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## **Review of Experimental Methods to Determine Spontaneous Combustion Susceptibility of Coal – Indian Context**

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### **ABSTRACT**

This paper presents a critical review of the different techniques developed to investigate the susceptibility of coal to spontaneous combustion and fire. These methods may be sub-classified into the two following areas:

- Basic coal characterisation studies (chemical constituents) and their influence on spontaneous combustion susceptibility.
- Test methods to assess the susceptibility of a coal sample to spontaneously heat.

This is followed by a critical literature review that summarises previous research with special emphasis given to Indian coals.

### **KEYWORDS**

Coal, Spontaneous Combustion, Susceptibility, Characterisation

## 1.0 INTRODUCTION

Coal is an affordable, reliable and abundantly available natural resource that is geographically well-distributed. It is currently the biggest single source of energy used for electricity generation worldwide and provides 40% of the total global energy power generation capacity (Holtberg, 2011). The electrical energy produced by coal generation currently supports 54.5% of the commercial energy supply in India. This plays a key role in underpinning the national economy and industrial growth irrespective of the environmental impact that coal utilisation may produce. Consequently, coal demand in India has risen from 70 million tons (MT) in the early 1970's to 554 MT in 2011-12 (MOC, 2014-15). India is the third largest coal producing country in world with approximately 80% of this production from surface coal mines. The surface mines extract virgin coal seams and/or reclaim the residual coal from previously worked shallow bord and pillar mines to increase the production. The more mature coalfields are the Jharia, Raniganj and Wardha coalfields, whose locations are shown in Fig.1(Joshua, 2011).

The Indian coal industry is currently facing a number of constraints imposed by economic, social and environmental issues. A major economic penalty experienced by the industry is the loss of potentially mineable coal reserves due to spontaneous combustions and uncontrolled fires. Over the last 140 years, the Indian coalfields have recorded a large number of extensive open and concealed fires (Raniganj, 1865; Jharia, 1916) (Dhar, 1996). During 1947–2010, around 40% of the total mining disasters and about 50% of the total fatalities of miners were due to coal mine fire and explosion in Indian coal mines in both underground and opencast mine workings. There are 25 major mine disasters (10 or more fatalities) due to fire and explosion have been recorded in the last ten decades (Table 1)(DGMS, 2011; Jitendra, Kumar, Singh, & Mohalik, 2016). Approximately 70% of these fires are due to spontaneous combustions (Zutshi, Ray, & Bhowmick, 2001). Spontaneous combustion (endogenous fires) of coal, the prime cause for coal mine fire depends on various mining, geological and environmental factors. Moreover, the unscientific exploitation of coal and other exogenous factors makes coal mine fire more catastrophic. These fires create a significant problem to the productivity, safety and environmental impact of these mining operations. The current major coal fires are located in the Jharia, Raniganj, Singrauli and Singareni coalfields (Mohalik et al., 2004; Song, Zhu, Jia, & He, 2014).

The spontaneous combustion of coal can result in the loss of a key national natural economic resource. In addition to the direct financial loss, the fires produce other associated hazards including: i) difficult geo-mining conditions, ii) a sterilisation of potential coal reserves, iii) a hindrance to production iv) increase the risk of explosions, and v) damage to the structure of adjacent properties (Mohalik et al., 2004; G. Singh & Shukla, 2008). Other adverse impacts of spontaneous combustion include the release of poly-aromatic hydrocarbons which have adverse health impacts on local communities and the release of greenhouse gases which contribute to the issue of global warming. These are produced by incomplete combustion of volatile or semi volatile organic compounds (either in saturated, unsaturated, mono-aromatic or polycyclic aromatic forms) and irritant to respiratory tracks causing several health problems (Melody & Johnston, 2015). The estimated GHG emission from coal mine fire in global scale varies 12 kg CO<sub>2e</sub> yr<sup>-1</sup> to 8200 kg CO<sub>2e</sub> yr<sup>-1</sup> approximately (Carras, Day, Saghafi, & Williams, 2009; Melody & Johnston, 2015; O'Keefe et al., 2010).

The determination and prediction of spontaneous combustion/fire of coal plays an important role for mine planners, operators, regulators and finally mine closure planners. In this scenario this paper presents a critical review of the different techniques developed to investigate the susceptibility of coal to spontaneous combustion/fire.

