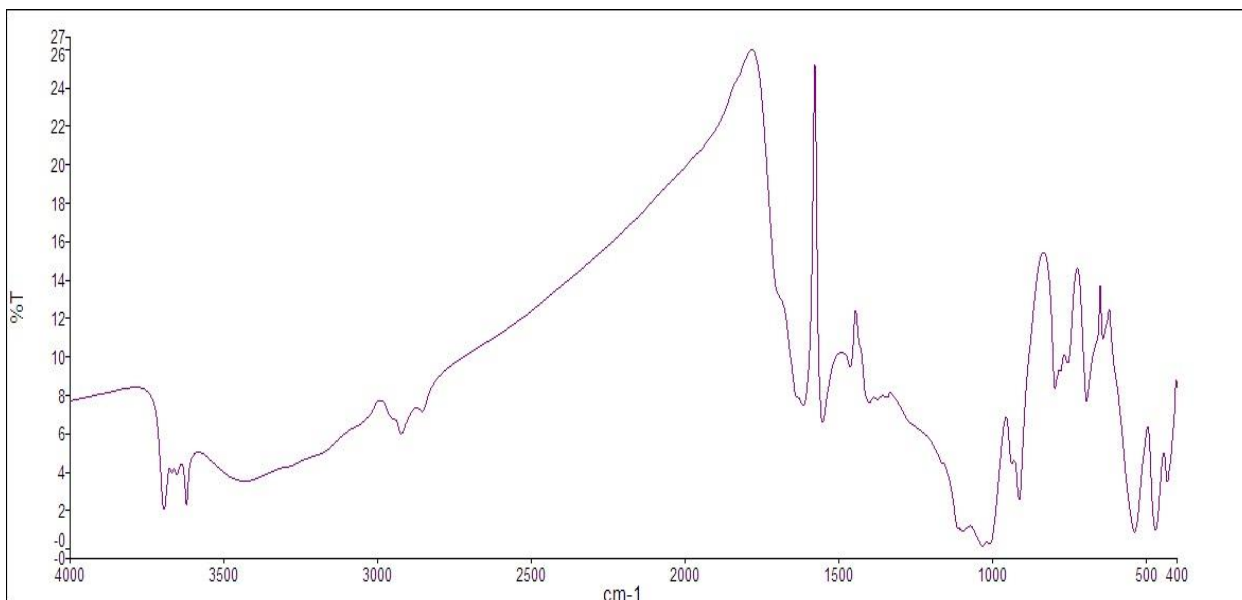




**Figure 4.13: FTIR Transmittance spectra for WCL - 1**



**Figure 4.14: FTIR Transmittance spectra for NECL - 1**



**Figure 4.15: FTIR Transmittance spectra for SCCL - 1**

Absorbance, A, is the amount of light absorbed from the initial incoming light. Absorbance is the base 10 logarithm of the reciprocal of transmittance.

$$A = \log_{10} \frac{1}{T}$$

Where, A is absorbance and T is transmittance in fraction.

### Beer-Lambert Law

The Beer-Lambert law gives the linear relationship between the absorbance and the concentration of absorbing species in the sample. The general law is:

$$A = a \cdot b \cdot c$$

Where,

a is the wavelength dependent absorvity coefficient

b is the path length

c is the concentration of species

Carbon aliphaticity is defined as:

$$F_{al} = \frac{\text{Absorbance at } 2925 \text{ cm}^{-1}}{\text{Absorbance at } 1600 \text{ cm}^{-1}}$$

2925 cm<sup>-1</sup> refers to the aliphatic bonds C-H while the 1600 cm<sup>-1</sup> refers to the aromatic bonds or conjugate bonds. The result of aliphaticity of all coal samples are samples are presented is given in table 4.4

**Table 4.4: Aliphaticity of Coal Samples**

Sl. No.	Sample Name	Aliphaticity
1	MCL - 1	1.515
2	MCL - 2	1.41
3	MCL - 3	1.057
4	MCL - 4	1.1
5	MCL - 5	0.918
6	MCL - 6	0.93
7	CCL - 1	1.109
8	CCL - 2	0.97
9	BCCL - 1	0.826
10	WCL - 1	1.355
11	NECL - 1	0.97
12	SCCL - 1	1.678

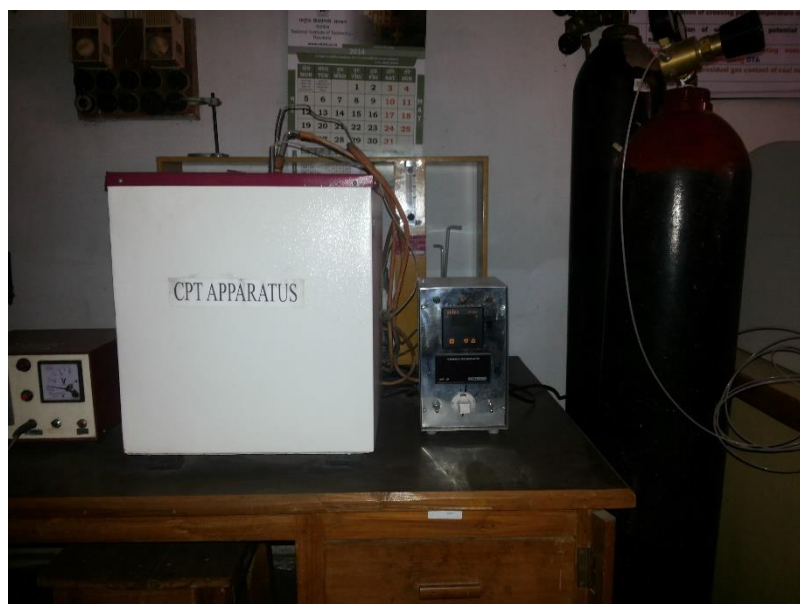
### 4.3 Determination of susceptibility indices

#### 4.3.1 Crossing Point Temperature (CPT)

The method involves heating of coal in an oxidizing atmosphere. The heating is programmed to increase the temperature at a fixed rate. The temperature at which the exothermic reactions taking place in coal becomes self-sustained is called as crossing point temperature. This is the point at which the temperature of coal exceeds the temperature of heating element. Lower is the CPT higher is the tendency of coal towards spontaneous heating.

