

RELATION OF PROXIMATE ANALYSIS WITH DTA DUE TO SPONTANEOUS HEATING OF SOME INDIAN COALS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY

IN

MINING ENGINEERING

BY

SASHIDHAR REDDY BASIREDDY

Roll No. 109MN0605



DEPARTMENT OF MINING ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA – 769008

(2012-2013)

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**National Institute of Technology
Rourkela**

CERTIFICATE

This is to certify that the thesis entitled **Relation of proximate analysis with DTA due to spontaneous heating of some Indian coals** being submitted by Sashidhar Reddy Basireddy (Roll No. 109MN0605) to National Institute of Technology, Rourkela; for the award of degree of Bachelor of Technology in Mining Engineering is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter substantiated in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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ABSTRACT

The auto oxidation of coal ultimately leads to spontaneous combustion which is the major root cause for the disastrous of coal mine. It has been a major problem in the leading producing coal countries like Australia, India and China. Therefore the assessment for this combustion is very much necessary. It depends upon different characteristics and properties of coal. Once if the combustion of coal has been occurred, it is very difficult to control which also disturbs the environment of the surroundings of the mine. The spontaneous heating susceptibility of different coals varies over a wide range and it is important to assess their degree of proneness for taking preventive measures against the occurrence of fires to avoid loss of lives and property, sterilization of coal reserves and environmental pollution and raise concerns about safety and economic aspects of mining etc.

This B.Tech dissertation deals with the relation of proximate analysis of coal with DTA-TG and due to the spontaneous heating tendency of some Indian coal. Eight insitu coal samples for the project were collected from SECL and SCCL mines, both from opencast as well as underground workings. The project deals with determination of spontaneous heating susceptibility of coal samples by Differential thermal analysis and its relation with proximate analysis of coal samples. The intrinsic properties as well as susceptibility indices of the coal samples were determined by following experimental techniques:

- Proximate analysis
- Differential thermal analysis (DTA-TG)

It was observed that the Proximate analysis values can be obtained by using small amount of sample and the volatile matter increases with the reduction in the weight of sample and the Transition temperature obtained from the DTA – TG plot cannot be taken as a sole parameter to assess the spontaneous heating of coal, rather Stage IIB and Stage II slopes give a better idea.

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CHAPTER-1

INTRODUCTION

1.1 Background

Coal is source of about 27% of world's primary energy consumption and it accounts for about 34% of electricity generated in the world, so much attention has been focused in recent years on coal as an alternative source of energy. Coal is the dominant energy source in India and meets 55% of country's primary commercial energy supply. Mine fires in Indian coalfields is generally caused by spontaneous combustion of coal despite various preventive technologies being adopted. The spontaneous heating of coal varies over a wide range and it is important to assess their degree of proneness for taking preventive measures against the occurrence of fires to avoid loss of lives and property, sterilization of coal reserves and environmental pollution and raise concerns about safety and economic aspects of mining [20].

In order to find out the susceptibility of coal to spontaneous heating different methods have been adopted by various researchers of the world. A number of experiments have been done for assessing the spontaneous heating susceptibility of coal viz., Crossing point temperature method, Wet oxidation potential method, Differential thermal analysis, Flammability temperature. A number of approaches have been developed over the years to assess the proneness of coal to spontaneous heating. This propensity to self heating of coal also decide the incubation period of coal seam, which decide the size of the panel to be formed, which is a most important safety measure in mine planning. It is therefore imperative that the planners of a mine determine in advance the spontaneous heating susceptibility of the seam/seams to be mined so that either the coal has been extracted before the incubation period, or advance precautionary measures are planned to tackle this menace. The methods used to assess the tendencies of coals to spontaneous heating in the present study are proximate analysis, Differential thermal analysis (DTA - TG) [20].

1.2 General

Coal has been a useful resource for years. It is primarily burned for the production of electricity and/or heat, production of chemicals and is also used for industrial purposes, such as refining metals. At least 40% of the world's electricity comes by coal, and in 2012, about one-third of the United States' electricity came from coal, down from approximately 49% in 2008. It is the largest source of energy for the generation of electricity worldwide. India has some of the largest coal reserves in the world (approx. 267 billion tonnes). The energy derived from coal in India is about twice that of energy derivative from oil, whereas worldwide; energy derived from coal is about 30% less than energy origin from oil. The top coal producing states are Orissa, Jharkhand, Chhattisgarh, West Bengal, Bihar, Andhra Pradesh and Madhya Pradesh. World coal consumption was about 7.25 billion tonnes in 2010 (7.99 billion short tons) and is expected to increase 48% to 9.05 billion tonnes (9.98 billion short tons) by 2030. China produced 3.47 billion tonnes (3.83 billion short tons) in 2011. India produced about 578 million tonnes (637.1 million short tons) in 2011. 68.7% of China's electricity comes from coal. The USA consumed about 13% of the world total in 2010, i.e. 951 million tonnes (1.05 billion short tons), using 93% of it for generation of electricity. 46% of total power generated in the USA was done using coal [8].

It has been estimated that there are over 861 billion tonnes of proven coal reserves worldwide. This means that there is enough coal to last us around 112 years at current rates of production. Coal reserves are available in almost every country worldwide, with recoverable reserves in around 70 countries. The biggest reserves are in the USA, China and India [8].

Coal mine fire is a major problem for the coal mining industry worldwide. Coal mine fires can be divided into near-surface fires, in which fire extend to the surface and the oxygen required for their ignition comes from the atmosphere, and fires in deep underground mines, where the oxygen comes from the ventilation. Some fires along coal seams are natural occurrences. Some coals may self-ignite at temperatures as low as 40 °C (104 °F) for brown coal in the right conditions of moisture and grain size. The fire usually begins a few decimeters inside the coal at a depth in which the permeability of the coal allows the inflow of air but in which the ventilation does not remove the heat which is generated. Major cause of these fires in different coalfields is spontaneous heating of coal. Globally, thousands of inextinguishable mine fires are burning, especially in China. The spontaneous combustion of coal is responsible for the majority of underground fires in South African collieries. Incidents

of spontaneous combustion are also preventing in open cast collieries and its stockpiles dicers dumps, train trucks and ships [9].

Spontaneous heating of coal occurs by self heating (increase in temperature due to exothermic internal reactions), followed by thermal runaway (self heating which rapidly accelerates to high temperatures) and finally, ignition. When coal is exposed to air, some of its exposed parts absorb free oxygen at a faster rate than others and oxidation results in the evolution of several gases such as CO CO₂, etc, water vapor, some heat. This takes place at ambient temperatures which leads to open fires [10].

Coal mine fire inside mine creates a lots of problems inside mines such as damage of expensive machines kept in mines, loss of life and property, environmental pollution, various health hazards,, roof fall, damage to the support systems and roadways such as haulage systems etc. These fires occurring every year are a function of the quality of the coal and the circumstances which it is subjected. The first fire was reported in Raniganj Coalfields in 1865. Many fires have been reported from Jharia and Raniganj coal fields having superior non-cocking coal. In 2010, 68 fires were burning beneath a 58-square-mile (150 km²) region of the Jhairia coalfield in Dhanbad, Jharkhand. This region has also been prone to mine fires. These fires continue to spread to adjoining areas s rapidly destroying the only source of prime coking coal in the country. To adapt some preventive measures against these fires, many researches were carried out to understand the mechanism of spontaneous heating of coal and to run the process of production of coal without any interruption and to save life, property and environment. A proper assessment of the spontaneous heating susceptibility of coal is done so that mine operators are notified well in advance so that production activities could be planned within the incubation period. Different methods have been adopted by various researchers of the world to find out the susceptibility of coal to spontaneous heating based on the measurement of oxidation rate and ignition temperature viz., Crossing point temperature method, Wet oxidation potential difference method, Differential thermal analysis, Differential Scanning Calorimetry Technique and Critical Air Blast [10].

1.3 Objectives of the project

The objectives of the project is to find out the relation of proximate analysis of coal with DTA due to spontaneous heating of coal in order to find out the mostly affected collected

coal sample susceptible to spontaneous heating. The project was divided into the following steps to achieve the above said objective –

- **Literature review** – collection of all the past works done by various academicians/researchers/scientists both national and international.
- **Sample collection and preparation** – Eight samples were collected from SCCL and SECL, for the purpose of analysis and the samples were collected and prepared as per the Indian Standards.
- **Experimentation** – the experimentation part divided into two stages:
 1. Determination of intrinsic properties of coal – proximate analysis.
 2. Determination of susceptibility indices of coal – differential thermal analysis.
- **Analysis** – Different graphs have been plotted between the intrinsic properties and susceptibility indices and the interpretation has been carried out on the basis of graphs and its relation.

CHAPTER – 2

LITERATURE REVIEW

2.1 Coal mine fires

Mine fires are associated mostly with coal mines, though fires in pyrite mines and occasional timber fires in certain metal mines are not unknown. Mine fires are common occurrences in coal mines but are rare in metal mines. Mine fires can be caused either by spontaneous heating, explosion of gases, electrical failures and blasting. In coal mines the major cause of mine fire is spontaneous heating of coal. An analysis of the causes of coal mine fires reveal that they may start either from open fires over the external mining agencies or originate due to very nature of coal. The propensity of coal liberating heat when come in contact with oxygen of air and its poor thermal conductivity favoring heat accumulation, may give rise to latter kind of heating. The former type of fire from external agencies is known as Exogenous Fires and the latter type i.e. due to self-heating characteristics of coal is called Endogenous Fires or Spontaneous Combustion [22].

Endogenous fires [22]

1. Pyrite fires: The iron ore of pyrite having chemical formula of FeS_2 is a polysulphide of iron. Pure pyrite contains 46.37% Fe and 53.33% S. as with coal pyrite also reacts with oxygen of air at room temperature liberating heat which under favorable conditions of heat accumulation gives rise to spontaneous fires. Susceptibility to spontaneous heating of pyrites is much less than that of coal but it increases if carbonaceous materials are present in pyrites. There are instances when pyrites with 5 - 6 % C and 10 – 12 % S have caught fire due to spontaneous heating (Amjhore Field, India).
2. Endogenous heating timber: Decayed timber may under extremely favorable conditions give rise to spontaneous heating believed to be mainly from bacterial origin.

Exogenous fires [22]

Electricity is one of the important causes of mine fires. It may originate from short circuiting, over heating of machines, electric bulbs, candles, flames from fires or explosion while blasting and ignition of inflammable materials like timber, oil or wastes. At times crushing of sulphide ores or fires from surface may also be the origin.

2.2 History of coal mine fires

Self-heating of coal leading to spontaneous combustion is the most significant cause of fires in coal mines across the world. Self heating of coal can occur in underground mines, opencast mines, coal stockpiles, transportation and during the disposal of wastes from coal using industries in heap wastes [17].

World scenario [18]

Up to 10 coal fires per year in the Ruhr area of Germany are caused by spontaneous heating. In china underground coal fires are widespread within a region stretching 5000 Km east – west and 750 Km north – south. It is assumed that fires in northern China consume an estimated amount of 100 – 200 MT of underground coal which is about 2 – 3 % of world CO₂ production. Surveys in the West Riding of Yorkshire (England) showed that 45 of the county's 153 collieries were on fire in 1931. A more recent example is the spontaneous combustion of spoil heaps at Middleburg colliery in Witbank coalfield in South Africa.

China

In China, the world's largest coal producer with an annual output around 2.5 billion tons, coal fires are a serious problem. It has been estimated that some 10-20 million tons of coal uselessly burn annually, and that the same amount again is made inaccessible to mining. They are concentrated in the provinces of Xinjiang, Inner Mongolia and Ningxia. Beside losses from burned and inaccessible coal, these fires contribute to air pollution and considerably increased levels green house gas emissions and have thereby become a problem which has gained international attention.

Germany

In Planitz, now a part of the city of Zwickau, a coal seam that had been burning since 1476 could only be quenched in 1860. In Dudweiler (Saarland) a coal seam fire ignited around 1668 and is still burning today. Also well-known is the so-called Stinksteinwand (stinking stone wall) in Schwalbenthal on the eastern slope of the Hoher Meibner, where several seams caught fire centuries ago after lignite coal mining ceased; combustion gas continues to reach the surface today.