## **2.0 SPONTANEOUS COMBUSTION MECHANISMS**

Spontaneous combustion of coal occurs when it is exposed to the atmosphere in natural conditions. However, various coals react differently to spontaneous combustion when exposed to similar atmospheric conditions. The process is a very complex physico-chemical reaction caused by several factors (intrinsic and extrinsic). The action of each agent is not well understood, but oxidation seems to be responsible for the observed compositional and structural changes. In the past, this behaviour of coal has been attributed to the intrinsic (density, calorific value (CV), moisture content (M), volatile matter (VM), fixed carbon (FC), ash (A), maceral content and rank, total iron, non-pyritic iron, total sulphur, pyritic sulphur, organic sulphur and sulphate sulphur contents of coal) as well as the extrinsic properties (particle size, geological condition, mining methods and environmental condition) of coal (Morris & Atkinson, 1988). There are several mining factors (including: mining methods, rate of advance of extraction, panel dimension, percentage of coal already extracted, coal residues in goaf, pillar stooks (residual coal pillars in underground depillaring operation), heat from machines, crushing, packing, stowing, roof and floor strata partings, changes to the ventilation pressure, barometric pressure and humidity), geological factors (including: seam

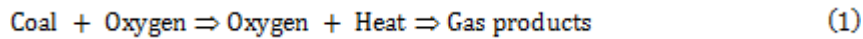
thickness, seam gradient, caving characteristics, depth of cover, geothermal gradient, nature and composition of strata, tectonic disturbances, faults, joints, cracks, fissures, and organic minerals) and environmental factors (solar intensity, air temperature, air pressure, air humidity, wind speed, wind direction) which influence the process of spontaneous combustion in coal (Morris & Atkinson, 1986; Ozdeniz, Sivrikaya, & Kelebek, 2015; Sensogut & Ozdeniz, 2005). These factors have been independently reviewed by various researchers (S. C. Banerjee, 1982; Chamberlain & Hall, 1973; Didari, 1988; Feng, Chakravarty, & Cochrane, 1973; Gunes & Gunes, 2002; M. Guney, 1968; D. J. Hodges & F. B. Hinsley, 1964; Kaymakci & Didari, 2002; A.G. Kim, 1977; D. C. Panigrahi & Sahu, 2004; R. N. Singh & Demirbilek, 1987; Van Doornum, 1954). Other potential contributory external factors such as ignition sources including: conveyor belt friction, electrical short circuits, and explosions, as well as ban tulusi (wild basil plant) conflagration, dumping of hot ash, and illicit distillation of liquor leads to spontaneous combustion/fire in coal (Mohalik et al., 2004). Furthermore, during coal oxidation several interconnected chemical reactions might occur. All of these factors contribute to the complexity of this process and explain why there is no general method proposed to explain the susceptibility of a particular coal to spontaneously combust. It is a challenge to explain how all these factors contribute to spontaneous combustion. Among all these factors, the availability of air or oxygen and the particle size of the coal play an important role in different laboratory based techniques to determine spontaneous combustion propensity of coal.

The spontaneous combustion of coal occurs due to an accumulation of the heat liberated following the interaction of the oxygen in the air and the coal at ambient temperature. If the heat produced is not dissipated at a sufficiently fast rate (by radiation, conduction, convection or all three processes), it produces a further rise in temperature, which may accelerate the rate of oxygen sorption and heat accumulation until the ignition temperature of the coal is reached. The poor thermal conductivity of coal also favours heat accumulation (Mohalik, Singh, Pandey, & Singh, 2006; J. Zhang, Wagner, Prakash, Mehl, & Voigt, 2004). The essential conditions for spontaneous combustion to occur in a mining area without the influence of external factors are:

- a. The coal MUST react with oxygen,
- b. The reaction MUST be exothermic accompanied by production of heat,
- c. The rate of heat production MUST be greater than rate of heat dissipation,

The phenomenon of spontaneous combustion is caused by two interrelated process, namely the interaction between oxygen and coal (oxidation) and the exothermic reaction (heat shedding process) that leads to heat retention. Previous research studies have

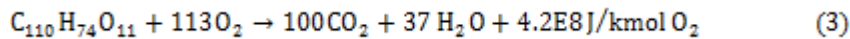
concluded that during spontaneous combustion, oxygen is adsorbed by a purely physical process, which rapidly converts into a chemical chain reaction resulting in the oxidation of certain constituents of coal with the production of small quantity of heat and releasing CO and/or CO<sub>2</sub>. The oxidation of coal starts with exothermic chemical reactions that are represented by equation 1 (Falcon, 2004; Itay, Hill, & Glasser, 1989; Kuenzer et al., 2007; A.S. Myles & Glasser, 2005a; A.S. Myles & Glasser, 2005b; Pone et al., 2007; Rosema, Guan, & Veld, 2001; D. Schmal, 1989; Sinha & Singh, 2005; Stracher & Taylor, 2004; H. Wang, Dlugogorski, & Kennedy, 2003).



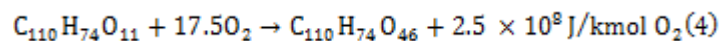
Generally, coal oxidation may be described as a process that involves three sequential steps. These are (i) physical adsorption, (ii) chemical adsorption or chemisorptions, to form coal-oxygen complexes, and (iii) chemical reaction. The chemical reaction breaks down less stable coal-oxygen complexes and results in the formation of gaseous products such as CO, CO<sub>2</sub>, and H<sub>2</sub>O (Mohalik, Singh, Singh, & Tripathi, 2009). The process may be simplified as follows in equation 2 (Rosema et al., 1999; Sinha & Singh, 2005).