#### Experimental Procedure

The apparatus consists of a reaction tube containing coal sample being heated inside a glycerin bath or an air oven. Glass wool is use to hold the coal sample in place. Oxygen is supplied at a rate of 80 ml/min. The oxygen enters the tube through a helical tube so that its temperature reaches that of heating media. A thermocouple is inserted inside the reaction tube to measure the temperature of coal. The photographic view of the CPT apparatus is give in fig. 4.16



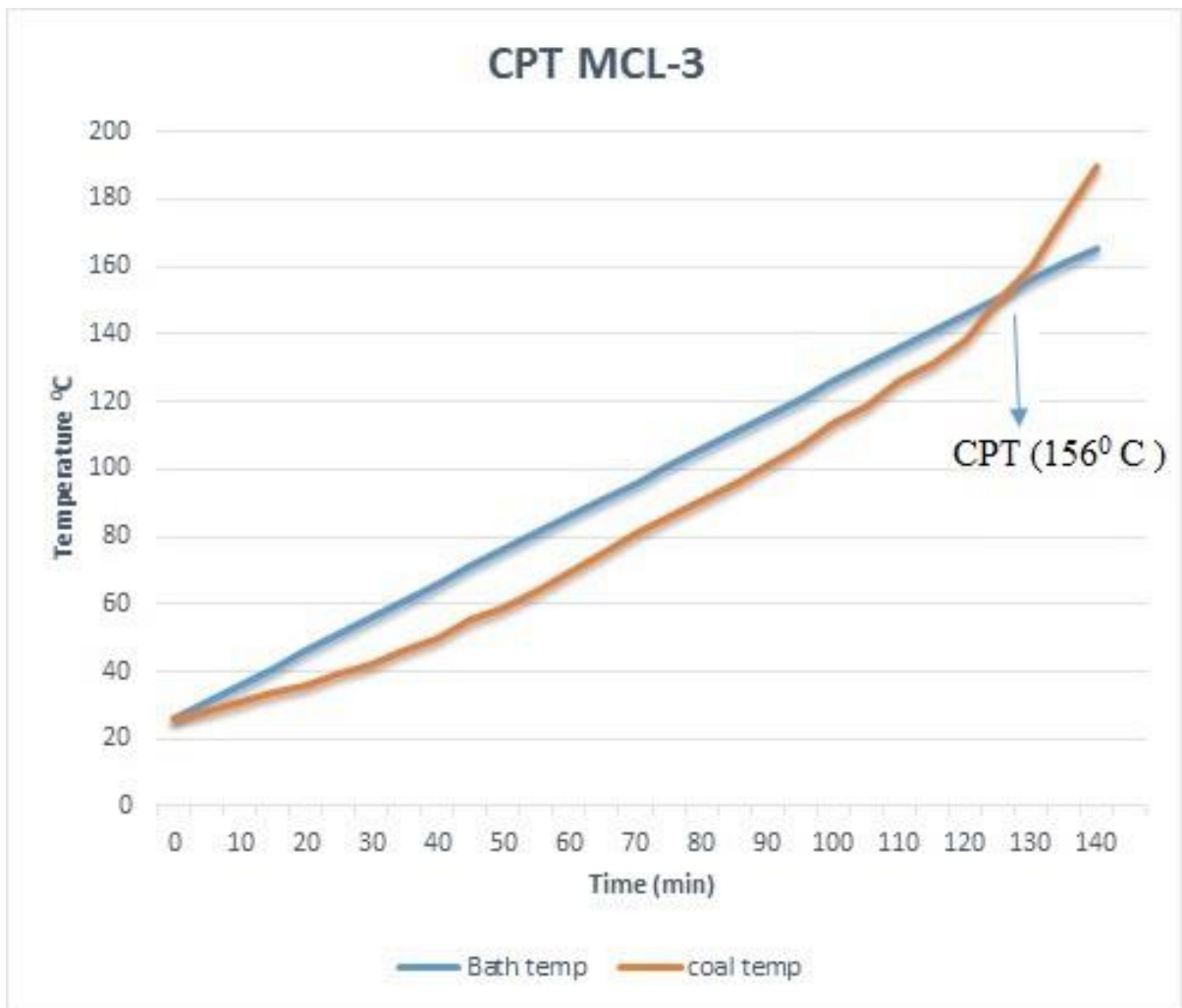
**Fig. 4.16: Photographic view of CPT Apparatus**

20gm coal sample of  $-212\mu$  size is kept inside the reaction tube and is supported on a glass wool. The sample is packed on top of glass wool by tapping the tube. The tube is closed with a cork with holes for inserting the thermocouple and a tube to provide an exit for the gases. The reaction tube was then kept inside a glycerin bath heated at a rate of  $5^{\circ}\text{C}$  per minute. Oxygen was supplied from the oxygen cylinder at the rate of 80 ml/min. The temperature of both glycerin bath and coal is recorded at an interval of 5 minutes. When the rate of temperature rise of coal starts to increase at a faster rate than that of glycerine bath from that point onwards start taking temperature readings at intervals of 1 min. After coal temperature exceeds the temperature of bath then the experiment is stopped.

A sample time vs. temperature plot for MCL – 3 coal has been presented in fig 4.17. The initial rate of coal temperature rise is very less. Initially the temperature of coal does not increase with bath's temperature because of the evaporation of water. Water evaporating from coal takes away the heat and the graph remains parallel to x-axis. After moisture removal coal temperature increases at a constant rate. After sometimes the rate of temperature increase in coal rises very rapidly and the coal temperature crosses the temperature of surrounding media. This point is known as crossing point temperature.

**Table 4.5: Crossing point temperature risk criteria (Ramlu et al., 1985)**

Crossing point temperature ( $^{\circ}\text{C}$ )	Risk
120-140	Highly liable to self- heating
140-160	Moderately liable to self- heating
160-180	Less liable to self-heating



**Fig 4.17: CPT plot for MCL -3 coal**

**Table 4.6: CPT values for coal samples**

Sl. No.	Sample Name	CPT (° C)
1	MCL - 1	145
2	MCL - 2	148
3	MCL - 3	156
4	MCL - 4	153
5	MCL - 5	149
6	MCL - 6	143
7	CCL - 1	145
8	CCL - 2	142
9	BCCL - 1	157
10	WCL - 1	160
11	NECL - 1	139
12	SCCL - 1	130

### 4.3.2 Wet Oxidation Potential Analysis

There are two types of molecules present inside coal viz. the aliphatic or hydro-aromatic structure and the condensed aromatic structure. Both molecules have different behavior. The aliphatic compounds are highly susceptible to spontaneous heating while the aromatic ones are less susceptible. Therefore a higher concentration of aliphatic compound or a lesser concentration of aromatic ones results in a high susceptibility of coal to spontaneous heating.

Higher rank coals contain a higher concentration of aromatic structure compounds while the lower rank coal have a higher aliphatic compound concentration. This is the reason why spontaneous heating susceptibility decreases with increase in rank of coal. The higher rank coals have a structure much closer to that of graphite. Due to this they undergo very less oxidation and produce very less potential difference.

Wet oxidation potential consists of reaction of coal with  $\text{KMnO}_4$  and  $\text{KOH}$ . The alkaline potassium permanganate solution oxidizes the coal and produces an emf. The solution acts like an electro-chemical cell and on stirring produces an emf against the standard emf of 0.56 V. The drop in emf is plotted against the time to get a measure of the susceptibility of coal to spontaneous heating.