Indonesia

Coal and peat fires in Indonesia are often ignited by forest fires near outcrop deposits at the surface. No accurate count of coal seam fires has been completed in Indonesia. Only a minuscule fraction of the country has been surveyed for coal fires. The best data available come from a study based on systematic, on-the-ground observation. In 1998, a total of 125 coal fires were located and mapped within a 2-kilometer strip either side of a 100-kilometer stretch of road north of Balikpapan to Samarinda in East Kalimantan, using hand-held Global Positioning System (GPS) equipment. Extrapolating this data to areas on Kalimantan and Sumatera underlain by known coal deposits, it was estimated that more than 250,000 coal seam fires may have been burning in Indonesia in 1998.

United States

Many coalfields in the USA are subject to spontaneous ignition. The Federal Office of Surface Mining (OSM) maintains a database (AMLIS), which in 1999 listed 150 fire zones. In Pennsylvania, 45 fire zones are known, the most famous being the fire in the Centralia mine in the hard coal region of Columbia County. In Colorado coal fires have arisen as a consequence of fluctuations in the groundwater level, which can increase the temperature of the coal up to 30 °C, enough to cause it to spontaneously ignite. The Powder River Basin in Wyoming and Montana contains some 800 billion tons of brown coal, and already the Lewis and Clark Expedition (1804 to 1806) reported fires there.

India

History of coal mines fires can be traced back to the year 1865, when the first fire was reported in Raniganj Coalfields. Over 140 years fires have been reported till the year 1967 from both Jharia and Raniganj coal fields and superior quality non-cocking coal in Raniganj coal fields. Fires occur whenever and wherever combustible material is present in mine working. They endanger not only the valuable lives of men in mine but also cause considerable economic losses to the organization affected by them. These fires not only continue to spread to adjoin areas, adding to the losses but also prevent economic exploitation of the seam in the vicinity. Again the open fire in these fields causes environmental pollution by emission of huge quantities of steam, smoke and noxious gases posing a serious health hazards. In Indian coal mines 75% of the coal fires occur due to spontaneous combustion. The main aspect of starting the fire in India is that the coal seams are thicker and there is a

tendency of spontaneous heating during the depillaring operation. The problem of extraction of thick seam and coal standing in pillars is a serious one particularly in cases where they are with high moisture, high volatile and low ash content which are more liable to spontaneous combustion. It is not practicable to extract all the coal by caving method or even by complete packing under Indian mining condition. Pillars standing for long time are liable to deteriorate in straight and spilling may occur.



Fig 2.1 Coal reserves of India [18]

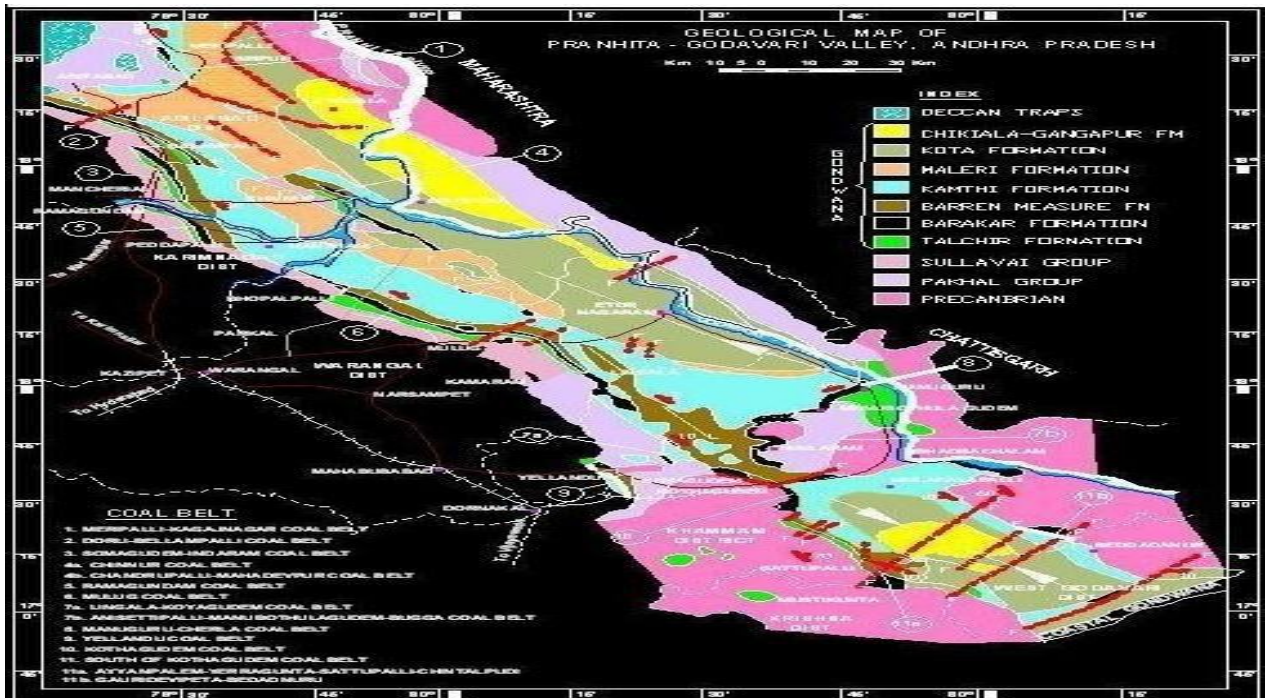


Fig 2.2 Area map of coal fields of SCCL



Fig 2.3 Area Map of coal fields of SECL

2.3 Spontaneous heating [2]

The phenomenon in which the coal catches fire automatically on coming in contact with oxygen in the atmosphere without any external source of fire which leads to mine fires is known as spontaneous heating of coal. It is primarily thought that the main cause of spontaneous heating is the self oxidation of coal. Although the actual mechanism of coal oxidation is yet unknown, there are many theories put forward for explanation of the coal oxidation and combustion.

2.4 Mechanism of spontaneous heating [2]

The oxidation of coal, like all oxidation reactions, is exothermic in character. The exact mechanism of the reaction is still not well understood. However, scientists agree that the nature of the interaction between coal and oxygen at very low temperatures is fully physical (adsorption) and changes into a chemisorption form starting from an ambient temperature. When coal is exposed to air it absorbs oxygen at the exposed surface. Some fraction of the exposed coal substance absorbs oxygen at a faster rate than others and the oxidation results in the formation of gases. Mainly CO, CO₂, water vapor along with the evolution of heat during the chemical reaction. The rate of oxygen consumption is extremely high during the first few days (particularly the first few hours) following the exposure of a fresh coal surface to the atmosphere. It then decreases very slowly without causing problems unless generated heat is allowed to accumulate in the environment. Under certain conditions, the accumulation of heat cannot be prevented, and with sufficient oxygen (air) supply, the process may reach higher stages. The loose coal-oxygen-water complex formed during the initial stage (peroxy-complexes) decomposes above 70-85° C, yielding CO, CO₂ and H₂O molecules. The rate of chemical reactions and exothermicity change with the rise in temperature, and radical changes take place, starting at about 100° C, mainly due to loss of moisture. This process continues with the rise in temperature, yielding more stable coal-oxygen complexes until the critical temperature is reached. The ignition temperature of bituminous coal is nearly 160-170° C and of anthracite coal nearly 185° C. Once the coal reaches its ignition point, the air supply to it will only increase the combustion.

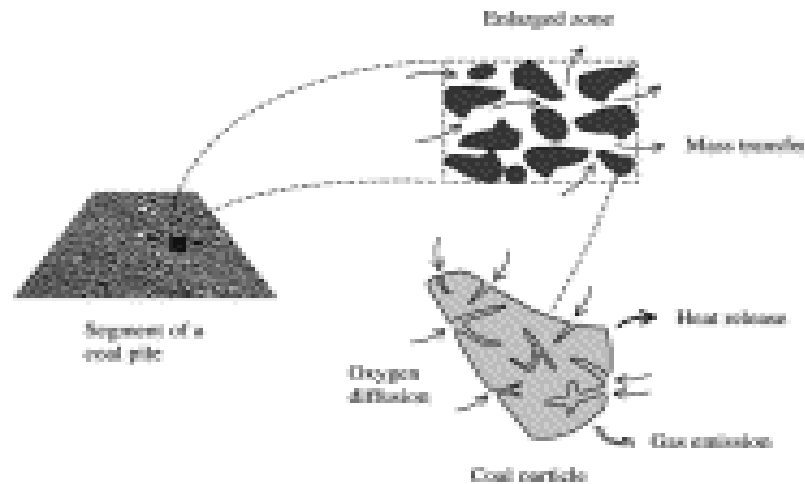


Fig 2.4 Mechanism of spontaneous heating [2]

2.5 Theories of spontaneous heating [2]

2.5.1 Coal oxidation theory– various stages of coal oxidation is given in the flow chart below

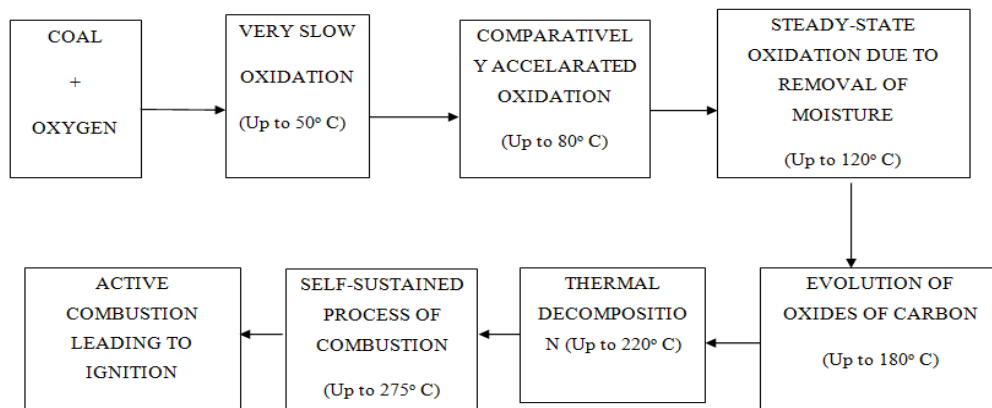


Fig 2.5: Sequential stages in the spontaneous combustion of coal [2]

The overall oxidation process of coal depends on the following factors:

- Temperature – usually rate of chemical reaction increases with temperature rise and is almost doubled for 10° C rise in temperature. The minimum temperature for coal-oxygen reaction is -80°C during which it is physical adsorption and at room temperatures it is chemisorption.
- Type of coal – the intrinsic oxidation mechanism is same for every type of coal but it is the availability of active centers in coal that defines the proneness of a particular coal to spontaneous heating. Usually high moisture and low rank coal

have higher oxygen avidity with better ease of peroxy-complex formation and hence higher tendency towards spontaneous heating.

- Extent of oxidation – as the time of exposure of coal surface to air increases the oxidation reaction gradually decreases and the coal gets weathered. In the initial stage the macro pores on surface determine oxygen consumption whereas in later stages the micro pores determine.
- Moisture – moisture adds to heat required for spontaneous combustion by heat of wetting released. It helps in the formation of peroxy complex and influences the rate of reaction. Release of moisture from coal produces more active centers making it more potent to oxidation.

2.5.2 Pyrite theory –

The pyrites present as impurities in coal acts as a major source of heat. The oxidation of pyrites is given by the following reaction:



The above reaction is exothermic and it produces heat which is capable enough of triggering spontaneous combustion. Moreover the products of the above reaction have greater amount of volume and hence break open the surface they are embedded in. But it has been found that pyrites can be the cause of spontaneous heating only when they are present in considerable proportions.

2.5.3 Bacterial theory –

Spontaneous heating observed in haystacks and in wood are known to be mainly due to bacterial action. Different evidences showed that bacteria were capable of living on coal and in some cases such bacteria caused a slight rise in temperature of the coal. Graham observed that sterilized coal oxidized at the same rate as the unsterilized coal and concluded that mechanism of oxidation did not include bacterial activity. Fuchs however concluded that bacteria could cause only a slight heating which may not play any significant role.