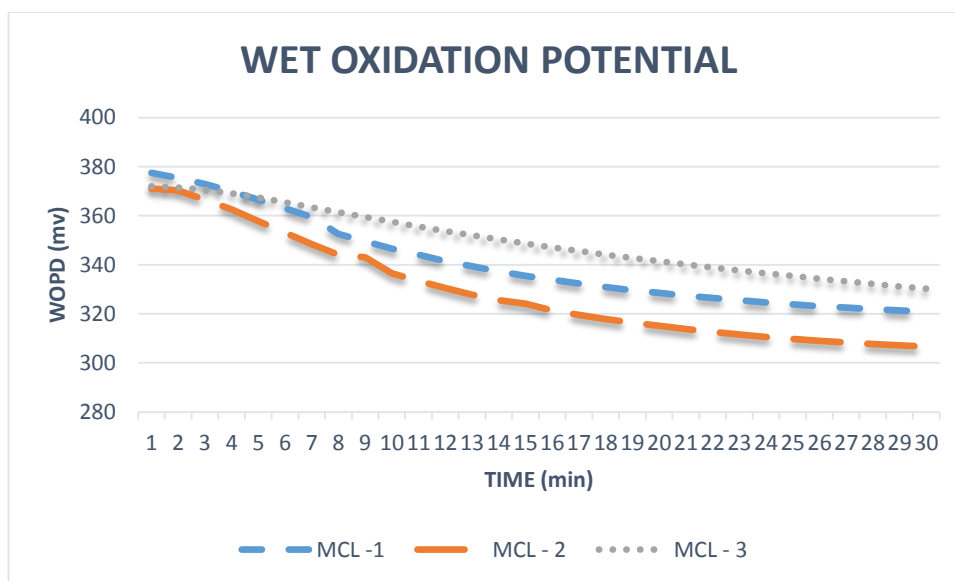
#### Experimental Procedure

A beaker containing 100 ml of deci-normal solution of potassium permanganate ( $\text{KMnO}_4$ ) in 1N potassium hydroxide ( $\text{KOH}$ ) solution is kept upon a magnetic stirrer to maintain a uniform composition. 0.5gm of -212 $\mu$  coal sample is mixed in the solution. Two electrodes one calomel and other carbon electrodes is kept inside the solution. The potential difference between the two electrodes is measured using milli-volt meter. The photographic view of wet oxidation potential apparatus is give in fig. 4.18. The measurement is done until a constant emf is attained. The emf is plotted against time to get an idea about the susceptibility of coal to spontaneous heating (Tarafdar and Guha 1984. The EMF vs. time plots have been presented below. It was observed that the constant emf was reached after 30 minutes for most samples therefore an observation time of 30 minutes was selected. Different coal samples produce different amount of potential drop and this can be used to compare their relative susceptibilities to spontaneous combustion. The WOPD after 30 minutes is as presented in table 4.7.

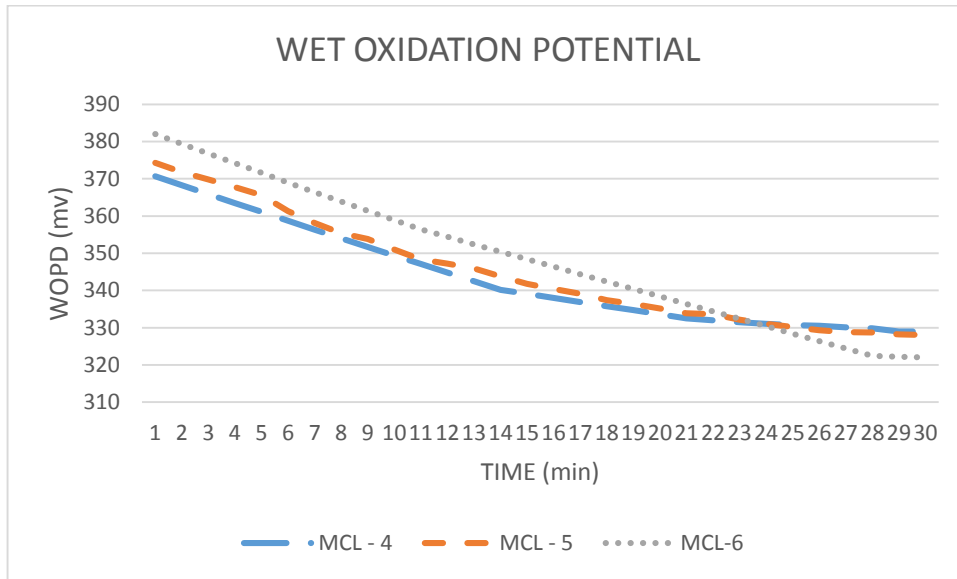


**Fig. 4.18: Photographic view of Wet Oxidation Potential Apparatus**

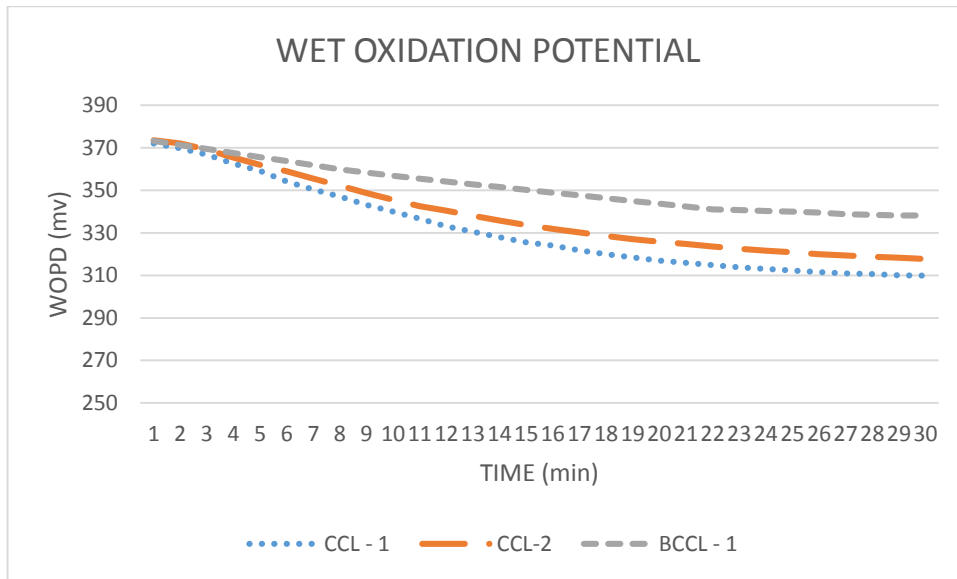
The plot of WOPD after 30 minutes of all the coal samples is presented in figures 4.19 to 4.22.