2.5.4 Phenol theory –

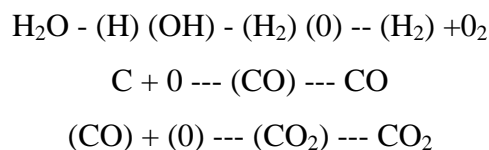
Experiments have shown that phenolic hydroxyls and poly phenols oxidize faster than many other groups. This theory is interesting because it offers a method of determining liability of coal to spontaneous heating.

2.5.5 Electro chemical theory –

It explains auto-oxidation of coals as oxidation-reduction processes in micro galvanic cells formed by the coal components.

2.5.6 Humidity theory –

Quantity of heat liberated by atmospheric oxidation of coal is much less than the quantity of heat required removing water from the coal. Thus it can be concluded that if the evaporation of water can be induced at the seat of heating, then the temperature of heating would decrease. Mukherjee and Lahiri (1957) proposed the following mechanism of the reaction between water and coal at 100°C. (Brackets indicate chemisorptions):



When it is recalled that water is an oxidation product of low temperature oxidation of coal, the above scheme well explains other possible sources of CO and CO₂ in low temperature reaction between coal and oxygen.

2.6 Factors affecting spontaneous heating [4]

The main reason for the difficulties in understanding the mechanism of spontaneous combustion is the presence of many internal and external factors affecting the initiation and development of the phenomenon. These factors have been reviewed by various researchers.

The main factors which have significant effects on the process are summarized below:

2.6.1 Intrinsic factors – These factors are mainly related to nature of coal

- Pyrites – As pyrite content increases the tendency of spontaneous heating increases.
- Inherent moisture – Changes in moisture content such as drying or wetting of coal have significant effects.
- Particle size and surface area – As particle size decreases the exposed surface area increases and the susceptibility increases.
- Rank and Petrographic constituents – Lower rank coals are more susceptible.
- Chemical constituents – Ash generally decreases liability for spontaneous heating but certain parts of ash such as lime, soda; iron compounds have accelerating effect whereas alumina and silica have retarding effects.
- Mineral matter – Some chemicals promote and others inhibit spontaneous heating.

2.6.2 Extrinsic factors – These factors are mainly related to atmospheric, mining and geological conditions

- Temperature – Higher surrounding temperature leads to increase in oxidation process and ultimately in spontaneous heating of coal.
- Extraneous moisture – Evaporation of surface moisture leads to release of heat of wetting which adds to the temperature rise and increases susceptibility. Presence of atmospheric moisture increases rate of oxidation of coal.
- Oxygen concentration – Higher the oxygen concentration in the atmosphere more rapid is the oxidation process as oxygen is readily available.
- Coal seam and surrounding strata – Presence of faults that lead to the passage of air and oxygen to the heating are generally increase the rate of heating.
- Method of working, ventilation and air flow rate – Mining methods such as bord and pillar mining that leave some pillars attracts more spontaneous heating than longwall methods. Air flow rate controls heating to a large extent. If the ideal flow rate is maintained then it helps in dissipation of heat but if too much of air is flowing then it stagnates the heat and increases the heating.
- Timbering, roadways, bacteria and barometric pressure – Presence of timbers in the mines leads to the danger of catching of fire which gives the heat required for spontaneous heating of coal. Bacterial decomposition of coal and other wood products also releases some amount of heat which increases susceptibility.

2.7 National and International status

Proximate analysis of coals by TG has been investigated by **Ottaway (1982)**, **Cumming & McLaughlin (1982)**, **Rosenvold et al. (1982)**, and **Elder (1983)**. Coals tested were from the United Kingdom and the United States. Reasonably good agreement was reported with the standard proximate analysis determinations of all the coals. However, a closer examination of the data from these studies reveals the agreement is not within precision limits set by the standards [1].

Larkin (1988) described an attempt to develop a standard method for compositional analysis using TG. This perhaps has contributed to the lack of precision in the results obtained to date, which is seen by some as a deficiency of the technique. Another factor that has often been raised is the question of representativeness of the sample used. However, as Charsley & Warrington (1988) point out, if a 1 g sample is seen as representative of 1000 t shipments, then 10 mg may be reasonably extracted from the 1 gm.

Ottaway (1982) read all values directly from the TG chart. However, **Cumming & McLaughlin (1982)** recognised the need for a buoyancy correction to allow for the small differences in balance reading at the various temperatures used in the test. They applied a fixed correction to both the volatile and ash readings, and assumed the correction at the moisture temperature to be negligible.

Cumming and McLaughlin (1982) corrections to early testing did not prove very successful. Consequently, the buoyancy correction was closely examined, and it was found that the ash value from the TG profile at the end of the test varied significantly from the value obtained by reweighing the residue and crucible. The difference between the two values does not appear to be systematic, and is certainly not a constant value as suggested by them. This is not surprising as the parameters affecting buoyancy are numerous, including the density of coal, which is known to vary according to coal rank and type, and the density of the mineral matter present in the coal. As both the volatile matter and fixed carbon content determinations were made at the same temperature, it is reasonable to assume that the volatile matter/fixed carbon ratio can be read directly from the TG data file. Therefore, the true weight losses are apportioned as follows:

- moisture content (M in mg) = weight loss recorded by TG
- ash content (A in mg) = residual weight measured by reweighing at the end of the test

- volatile matter (VM in mg) = VM of TG * (sample weight -MA)/(VMTG + FCTG)
- fixed carbon (FC in mg) = FC of TG * (sample weight -M -A)/(VMTG + FCTG)

Elphick (1960) reported on the “effects of sample weight on the volatile matter” yield of New Zealand coals and showed that by decreasing the sample weight from the standard 1 g down to 0.1 g the measured volatile matter yield increased. Therefore, to make the results comparable to the standard, correction factors were necessary. This difference in volatile matter yield with sample weight was not addressed by any of the previous studies on proximate analysis of coals by TG.

Elphick (1960) The ash content values did not vary with sample weight, consistent with the findings of Elphick (1960). The moisture content variations can be attributed to sampling, as it was noted that samples taken from the top layer of the stored coal produced lower moisture values (presumably as a result of surface drying). A plot of volatile matter against sample weight yields a similar trend to that observed by Elphick (1960). As the weight of the sample decreases the measured volatile matter increases.

Mohalik et al. (2009) presented the review of application of three thermal techniques viz; differential thermal analysis (DTA), thermogravimetry (TG) and differential scanning calorimetry (DSC); for studying the susceptibility of coal to spontaneous heating and fire. It also critically analyses the experimental standards adopted by different researchers, while applying these techniques in studying thermal behavior of coal samples. The paper also presents the future direction of research in this subject area.

Nimaje et al. (2010) made thermal studies on spontaneous heating of coal. Of all the experimental techniques developed thermal studies play an important and dominant role in assessing the spontaneous heating susceptibility of coal. They made an overview of thermal studies carried out by different researchers across the globe for determination of spontaneous heating of coal and revealed that lot of emphasis on experimental techniques is necessary for evolving appropriate strategies and effective plans in advance to prevent occurrence and spread of fire.

Gouws et al. (1988) gave three characteristics on a differential thermal analysis thermogram (i.e., the crossing-point temperature, stage II exothermicity gradient and the transition temperature to high-level exothermicity) are generally believed to be indicative of the self-

heating propensity of coal. A new index was developed and applied to 58 coals, enabling known dangerous and safe coals to be identified.

Jose, et al. (1995) used differential thermal analysis (DTA) as a method to study the self-heating behaviour of fresh and oxidized coals. Oxidation was performed in air at 200°C for periods of up to 72 h. As the rank of the coal increases, both the self-heating and the end of combustion temperatures also increase. The total heat loss (area under the DTA curve) increases with the rank of the coal. An increase in the self-heating temperature, a decrease in the temperature of the end of combustion and a decrease in total heat flow were observed as a consequence of coal oxidation. A relationship between the total heat loss and the calorific value as determined using the ASTM standard method is pointed out.

Vancea, et al. (1995) work investigates the effect of the moisture content of coal on its spontaneous ignition in oxygen (40°C–140°C). It has been found that the highest heating rate is achieved at a medium moisture content of 7 wt% for an initial inherent moisture content of the coal before drying (in dry nitrogen at 65°C) of 20 wt%. This is particularly noticeable at temperatures below 80°C and tends to support previous studies showing that a maximum oxidation rate occurs at such moisture content in the same temperature range.

Table 2.1 Experimental parameters used by different researchers in DTA studies on spontaneous heating of coal [18]

Sl. No.	Name of author	Year	Parameters							
			Particle size/mesh	Heating Rate °C/min	Atm.	Sample Amt.	Flow rate ml/min	No. of samples studied	Ref. material	Temp range/ °C
01	Whitehead and Breger	1950	-	10,20	Air/ Vacuum	-	-	-	-	Amb. to 550
02	Glass	1955	-100	10,20	-	-	-	7	-	Amb. to 1000
03	Berkowitz	1957	-65	6	N2	100	2.5	6	Dry quartz	Amb. to 500
04	Banerjee and Chakraborty	1967	-72, -200, -10 +60	1,3,5,10,15	Atm. Air	600	-	6	Calcined alumina	Amb. to 400
05	Banerjee et al.	1972	-72	5	Atm. Air	600	-	6	Calcined alumina	Amb. to 400
06	Haykiri-Acma et al.	1993	-	15	N2	-	-	-	-	Amb. to 1000
07	Podder et al.	1995	-100	10	Ar	10	100	5	-	30 - 900
08	Iordanidis et al.	2001	-16	10	N2	-	150	7	Alumina	Amb. to 1000
09	Kok	2002	-60	10	Air	10	167	4	Alumina	20 - 900
10	Panigrahi, Sahu	2004	-72	30	Atm. Air	600	-	31	Alpha Alumina	Amb. to 400
11	Elbeyli and Piskin	2006	-65	10	Air/ N2	10	100	1	-	Amb. to 1000
12	Haykiri-Acma et al.	2006	-65	20	Atm.	20	-	7	Alumina	Amb. to 1000
13	Sis	2007	-8+10 to -400	10	Air	10	50	1	Alpha Alumina	Amb. to 900
14	Ozbas	2008	-	10	N2	10	50	15	Alpha Alumina	Amb. to 900

CHAPTER – 3

EXPERIMENTAL TECHNIQUES

To study the effects of various parameters of coal that affect the spontaneous heating tendency of coal, the following experiments are needed to be carried out:

- Proximate analysis
- Differential thermal analysis

3.1 SAMPLE COLLECTION AND PREPARATION

It is the process by which the physical and chemical properties of the mineral or ore can be ascertained with the desired accuracy. It is the process of collecting the small portion of a whole such that consistence of that portion represents that of a whole.

Different types of sampling are:

- Channel sampling
- Chip sampling
- Grab sampling
- Bulk sampling
- Drill hole sampling

Chip sampling is done in hard ores where it is difficult to cut the channels. It can be taken in case of uniform ores and where the rock structures are independent of the values. The sample is collected by breaking of small equal sized chips from a face at points usually equally spaced both vertically and horizontally.

Grab sampling is applied to the broken ore in the stope or at the face, ore transported. Usually grab sampling of the ore broken in the stope is unreliable as accurate estimation of the volume of broken ore is impossible. Grab sampling of tubs or ships is however more representations since samples are collected from units of regular volume.

Bulk sampling is done where conventional sampling methods do not give a representative scale; large scale sampling or bulk sampling resorted to. Bulk samples eliminate the effect of irregular distribution of value or minor.

For our Project work, channel sampling method has been carried out which is common among various techniques discussed above.

3.1.1 Channel sampling [14]

The section of seam to be sampled shall be exposed from the roof to the floor. The seam sample shall, be taken in a channel representing the entire cross-section of the seam having the dimensions of 30 x 10 cm, that is, 30 cm in width and 10 cm in depth. For this purpose, two parallel lines, 30 cm apart end at right angles to the bedding planes of the seam shall be marked by a chalked string on the smooth, freshly exposed surface of the seam. Obvious dirt bands exceeding 10 cm in thickness shall be excluded. The channel between the marked chalk lines in the seam shall be cut to a depth of 10 cm and the coal sample collected on a clean strong cloth or tarpaulin placed immediately at the bottom so that the chances of pieces flying off during excavation of coal are minimized.