**Figure 4.19 Wet oxidation potential vs Time plot for MCL -1,2,3**

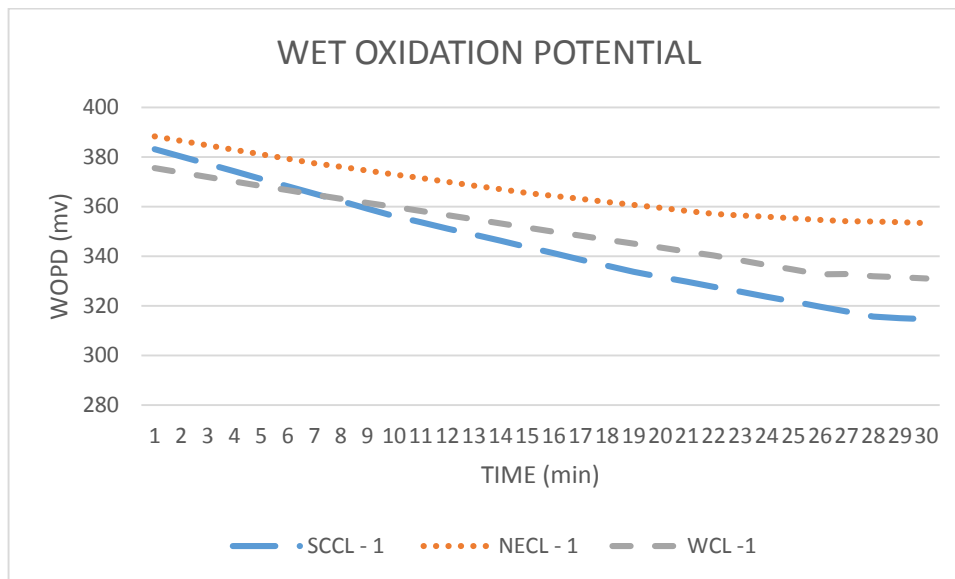


**Figure 4.20 Wet oxidation potential vs Time plot for MCL -4,5,6**



**Figure 4.21 Wet oxidation potential vs Time plot for BCCL-1 AND CCL -1,2**





**Figure 4.22 Wet oxidation potential vs Time plot for NECL – 1, SCCL – 1, WCL - 1**

**Table 4.7: WOPD for coal samples**

Sl. No.	Sample Name	WOPD after 30 minutes (mv)
1	MCL - 1	56.7
2	MCL - 2	64.4
3	MCL - 3	41.8
4	MCL - 4	41.7
5	MCL - 5	46.3
6	MCL - 6	60
7	CCL - 1	62.1
8	CCL - 2	56
9	BCCL - 1	35.1
10	WCL - 1	44.4
11	NECL - 1	35
12	SCCL - 1	68.3

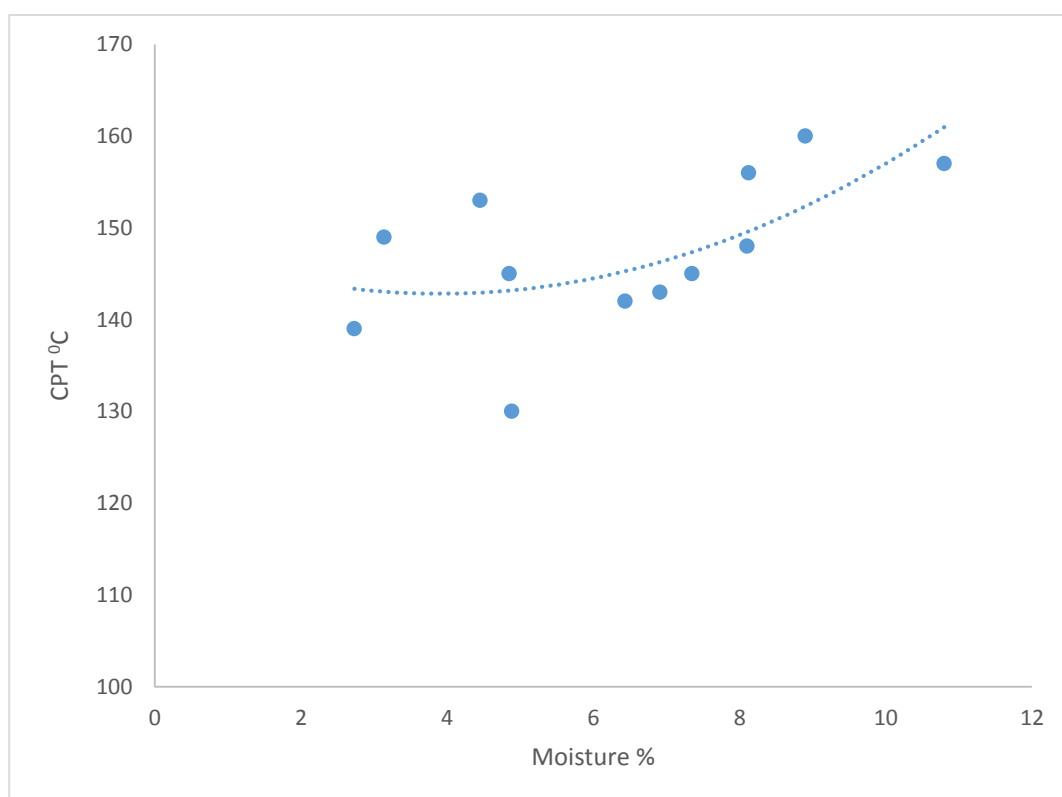
## **CHAPTER 5**

### **CORRELATION ANALYSIS**

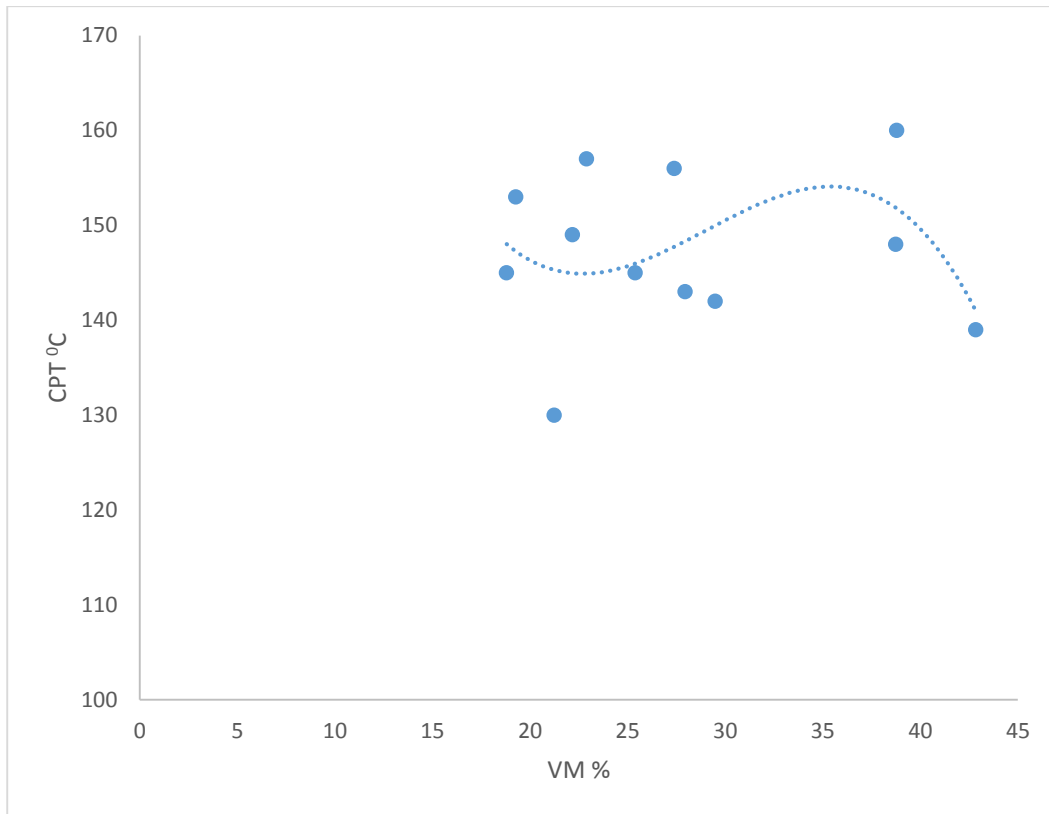
## 5. CORRELATION ANALYSIS

In order to analyze the relationship between the intrinsic properties of coal and the susceptibility indices correlation study was done. Correlation was done using the susceptibility indices like wet oxidation potential and crossing point temperature as dependent variable and moisture, ash, volatile matter, calorific value and aliphaticity as dependent variables. Multi-variate correlation was also done to get further knowledge of effect of intrinsic properties on susceptibility indices.