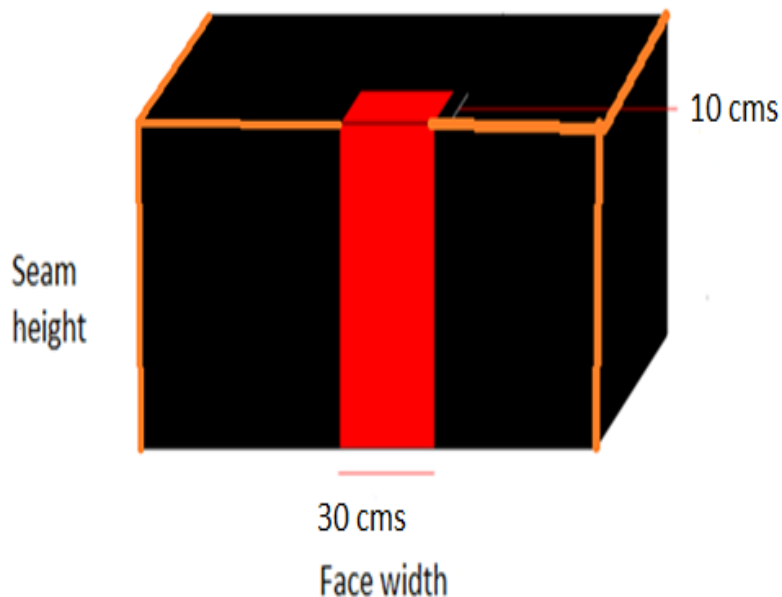


Fig 3.1 Channel Sampling

3.1.2 Sample preparation [14]

The samples received from the field via channel sampling are crushed in the laboratory as per the experimental requirements. The crushed sample is then sieved to required sizes and stored in air tight polythene packets. The packets are stored in air tight containers for further use in experimentation.

Table.3.1 List of coal samples

Sl. No	Sample	Coalfields
1.	SECL-1	SOUTH EASTERN COALFIELD LIMITED
2.	SECL-2	
3.	SECL-3	
4.	SECL-4	
5.	SECL-5	
6.	SCCL-1	SINGARENI COLLIERIES COMPANY LIMITED
7.	SCCL-2	
8.	SCCL-3	

3.2 METHODS FOR DETERMINING INTRINSIC PROPERTIES OF COAL

Proximate analysis [13]

Proximate analysis of coal was developed as a simple method for characterizing and determining the distribution of various constituents found in coal. Here the coal sample is subjected to heat under specified conditions, and the generated products can be grouped into: i) moisture; ii) volatile matter iii) fixed carbon, iv) ash, the inorganic residue remaining after combustion. The method determined by IS (Indian Standard) 1350 (Part- I)-1984 was followed for the proximate analysis, i.e. for the determination of volatile matter, moisture, ash and fixed carbon

Determination of Moisture Content (M)

As all coals are mined wet they are always associated with some amount of moisture. The moisture can be physically or chemically bound, due to its nature, origin and occurrence.

Extraneous and inherent moisture must be properly differentiated. Extraneous moisture evaporates just by exposing coal to sunlight, even then coal still have moisture inside it or it should be removed while keeping the sample in a furnace for 2 hours at 35 to 40°C.. This internal moisture can be removed by heating coal above 100°C. The mode of occurrence and handling of coal is responsible for the quantity of external but the air-dried moisture is associated due to the inherent hygroscopic nature of the coal.

Procedure

- About 1g of air-dried coal sample finely powdered (-212 μ) and weighed in a silica crucible.
- It is then placed inside an electric hot air oven (Plate 4.1) which was maintained at 108°C and allowed to remain there 1.5 hours
- It was then taken out with a pair of tongs, and cooled in desiccators for about 15 minutes and then weighed.
- The loss in weight was reported as moisture (on percentage basis).

Moisture is calculated as per the following formula.

$$\text{Moisture\%} = \frac{(Y-Z)}{(Y-X)} * 100$$

Where,

X= weight of empty crucible, g

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

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



Plate 3.1 Oven for Moisture Content Determination

Determination of Ash Content (A)

The residue left after the combustion of coal under some specified conditions is called the coal ash. It is formed as the result of chemical changes that take place in the mineral matter and does not occur as such in coal. Hence ash and mineral matter of coal are not identical. Two types of ash forming materials in coal are mainly the extraneous and inherent mineral matters.

The extraneous mineral matter comprises of materials like calcium, magnesium and ferrous carbonates, pyrite, marc site, clays, shale's, sand and gypsum. The extraneous mineral matter builds on its origin by two types that are given below:

- The substances which got coupled with the rotting vegetable material during its alteration to coal, which is difficult to remove by mechanical methods,
- Rocks and dirt that mixed up during mining and handling of coal.

The intrinsic mineral matter is the inorganic elements linked with organic components of coal. The origin of such materials is most probably from the plant materials from which the coal is produced. When the total quantity of ash is pertained ash from intrinsic mineral matter becomes insignificant as far as the total quantity of ash is pertained. Indian coals suffer from the major shortcoming, that the mineral matter content is not only high, but of intimately linked type, due to its drift origin. Ash is quantitatively and qualitatively different from the mineral matter originally present in coal because the numerous modifications that occur, such as loss of water 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. In fact, combustion circumstances determine the amount to which the weight change takes place and it is necessary that standardized operations should be closely followed to ensure productivity.

Procedure:

The empty crucible was cleaned by heating in a muffle furnace for one hour at 800⁰C so that other mineral matter if presents get burnt. It was taken out, cooled to room temperature and the weight is taken. Approximately 1gm of coal sample was weighed in the crucible and placed in a muffle furnace at 450⁰C for 30 minutes and the temperature of the furnace was raised to 850⁰C for 1hour. The crucible was taken out and placed in desiccators and weighed.

Ash is calculated as per the following formula:

$$\text{Ash\%} = (Z-X/Y-X)*100$$

Where,

X = weight of empty crucible in grams

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

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



Plate 3.2 Muffle Furnace for Determination of Volatile Matter and Ash Content

Determination of Volatile Matter (VM)

When a coal sample is heated in specified equipment's under prescribed conditions in Indian standard, the loss of mass in coal, corrected for moisture, is referred to as volatile matter of coal. Some of the components of coal transformed to volatile matter are hydrogen, carbon monoxide, methane and other hydrocarbons, vapors, ammonia, some organic sulphur and oxygen containing deepens and some incombustible gases, such as CO and water vapor, all of which come from the disintegration of organic matter in coal. Inorganic materials in coal are responsible for the presence of the water of hydration of mineral matter, carbon dioxide from carbonates and hydrogen chloride from inorganic chlorides to the volatile matter.

Procedure

For the determination of volatile matter a special volatile matter silica crucible (38 mm height, 25 mm external diameter and 22 mm internal diameter) was used. The empty volatile

matter crucible was weighed. 1g of coal sample (-212 μ size) was weighed in the volatile matter crucible and it was placed inside a muffle furnace maintained at 925°C with the lid covering the crucible for 7 minutes. The heating was carried in the muffle furnace (Plate 3.2) out exactly for seven minutes, after which the crucible was removed, cooled in air, then in desiccators and weighed again. The calculation was done as per the following formula:

$$\% \text{ Volatile Matter} = (Y-Z/Y-X)*100 - \text{Moisture } \%$$

Where,

X = weight of empty crucible, g

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

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

Determination of Fixed Carbon (FC)

After the determination of moisture, volatile matter and ash the mathematical remaining is fixed carbon by definition. Fixed carbon plus ash present the approximate yield of coke from coal. The value of fixed carbon is evaluated by subtracting the resultant summation of moisture (M), volatile matter (VM) and ash (A) from 100 with all parts on the same moisture reference basis

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

3.3 METHODS FOR DETERMINING SPONTANEOUS HEATING SUSCPETIBILITY OF COAL

3.3.1 Differential thermal analysis [20]



Plate 3.3 DTA – TG apparatus

It is often used to determine the physical property of a substance as a function of temperature. This method analyses the effect of temperature on the properties of the sample and compares it with an inert reference material. In DTA the temperature difference between sample and inert reference is measured when both are subjected to identical heat treatments and then plotted against time or temperature. The DTA apparatus consists of a sample and reference holder, a furnace, a temperature programmer to maintain constant temperature rate and an output to monitor the test. The sample holder has two thermocouples each for reference and sample. The sample is contained in a small crucible. The thermocouple should not be in direct contact with sample. By the temperature programmer the temperature is made to rise at a constant rate and the temperature difference of the sample and reference is plotted against time. The plot consists of three parts – stage I, II, III.

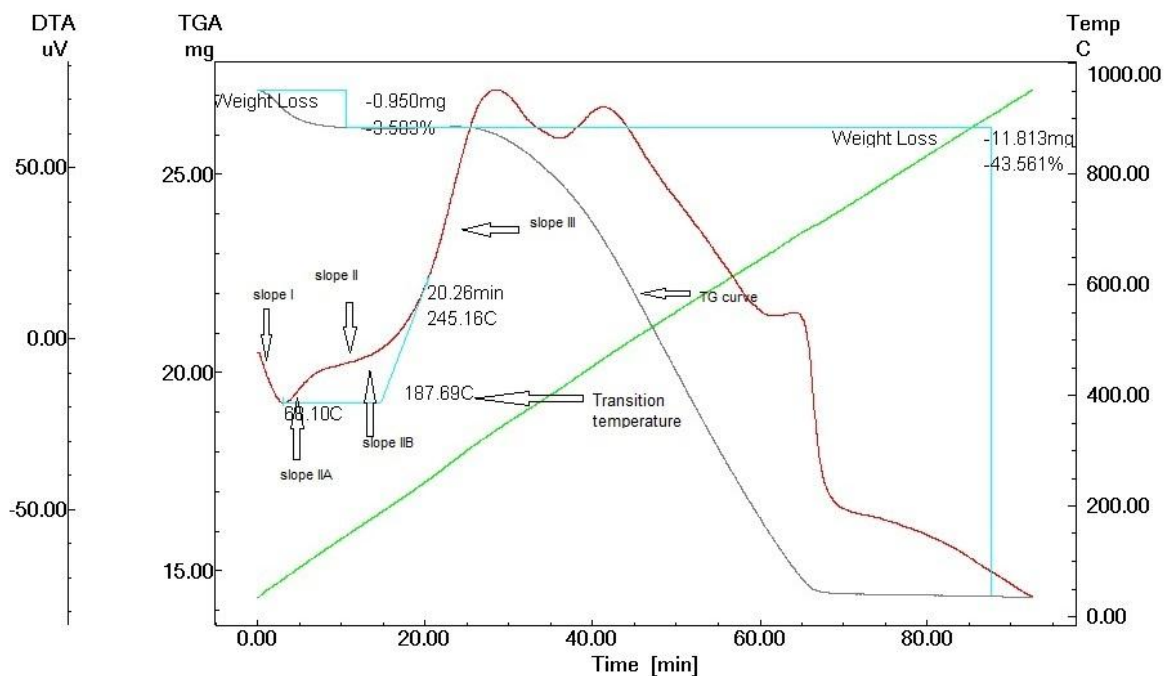


Fig 3.2 Different stages of DTA

During stage I mostly endothermic reactions take place and the temperature falls due to release of moisture.

Stage II consists of two parts – II A and II B. From the beginning of II A, the heating tendency starts accompanied by a small amount of endothermic reactions. The point where II A begins is called as the inflexion point as the thermogram suddenly rises. In II B exothermic reactions start to dominate.

The beginning of stage III is called as Transition temperature. This temperature is very important as it is directly related to spontaneous heating tendency of coal. The lower the transition temperature the higher is the susceptibility of coal and vice versa. From stage III onwards complete exothermic reactions occur and the temperature continues to rise steeply.

From the thermogram plot four important points are required – slope of II A, II B, overall slope of II and the transition temperature. Lower the slope values lower is the susceptibility. The transition temperature is found out by drawing tangents at the inflexion point and any point on stage III. Their intersection gives the transition temperature.

Procedure:

- About 20 mg of -212 micron (BSS) coal sample is taken in the crucible.
- The reference material taken is alpha alumina.
- The DTA machine is switched on and the software is set according to required conditions viz., heating rate is maintained at 10° C/minute and the final temperature is taken as 950°C.
- The plots obtained are then analyzed for the slopes of various stages and transition temperature.

CHAPTER- 4

RESULTS AND ANALYSIS

ABSTRACT OF EXPERIMENTAL TECHNIQUES

1. Proximate analysis

A. Determination of moisture

- Amount of coal : 1 g coal
- Size of coal : - 212 micron (-72 mesh)
- Heating time : 1.5 hours at 110° C

B. Determination of volatile matter

- Amount of coal : 1 g of coal
- Size of coal : - 212 micron (-72 mesh)
- Heating time : 7 minutes at 925° C

C. Determination of ash

- Amount of coal : 1 g of coal sample
- Size of coal : - 212 micron (-72 mesh)
- Heating time : 30 minutes at 450°C and 60 minutes at 850°C

2. DTA – TG

- Amount of coal : 20 mg of coal sample
- Size of coal : - 212 micron (-72 mesh)
- Heating rate : 10° C/min
- System : coal + air

4.1 RESULTS

The results of the parameters of proximate analysis (Moisture, Volatile matter, Ash and Fixed carbon) after removing the extraneous moisture are calculated using the standard procedure of all the collected coal samples covering some coalfields of India and are listed in Table 4.1.

Table.4.1 Results of the parameters of proximate analysis

Sample	Moisture % by standard procedure	Moisture % by TG	Ash% by standard procedure	Ash % by TG	Volatile Matter % by standard procedure	Volatile matter % by TG	Fixed Carbon % by standard procedure	Fixed carbon % by TG
SCCL-1	10.17	2.960	15.735	14.825	35.945	42.612	38.15	39.603
SCCL-2	10.02	3.389	9.77	10.57	32.12	42.523	48.09	43.518
SCCL-3	9.805	3.706	18.915	16.86	33.15	36.855	38.13	42.579
SECL-1	10.735	3.135	17.11	17.515	31.815	40.923	40.14	38.427
SECL-2	9.92	2.523	15.315	15.325	32.25	39.665	42.515	42.487
SECL-3	10.60	3.503	16.615	16.115	33.15	43.561	39.635	36.821
SECL-4	9.21	2.811	18.86	19.86	33.775	29.248	38.155	48.081
SECL-5	10.59	3.519	16.305	15.305	33.015	44.452	40.09	36.724

4.2 The Results of DTA-TG analysis:

The results of the parameters of proximate analysis (Moisture, Volatile matter, Ash and Fixed carbon) after removing the extraneous moisture are calculated using the DTA-TG of all the collected coal samples covering some coalfields of India and are listed in Table 4.2.

Table.4.2 The results of DTA-TG slopes of stage II

Sample	Slope of Stage IIA	Slope of Stage IIB	Slope of Stage II	Transition temp. (°C)
SCCL-1	0.173	0.161	0.170	175.55
SCCL-2	0.187	0.165	0.173	145.54
SCCL-3	0.185	0.157	0.173	160.72
SECL-1	0.181	0.150	0.170	163.87
SECL-2	0.177	0.144	0.169	195.67
SECL-3	0.178	0.149	0.170	187.69
SECL-4	0.180	0.145	0.171	174.21
SECL-5	0.175	0.164	0.171	150.49

The graphical presentations of all the DTA-TG curves are shown below:

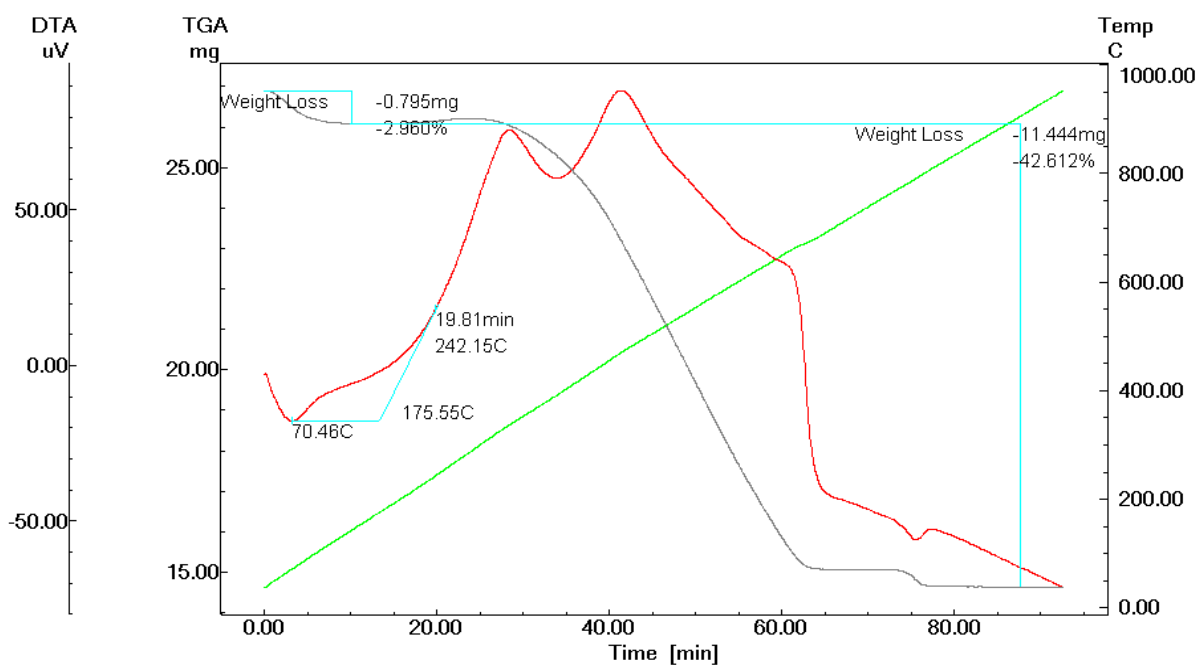


Fig. 4.1 DTA-TG Curve of SCCL-1 sample

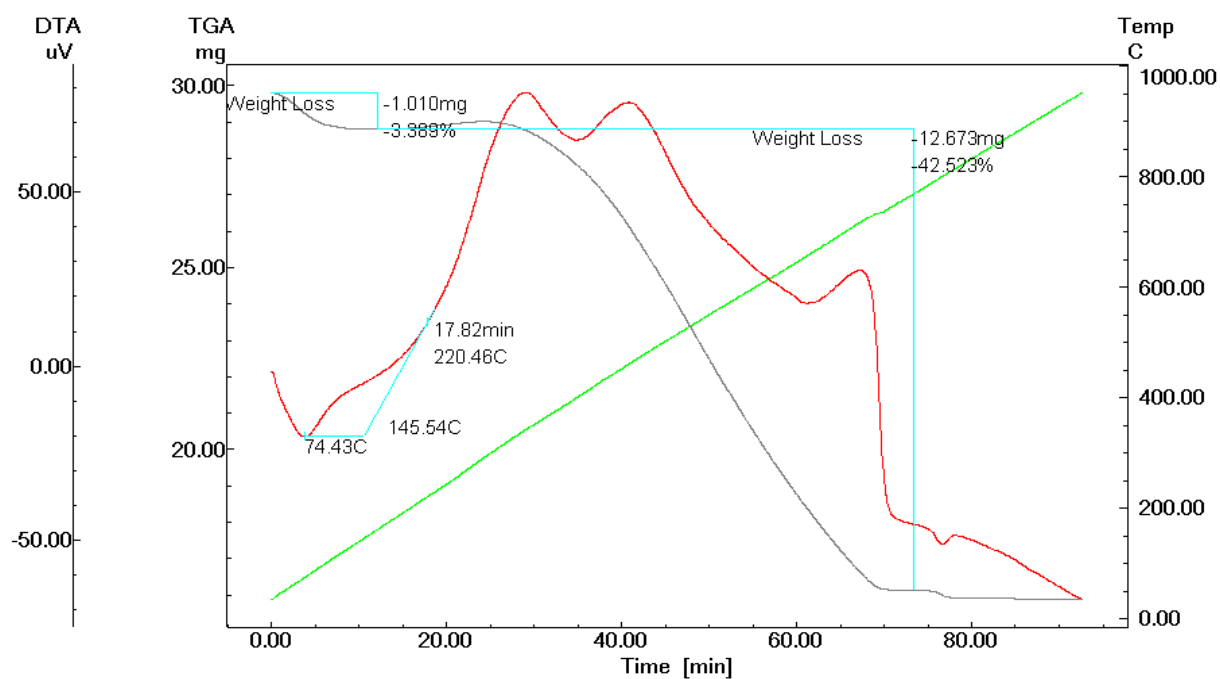


Fig. 4.2 DTA-TG Curve of SCCL-2 sample

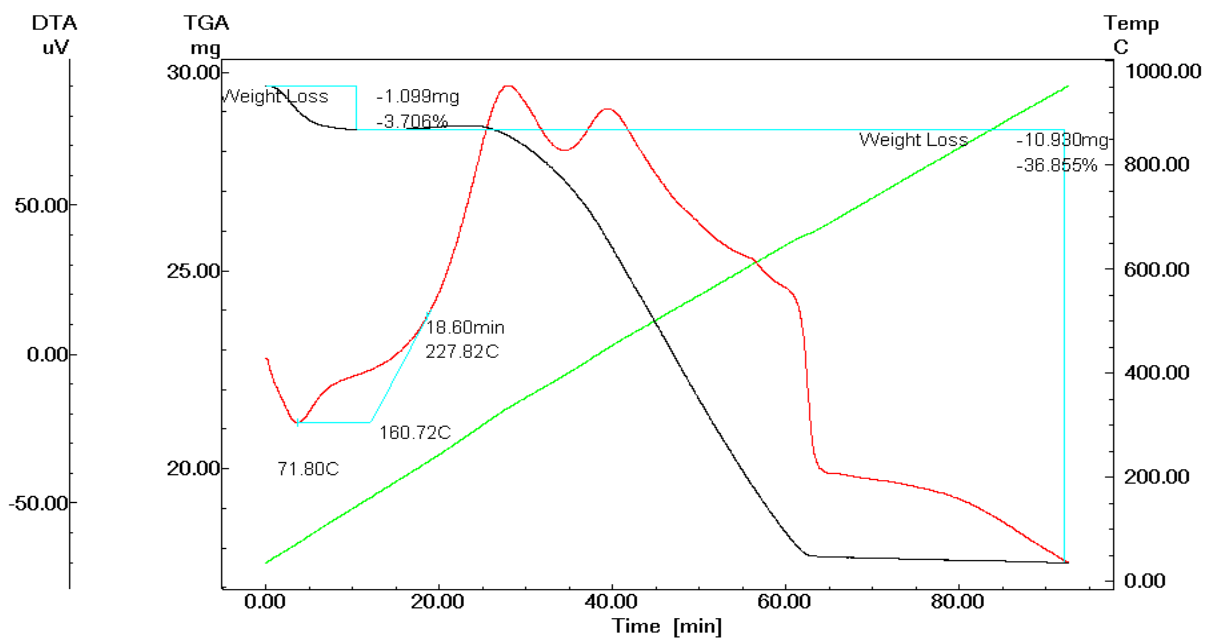


Fig. 4.3 DTA-TG Curve of SCCL-3 sample

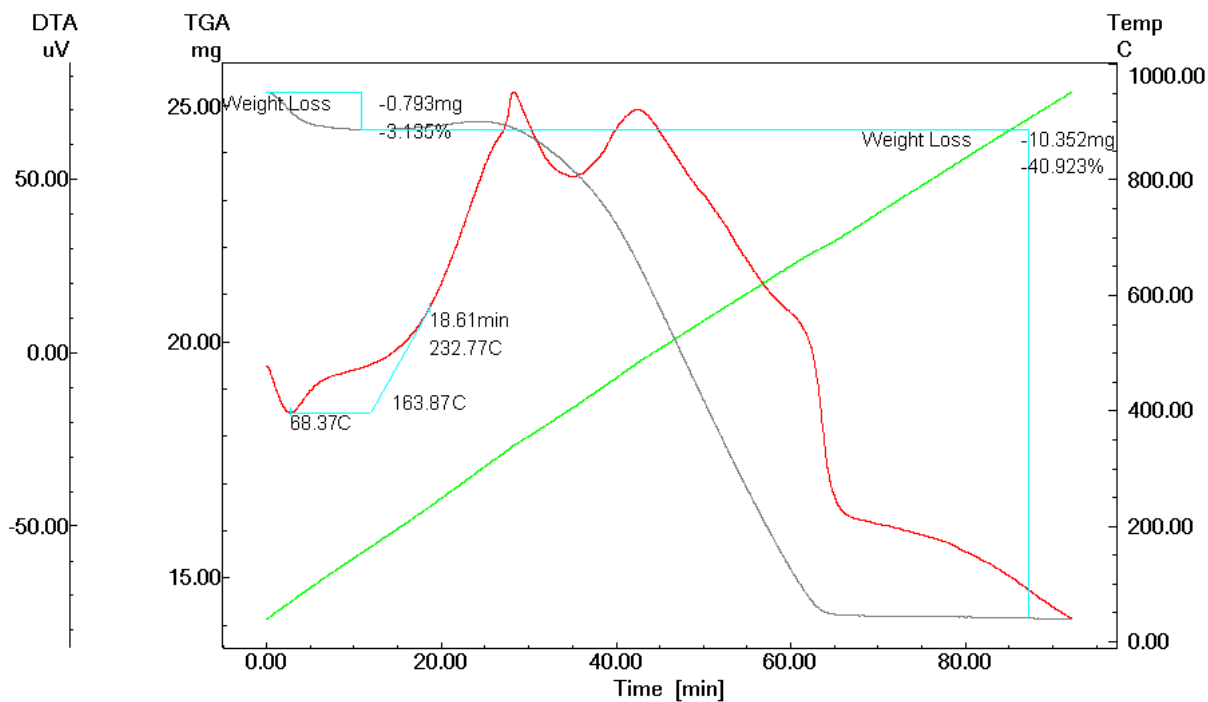


Fig. 4.4 DTA-TG Curve of SECL-1 sample

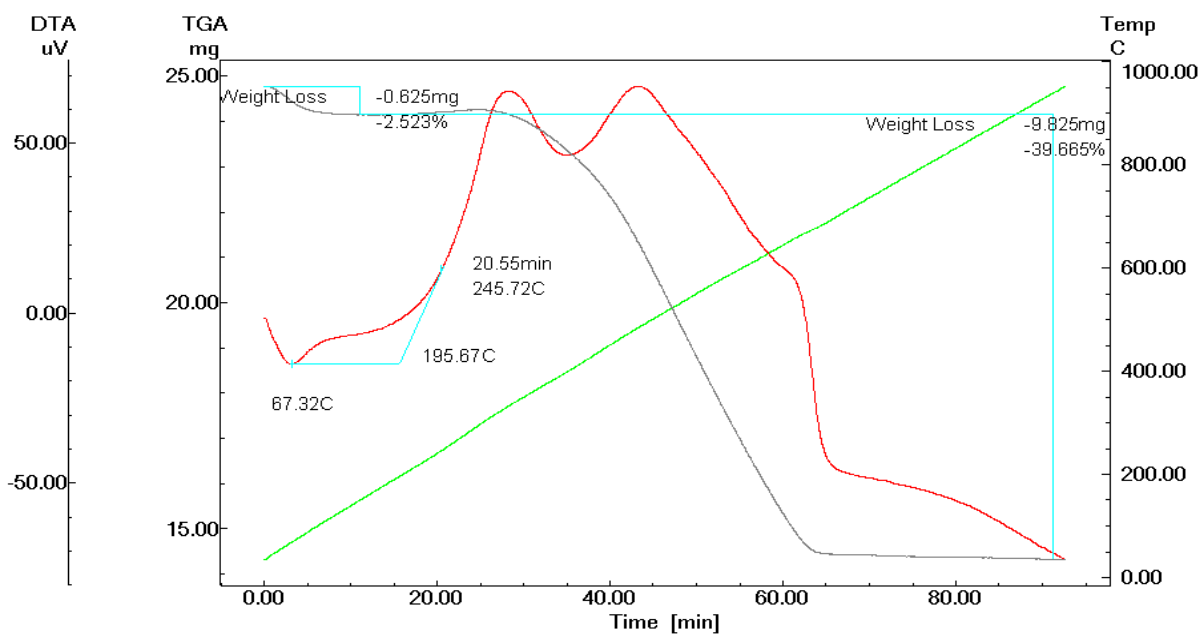


Fig. 4.5 DTA-TG Curve of SECL-2 sample

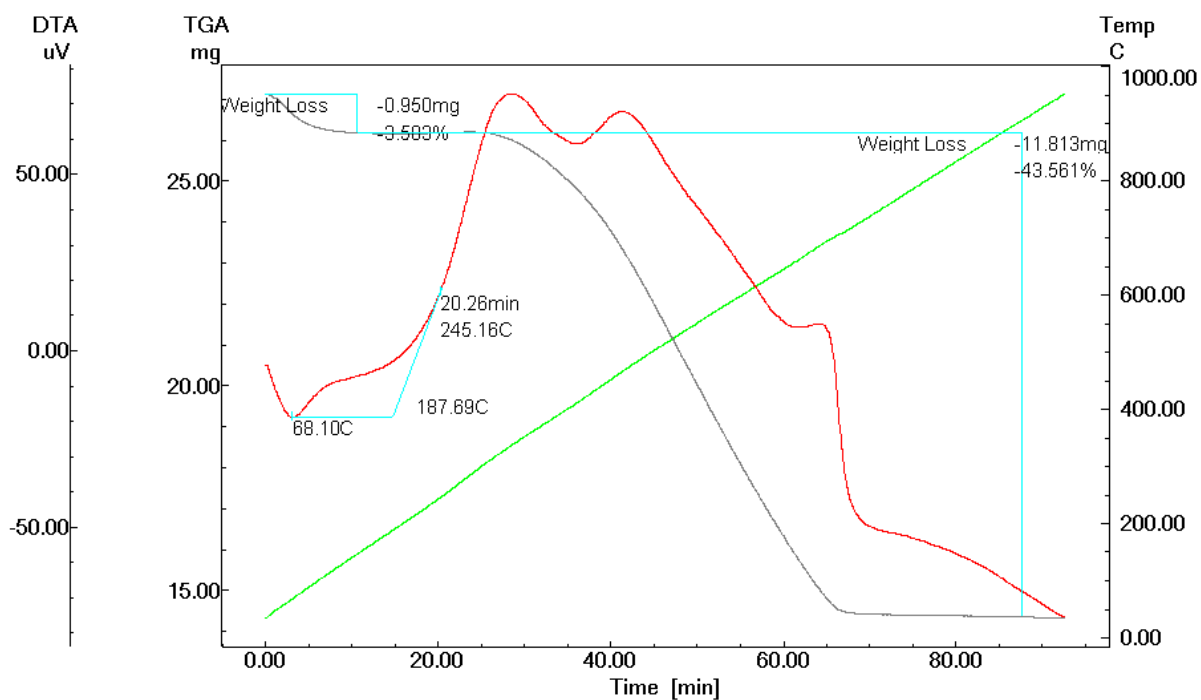


Fig. 4.6 DTA-TG Curve of SECL-3 sample

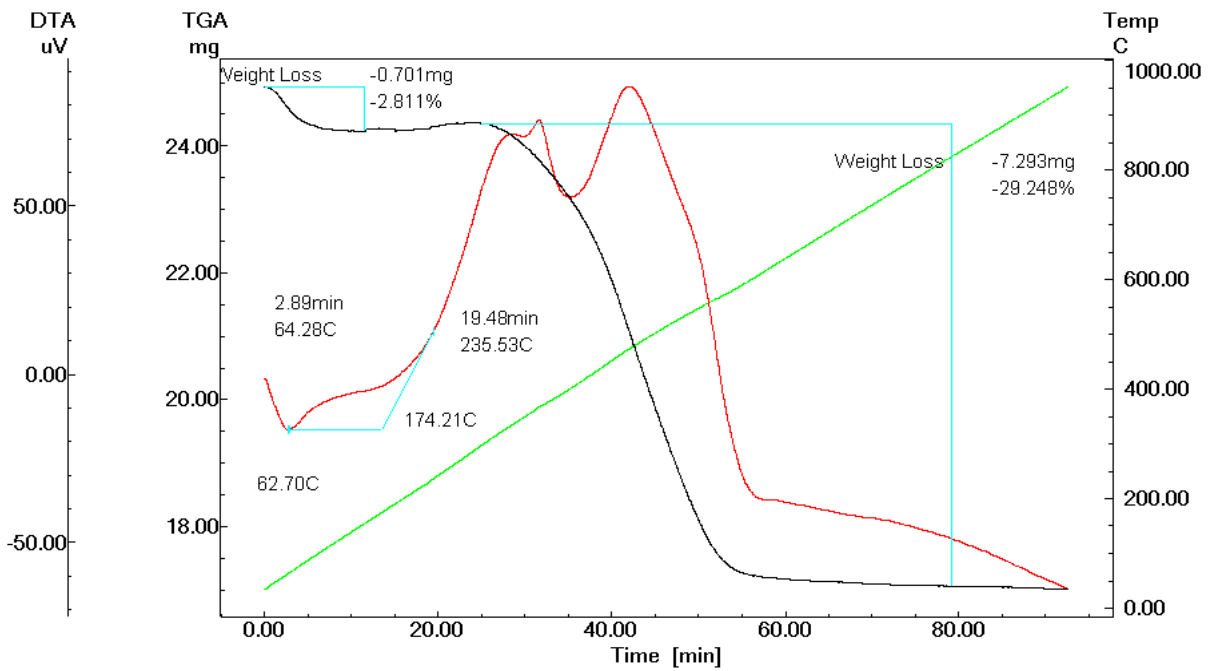


Fig. 4.7 DTA-TG Curve of SECL-4 sample

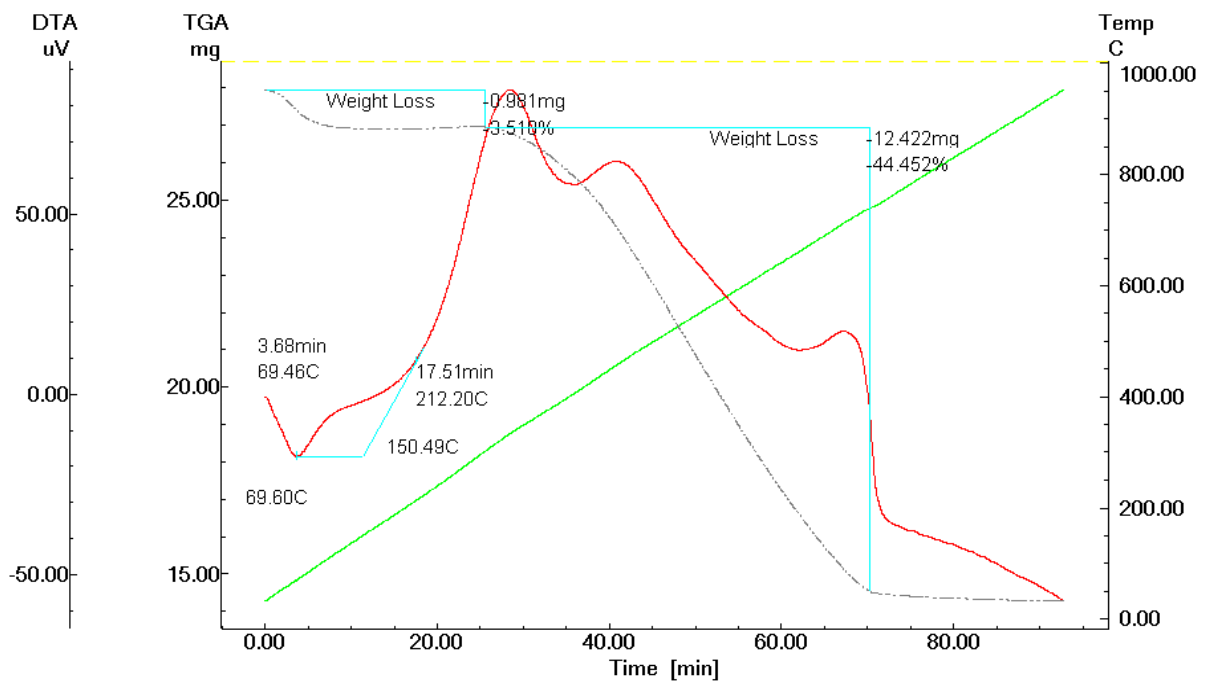


Fig. 4.8 DTA-TG Curve of SECL-5 sample

4.3 Correlation analysis

Correlation analysis was done using the MS Excel 2007 package. Microsoft Office Excel 2007 provides powerful tools and features that one can use to analyze, share, and manage data with ease. Commands and features that were often buried in complex menus and toolbars are now easier to find on task-oriented tabs that contain logical groups of commands and features.