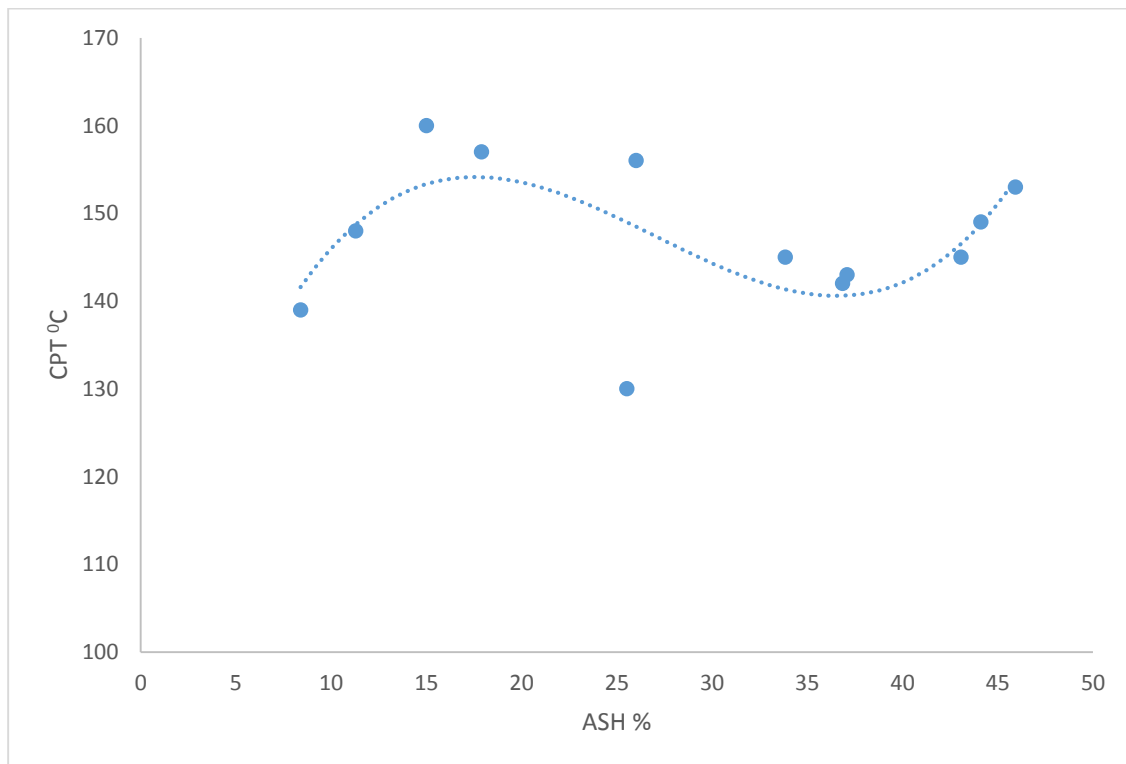
The correlation was done for CPT with moisture, volatile matter, ash, calorific value and aliphaticity. Also correlation of wet oxidation potential with moisture, volatile matter, ash, calorific value and aliphaticity. Multi-variate correlation was attempted for both the wet oxidation potential and CPT. The plots are constructed with independent variables on horizontal axis and dependent on vertical axis and are as presented in figures 5.1 to 5.10. The summarized correlation table is presented in table 5.1 and 5.2 respectively.



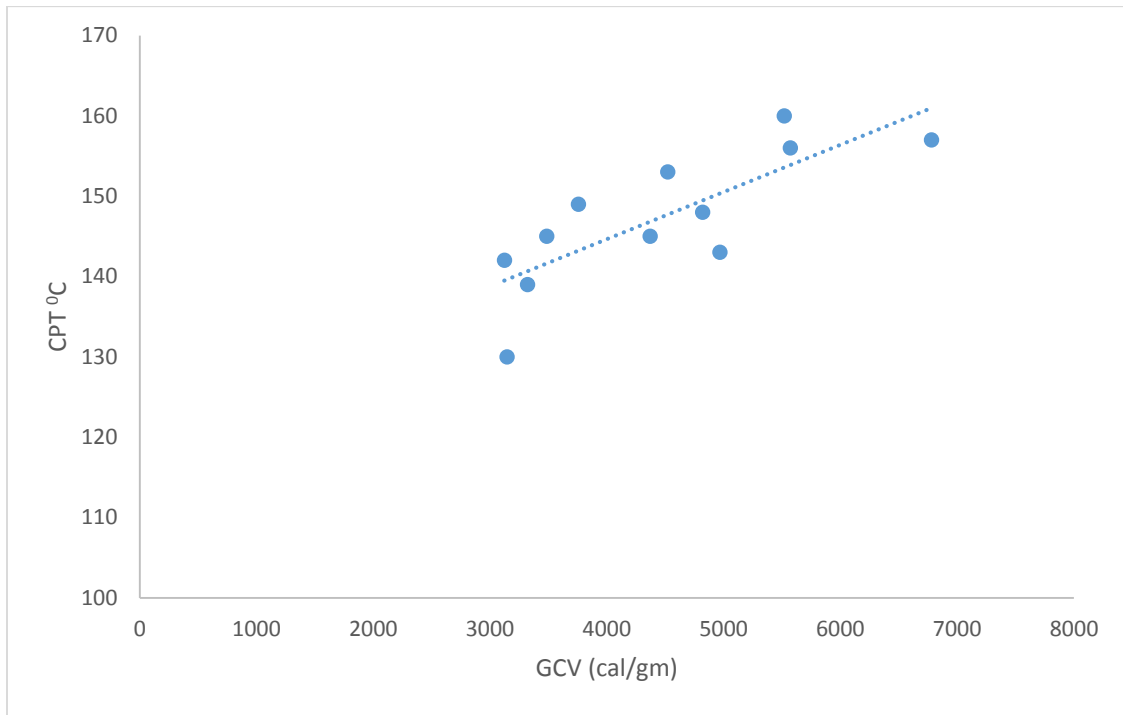
**Fig. 5.1 Correlation plot between CPT and moisture content**