The following tables shows the correlation between the parameters of proximate analysis and DTA

Table 4.3 Results of correlation analysis between DTA and parameters of proximate analysis (standard)

Proximate analysis parameters	DTA Analysis			
	Slope II	Slope IIA	Slope IIB	Transition Temp.
Moisture%	-0.30151	-0.27284	0.27610	-0.16287
Ash%	-0.22674	-0.27540	0.50192	0.12429
Volatile Matter%	-0.12923	-0.56726	0.20239	-0.04906
Fixed Carbon%	0.294111	0.49338	0.31256	-0.05932

Table 4.4 Results of correlation analysis between DTA and parameters of proximate analysis (DTA-TG)

Proximate analysis parameters	DTA Analysis			
	Slope II	Slope IIA	Slope IIB	Transition Temp.
Moisture%	0.66045	0.38750	0.58824	-0.60968
Ash%	-0.31158	0.21943	-0.67656	0.37792
Volatile Matter%	-0.15197	0.26441	0.54476	-0.16816
Fixed Carbon%	0.33824	0.44738	-0.29440	0.01998

4.4 FINDINGS

- From the table 4.3 it is found that the slope II and slope IIA are giving good result than the Transition temperature
- From the table 4.4 it is found that slope II and slope IIB are giving good result than transition temperature.

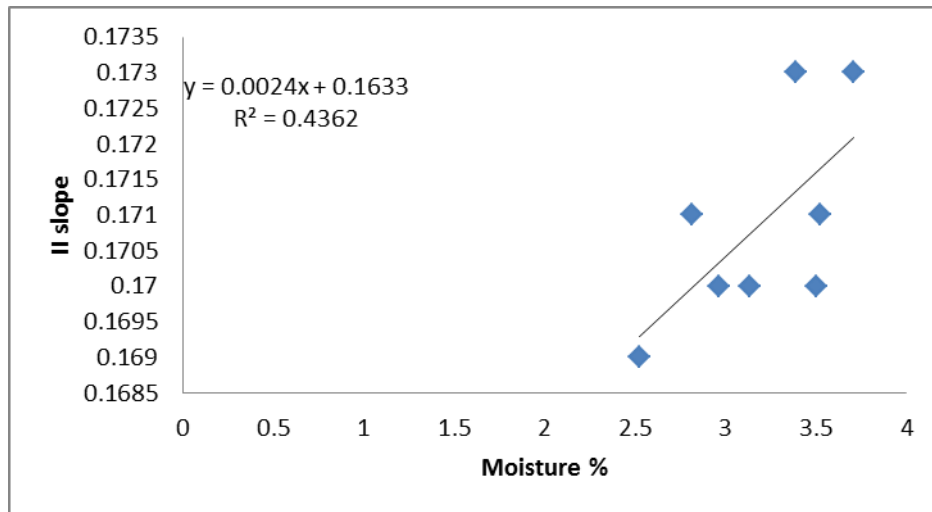


Fig. 4.9 Correlation between Moisture% and II slope of DTA-TG

From the correlation graph it is found that with increase in Moisture the slope of stage II of DTA-TG increases

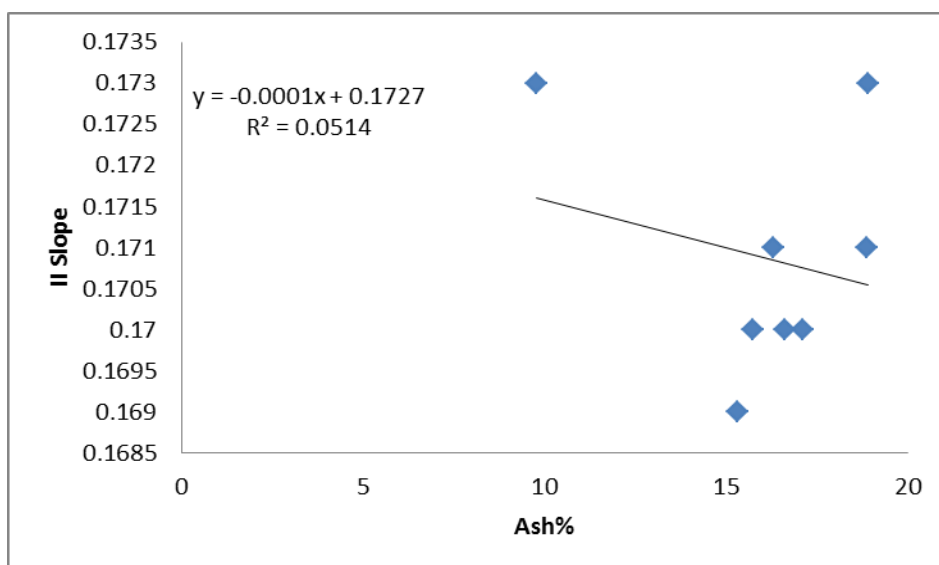


Fig. 4.10 Correlation between Ash% and II slope of DTA-TG

With the increase in Ash% from the correlation graph it is found that the slope of II stage of DTA-TG decreases

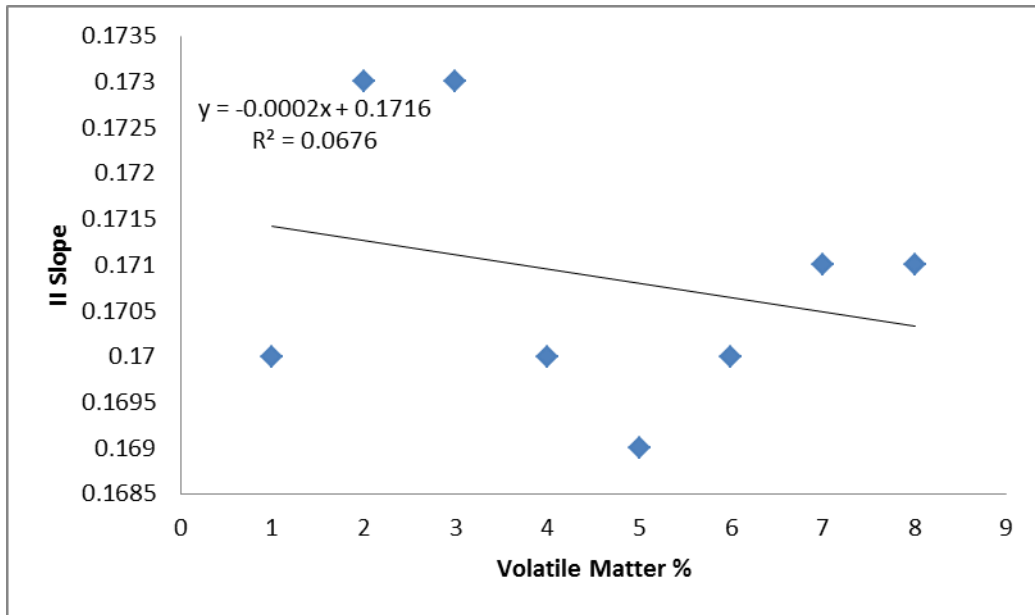


Fig. 4.11 Correlation between Volatile matter% and II slope of DTA-TG

It is observed that by the increase in volatile matter % the slope of II stage of DTA-TG decreases and vice-versa.

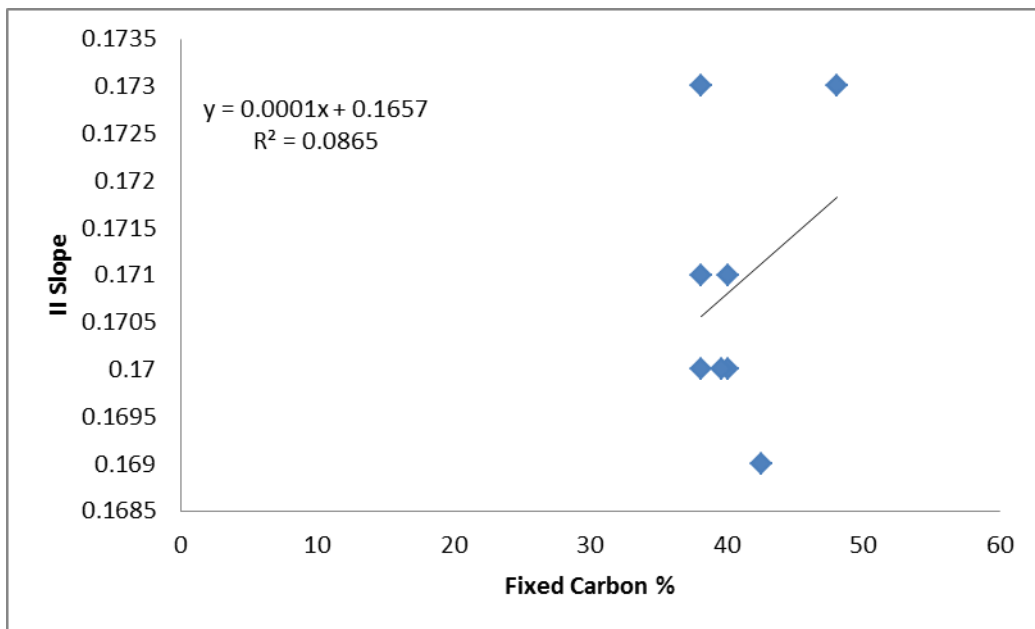


Fig. 4.12 Correlation between Fixed Carbon% and II slope of DTA-TG

From the correlation graph it is found that with the increase in Fixed carbon content the slope of stage II of DTA-TG curve also increases and vice-versa.

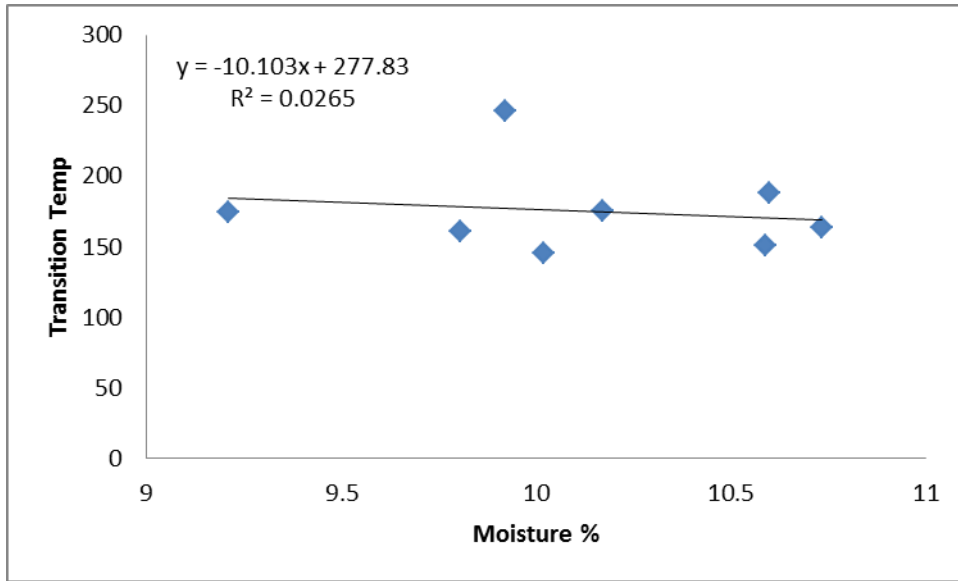


Fig. 4.13 Correlation between Moisture% and Transition temperature

From the correlation graph it is found that with the increase in moisture the Transition temperature decreases and vice-versa.

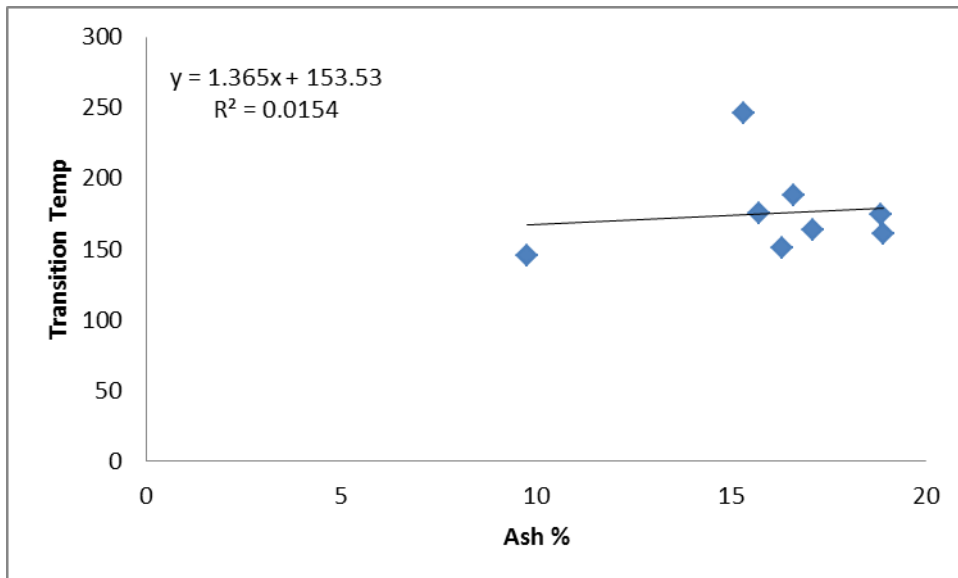


Fig. 4.14 Correlation between Ash% and Transition temperature

From the correlation graph it is found that with the increase in Ash the Transition temperature also increases and vice-versa.

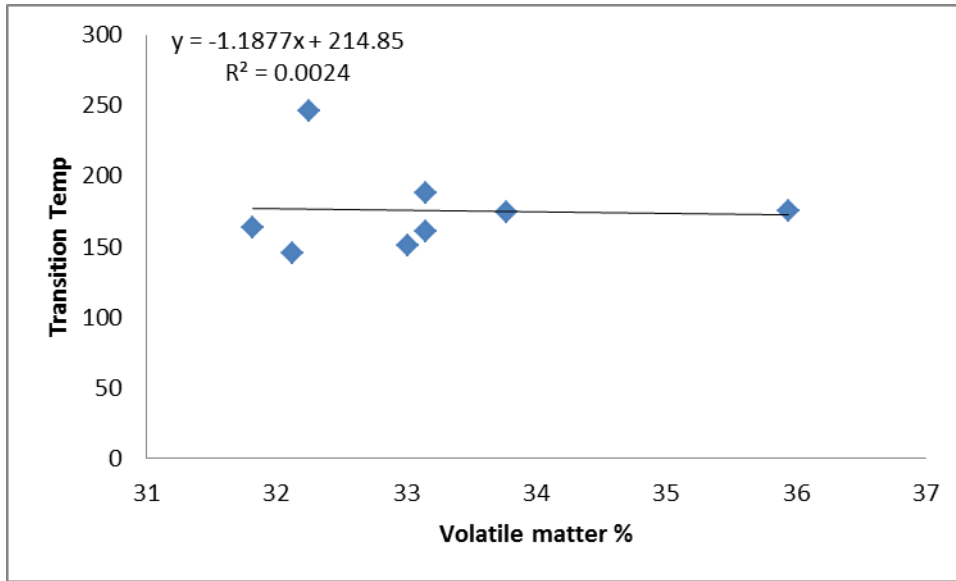


Fig. 4.15 Correlation between Volatile matter% and Transition temperature

From the correlation graph it is found that with the increase in volatile matter the Transition temperature decreases and vice-versa.

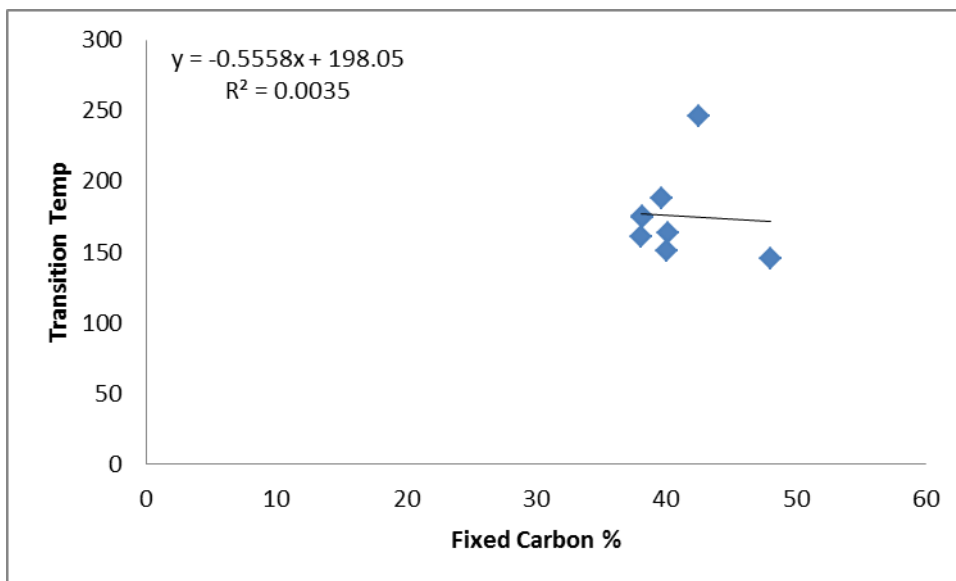


Fig. 4.16 Correlation between Fixed Carbon% and Transition temperature

From the correlation graph it is found that with the increase in fixed carbon content the Transition temperature decreases and vice-versa.

CHAPTER-5

CONCLUSIONS

In order to evaluate the role of intrinsic factors in the spontaneous heating of coal, 8 coal samples were collected from different mines of our country. Out of the 8 coal samples collected for analysis, 5 from SECL and 3 from SCCL using the standard sampling method. The intrinsic properties were measured by proximate analysis and the spontaneous heating susceptibility of the coal samples were determined by Differential Thermal Analysis (DTA-TG) in the laboratory and Correlation studies have been carried out using MS Excel to find out the correlation co-efficient and linear curve drawn between proximate analysis parameters and stage II slope of DTA curve and also find out the best R^2 (correlation factor).

- It was also observed that the Transition temperature obtained from the DTA – TG plot cannot be taken as a sole parameter to assess the spontaneous heating of coal, rather Stage II slopes
- It is also observed from thermo gravimetric analysis the volatile matter increases with decrease in the weight of sample at the rate

$$\text{Volatile matter} = 51.66 - 7.16(\log(\text{weight})) \text{ and } R^2 = 0.94$$

- From the correlation analysis between the intrinsic properties, it is found that the II slope of DTA-TG is varying with respect to Moisture, Volatile Matter and Ash content following the trend $y=0.0024x+0.1633$ with $R^2 = 0.4362$, $y= -0.0002x+0.1716$ with R^2 value 0.0676 and $y=-0.0001x+1727$ with R^2 value 0.0514 respectively.
- From the correlation analysis, it is also found that with increase in Moisture percentage, II slope of DTA-TG curve increases and decreases with increase in ash and volatile matter percentages.
- From the correlation analysis it is also found that with increase in moisture and volatile matter transition temperature decreases while it's reverse in case of ash.
- From the DTA analysis it is found that SCCL-3 sample have highest slope and least transition temperature while SECL-2 sample have least slope and highest transition temperature. So SCCL-3 is highly liable and SECL-2 is poorly liable to spontaneous heating.

- From the DTA-TG and proximate analysis it is found that at 2 to 4% of moisture, the spontaneous heating susceptibility increases while in general with increase in moisture the spontaneous heating susceptibility decreases.
- With the increase in ash percentage the spontaneous heating susceptibility decreases and vice-versa.
- While with increase in volatile matter in the coal the spontaneous heating susceptibility increase and vice versa.
- With increase in fixed carbon content in the coal the spontaneous heating susceptibility also increases and vice-versa.

REFERENCES

- 1 Beamish B. B. (1994), Proximate analysis of New Zealand and Australian coals by thermogravimetry, *New Zealand Journal of Geology and Geophysics*, vol. 37, pp. 387-392
- 2 Banerjee S.C. (1985), *Spontaneous Heating of coal and Mine Fires*, Oxford and IBH publishing Co Pvt. Ltd, 1st edition, pp.1-40
- 3 Charsley E. L., Warrington S. B. (1988), *Compositional analysis by thermogravimetry*, ASTM STP 997, Philadelphia, American Society for Testing and Materials, pp. 19-27.
- 4 Cumming, J. W., McLaughlin J. (1982), The thermogravimetric behavior of coal. *Thermochemical acts*, 57, pp. 253-272.
- 5 Didari V. and Kaymkci E. (2002), Relations between coal properties and spontaneous heating parameters, *Turkish Journal of Engineering and Environmental sciences*, Vol. 26 pp.59-64.
- 6 Elder J. P. (1983), Proximate analysis by automated thermogravimetry, *Fuel* 62, pp. 580-584.
- 7 Elphick J. O. (1960), Analysis of very small samples of New Zealand coals, *Fuel* 39, pp. 183-186.
- 8 en.wikipedia.org/wiki/coal
- 9 [en.wikipedia.org/wiki/coal seam fire](http://en.wikipedia.org/wiki/coal_seam_fire)
- 10 [en.wikipedia.org/wiki/spontaneous-combustion](http://en.wikipedia.org/wiki/spontaneous_combustion)
- 11 Gamson P. D., Beamish B. B., Johnson, D. P. (1993), Coal microstructure and microporosity and their effects on natural gas recovery, *Fuel* 72, pp. 87-99.
- 12 Gray V. R. (1983), *Coal analysis in New Zealand*. Coal Research Association of New Zealand (Inc.), NZERDC report 97, pp. 75.
- 13 I.S. (Indian Standard): 1350 (Part-I) – 1984, *Methods of Test for Coal and Coke: Proximate Analysis*, Bureau of Indian Standards, New Delhi, pp. 3-28.
- 14 I.S. (Indian Standard): 436 (Part I, Sec. 1) – 1964, *Method for sampling of coal and coke: sampling of coal, Manual sampling*, Bureau of Indian Standards, New Delhi
- 15 Larkin D. E. (1988), *Compositional analysis by thermogravimetry*, ASTM STP 997. Philadelphia, American Society for Testing and Materials, pp. 28-37.
- 16 Levine J. R., Johnson P. W., Beamish B. B., (1993), High pressure microbalance sorption studies, *The 1993 International Coalbed Methane Symposium proceedings Birmingham, Alabama, The University of Alabama/ Tuscaloosa*, vol. 1, pp. 187-196.

- 17 Mahadevan V. and Ramlu M.A. (1985), Fire risk rating of coal mines due to spontaneous heating, *Journal of Mines, Metals and Fuels*, 33 (8), pp. 357-362.
- 18 Nanda A. (2010), Correlation analysis of Spontaneous heating of some SECL coals, B.Tech. Thesis, pp. 4-5.
- 19 Nimaje D.S., Tripathy, D.P. and Nanda S.K (2013), Development of regression models for assessing fire risk of some Indian coals, *I.J. Intelligent systems and applications*, 02, pp. 52-58.
- 20 Nimaje D.S, Tripathy D.P, (2010), Thermal studies on spontaneous heating of coal, *The Indian Mining & Engineering Journal*, pp. 10 – 21.
- 21 Ottaway M. (1982), Use of thermogravimetry for proximate analysis of coals and cokes, *Fuel* 61, pp. 713-716.
- 22 Ramlu M. A. (2007), *Mine disasters and mine rescue* Oxford and IBH Publishing Co., New Delhi, 2nd edition, pp.2-15.
- 23 Rosenvold, R. J., Dubow, J. B., Rajeshwar K. R. (1982), Thermal analyses of Ohio bituminous coals. *Thermochimica, acta* 53, pp. 321-332.
- 24 Warne S. St J. (1991), Proximate analysis of coal, oil shale, low quality fossil fuels and related materials by thermogravimetry, *Trends in analytical chemistry*, vol. 10, pp. 195-199.