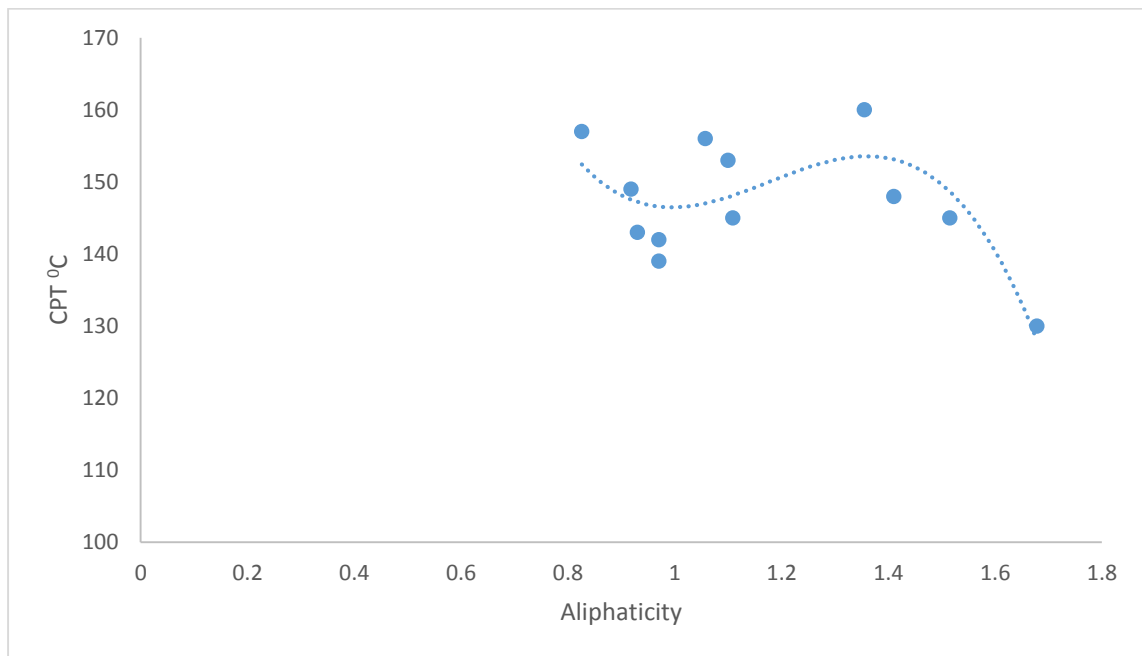
**Fig. 5.2 Correlation plot between CPT and volatile matter content**



**Fig. 5.3 Correlation plot between CPT and Ash content**



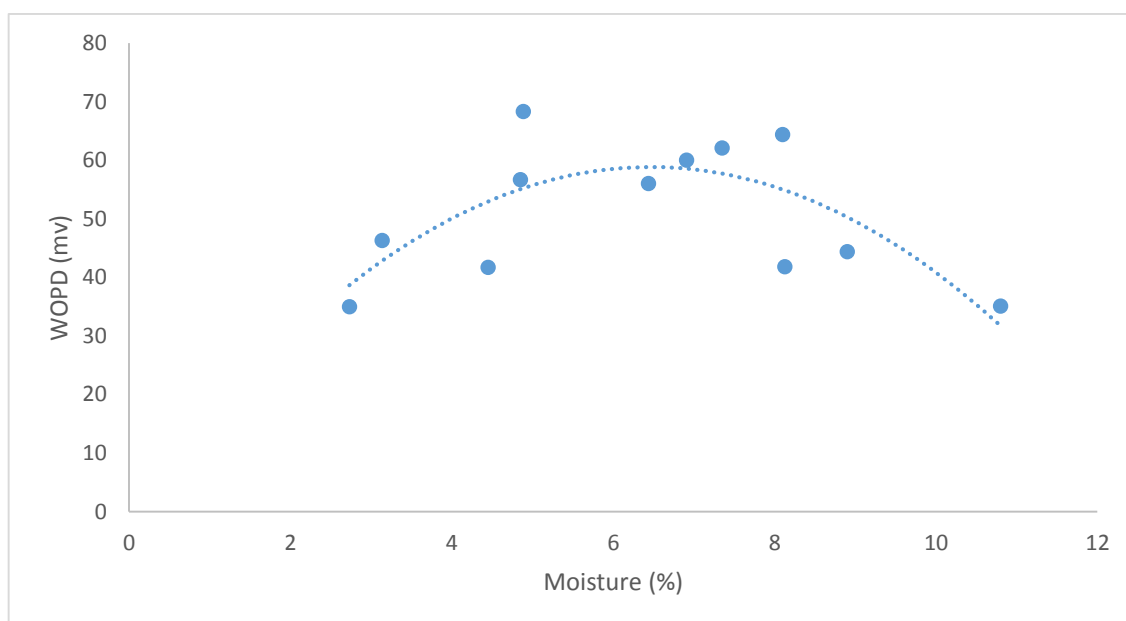
**Fig. 5.4 Correlation plot between CPT and Gross Calorific Value**



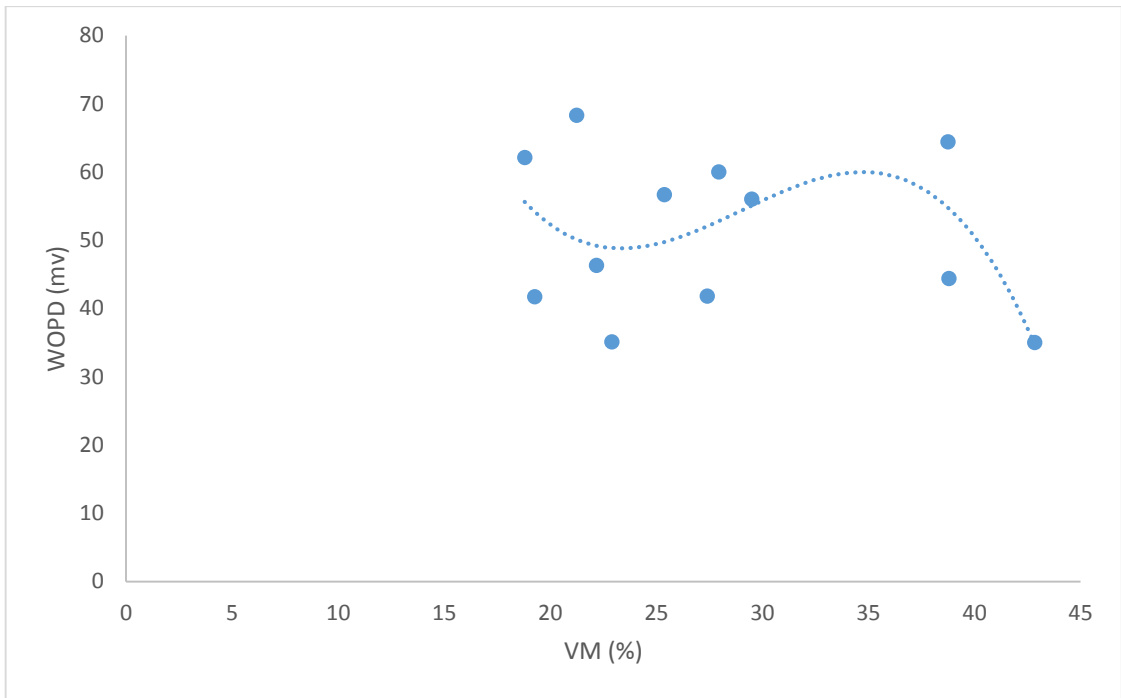
**Fig. 5.5 Correlation plot between CPT and Aliphaticity**

**Table 5.1: Correlation between Crossing Point Temperature (CPT) and Intrinsic Properties**

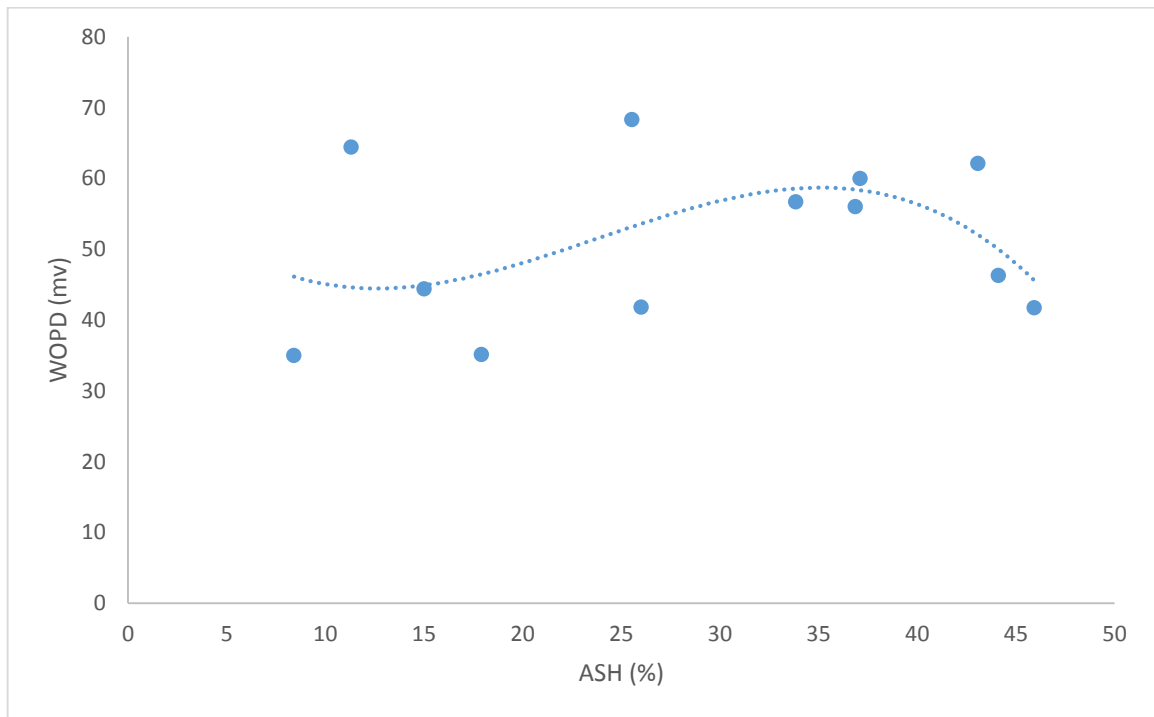
Sl. No.	Independent Variables	Empirical Relation	Correlation Coefficient
1.	M	$CPT = 0.3819M^2 - 2.9821M + 148.64$	0.3976
2.	V.M.	$y = -0.0089VM^3 + 0.7784VM^2 - 21.531VM + 337.02$	0.1347
3.	A	$CPT = 0.004A^3 - 0.3216A^2 + 7.6298a + 97.859$	0.3678
4.	CV	$y = 0.0059x + 121.12$	0.6272
5.	F <sub>al</sub>	$CPT = -290.95 F_{al}^3 + 1027.8 F_{al}^2 - 1181.2 F_{al} + 590.84$	0.5955
6.	M, V.M, A, CV, F <sub>al</sub>	$CPT = 0.147104 M + 0.431 VM + 0.3418 A - 2.01669 F_{al} + 0.0068 CV$	0.741188



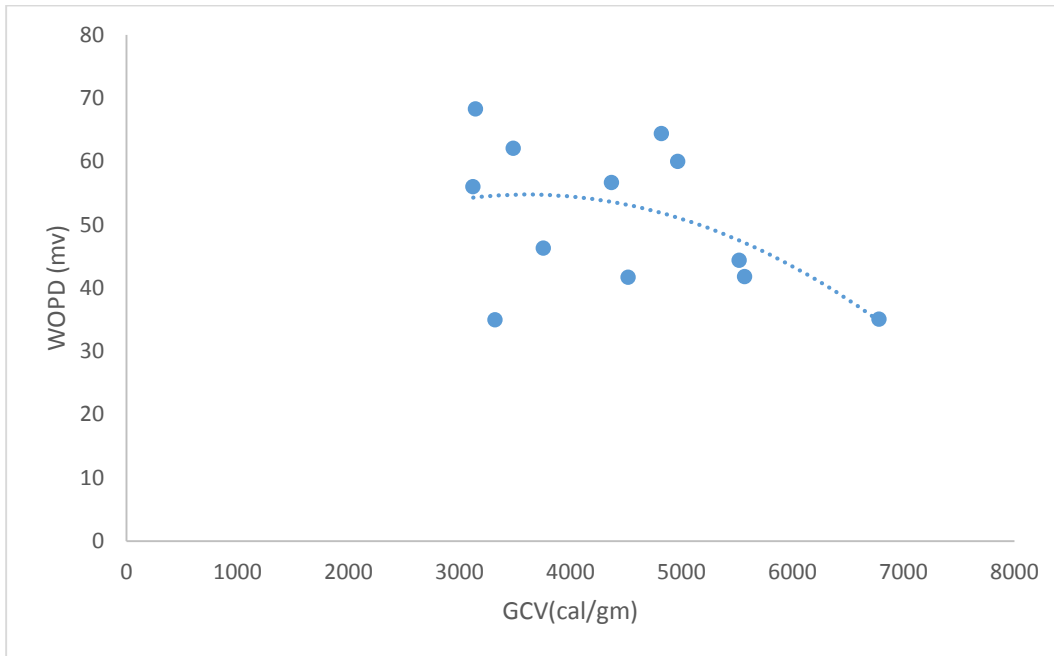
**Fig. 5.6 Correlation plot between WOPD and moisture content**



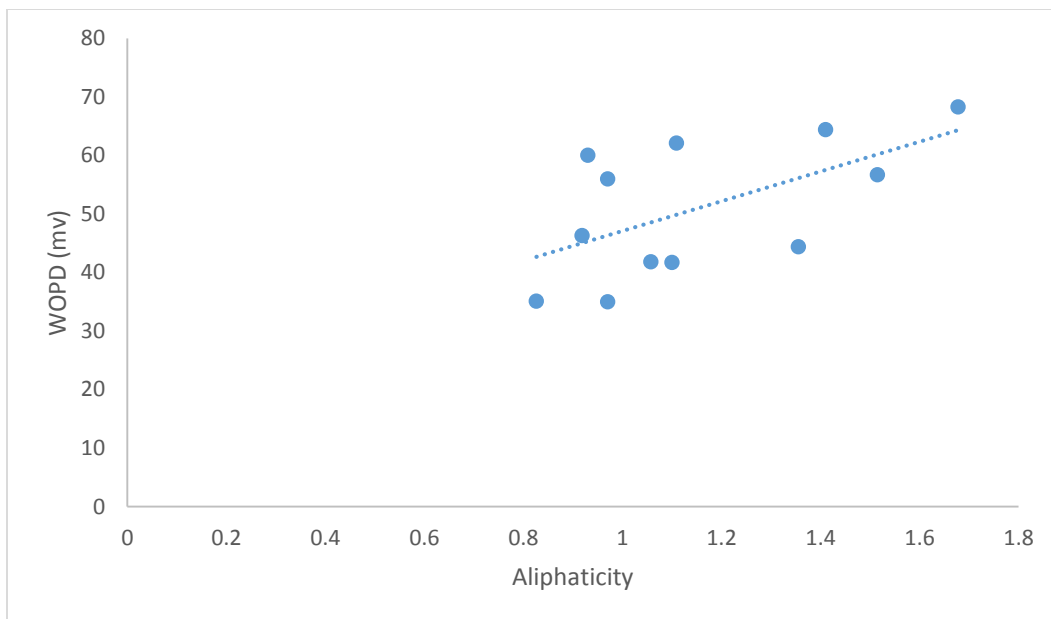
**Fig. 5.7 Correlation plot between WOPD and Volatile matter content**



**Fig. 5.8 Correlation plot between WOPD and Ash content**



**Fig. 5.9 Correlation plot between WOPD and GCV**



**Fig. 5.10 Correlation plot between WOPD and Aliphaticity**



**Table: 5.2 Correlation between wet oxidation potential difference (WOPD) and Intrinsic Properties**

Sl. No.	Independent Variable	Empirical Relation	Correlation Coefficient
1.	M	$WOPD = -1.4453M^2 + 18.694M - 1.5983$ $R^2 = 0.5552$	0.5552
2.	V.M.	$WOPD = -0.0152 V.M.^3 + 1.3248 V.M.^2 - 36.995 V.M. + 383.95$	0.2397
3.	A	$WOPD = -0.0025x^3 + 0.179x^2 - 3.3206x + 62.88$	0.2227
4.	CV	$WOPD = -43.404 CV + 6661.5$	0.1944
5.	F <sub>al</sub>	$WOPD = 25.366 F_{al} + 21.732$	0.3491
6.	ALL	$WOPD = 2.621 M - 0.758 VM - 0.145 A - 0.00499 CV + 10.851 F_{al}$	0.735

## **CHAPTER 6**

### **DISCUSSION AND CONCLUSION**

## 6. DISCUSSION AND CONCLUSION

### 6.1 Discussion

Twelve coal samples collected from different mines in India were analyzed. Their Intrinsic properties and susceptibility indices were determined. The samples were collected from following mines 2 samples belonged to CCL, 1 sample from SCCL, 1 from NCL, 6 from MCL, 1 from WCL and 1 from NECL. Their intrinsic properties were correlated with spontaneous heating susceptibilities. In the current work an attempt has been made to correlate the intrinsic properties of coal with that of their spontaneous tendency. Twelve coal samples from different coalfields of India were collected for the purpose. 6 samples were collected from MCL, 2 from CCL and 1 each from BCCL, NECL, WCL and SCCL.

The intrinsic properties were determined by proximate analysis, Aliphaticity from Fourier transform infrared spectroscopy (FTIR) and Gross calorific value (GCV) using Bomb calorimetry. It was noted that the maximum moisture content was found to be 10.8% (in BCCL-1) and the lowest was found to be 2.73% (in NECL -1). The more the moisture content higher is the susceptibility to self-heating. However, the relationship is not linear from the WOPD and moisture correlation it can be seen that the susceptibility first increases than after certain point decreases.

The volatile matter was highest 42.85% in NECL-1 Sample and lowest in 18.8 % in CCL-1 sample. The NECL-1 coal has highest volatile matter content followed by MCL and BCCL. The MCL coals have high to medium volatile matter content.

The maximum ash content is in MCL- 4 coal (45.93%) and lowest ash 8.4% in NECL-1 sample. The mean ash content is 28.29%. In ash also the susceptibility first increase then decreases with concentration.

The BCCL – 1 sample has the highest Gross calorific value with 6780.51 cal/gm. Lowest calorific value is of CCL-2 sample with 3123.503 cal/gm. MCL coals have medium calorific value while WCL-1 has high calorific value.

The Aliphaticity varied from 0.826 for BCCL-1 to 1.678 for SCCL-1. Thus the coal structure of SCCL-1 contains a high number of aliphatic bonds as compared to aromatic bonds. This makes

the SCCL-1 sample to be highly susceptible to spontaneous heating which is corroborated by CPT value.

The susceptibility indices were determined using CPT and WOPD analysis. The CPT value of SCCL-1 coal was the least at 130<sup>0</sup>C and Highest CPT was observed in WCL-1 160<sup>0</sup> C. This shows that the SCCL-1 sample is the most susceptible to spontaneous heating while the WCL-1 sample is the least susceptible. NECL-1 and SCCL-1 have CPT less than 140<sup>0</sup> C making them highly susceptible to spontaneous heating. All other coal lie between 140<sup>0</sup> -160<sup>0</sup> C and are therefore moderately susceptible.

The wet oxidation potential difference was maximum for SCCL-1 (68.3 mv) and lowest for NECL-1 (35 mv). Some MCL coal samples (MCL 1, 2, 6) and SCCL-1 has high susceptibility to spontaneous heating while other samples have medium to low susceptibility.

The susceptibility indices were correlated with the intrinsic properties and a brief overview of the correlation is presented in the table below:

**Table 6.1 Correlation coefficients for correlation between Intrinsic Properties and Susceptibility Indices**

Sl. No.	Intrinsic Properties	Correlation Coefficient CPT	WOPD
1	Moisture	0.3976	0.5552
2	V.M.	0.1347	0.2397
3	ASH	0.3678	0.2227
4	GCV	0.6272	0.1944
5	Aliphaticity	0.5955	0.3491
6	All	0.7412	0.735

## 6.2 Conclusion

From the results of susceptibility indices it can be concluded that the coal sample SECL-1 and NECL-1 are the most susceptible to spontaneous heating. This is supported by both CPT and Wet oxidation potential difference.

The correlation of intrinsic properties with the susceptibility indices showed that the properties like moisture and ash content have good correlation with both CPT and WOPD. This is consistent with earlier works of other researchers. Aliphaticity also showed a good correlation with both the indices used and therefore it is concluded that it can be used for assessment of spontaneous heating liability of coal. The use of aliphaticity over other properties for prediction of spontaneous heating liabilities is also supported by the fact that the process of measuring aliphaticity by FTIR analysis is very accurate and results are reproducible.

Both the susceptibility indices used for assessment of spontaneous heating liability showed almost similar correlation with intrinsic properties. Therefore, it is concluded that both are equally relevant in predicting spontaneous heating liabilities.

It was also observed that the correlation increased dramatically when multiple linear regression was used to correlate all intrinsic properties with liability indices. The high correlation obtained as compared to correlation obtained from simple linear regression indicates that use of single intrinsic property to predict the coal's liability to spontaneous heating may not be suitable. Therefore it is concluded that to get a more accurate indication of spontaneous heating liability it may be better to use multiple parameters instead of a single one.

## **CHAPTER 7**

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