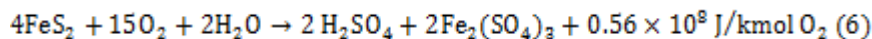
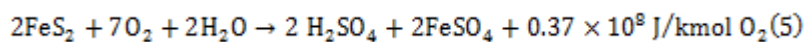
The above reaction may be completed in several steps and pathways. The complete oxidation of a coal may be exemplified by equation 3 (D. Schmal, 1989; Sinha & Singh, 2005).



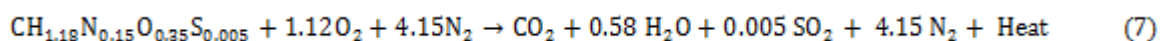
The initial step of the reaction is the chemical absorption or chemisorption of oxygen on the coal surface represented by equation 4 (D. Schmal, 1989; Sinha & Singh, 2005).



In the presence of catalytic substances like pyrite, exothermic reactions as shown in equations 5 and 6 take place (D. Schmal, 1989; Sinha & Singh, 2005; Wilke, 1966).



However, in addition to carbon, the organic component of coal also contains hydrogen, oxygen, nitrogen and sulphur that may result in the initiation of a number of other simultaneous reactions. It has thus been suggested by (Ann G. Kim, 1993; Sinha & Singh, 2005) that the oxidation of coal in air can be better represented by the reaction shown in equations 7:



### 3.0 METHODOLOGY

Different countries have adopted different methods to assess the propensity of coals to spontaneous combustion in the laboratory for their applicability. The objective of

different entity (i.e. mine planners, mine operators, regulators and mine closure planners) of mining operations is to assess the hazard of spontaneous combustion and its related risk to ensure safety of miners and machines. The criteria for determination of spontaneous combustion/fire should be purpose of investigation and its use. Mine planners need information regarding the assessment and prediction of spontaneous combustion prior to design whereas operators need information during mining activity or when moving onto a new areas or seam. The mine regulator/ government agencies may require testing to ensure hazards due to spontaneous combustion/fire are identified and ensure mine management are employing effective counter measures. Mine closure planners need to be able to identify materials that constitute a long-term environmental risk.

All of these investigations have attempted to characterise the coal samples in the laboratory to identify the mechanisms that govern the susceptibility of a particular coal to spontaneously heat under field conditions. The reliability and repeatability of these test results to its applicability or with field conditions/mechanisms plays an important role for selection of testing methods. Similarly the cost, duration of experiment and expertise of user are most concern for different testing methods of spontaneous combustion. These different methods may be broadly classified into three categories: a determination of the chemical constituents, oxygen avidity studies and thermal studies in Table 2 (S. C. Banerjee, 2000). The laboratory test methods used all over world to assess the spontaneous combustion potential of coals may be reclassified into five sub-categories: chemical compositional studies, microscopy studies, thermal studies, oxygen avidity studies and advanced miscellaneous techniques. A summary of these methods are shown in Table 3.

The chemical composition of coal is the most widely used coal characterisation study used to identify their potential use (D. Chandra, Behera, Karmakar, & Tarafdar, 1991; D. Chandra & Prasad, 1990; Miron, Lazzara, & Smith, 1992; Mukherjee & Sarkar, 1997; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi & Saxena, 2001; D. C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000; Sahu, Mahapatra, & Panigrahi, 2009). Amongst the common chemical constituent analyses employed, the proximate and elemental analyses are simple, economical with the analytical equipment widely available. Many researchers have benchmarked the results of their laboratory studies with those available in the literature, to determine the spontaneous combustion tendencies of the coal samples studied. The mineral matter was calculated from the ash content of the coal due to its complexity in terms of techniques and time. Currently the effects of mineral matter content on the propensity of coals to spontaneously combustion are studied using

sophisticated analytical methods (B. Basil Beamish & Arisoy, 2008; B. Basil Beamish & Blazak, 2005; B.B. Beamish, Blazak, Hogarth, & Jabouri, 2005; Humphreys, Rowlands, & Cudmore, 1981 ; A.C. Smith, Miron, & Lazzara, 1988). Basic coal characteristics are required by mine planners for design purposes whereas mine operators require for quality coal production and safety of mines. Similarly, microscopy petrographic studies are widely used due to its simplicity, low cost and wide availability. A geological examination may be used to predict the propensity of a coal to spontaneously heat by performing a study of the maceral compositions and rank of the samples (Avila, 2012; Calemma, Del Piero, Rausa, & Girardi, 1995; D. Chandra, 1958, 1965, 1975; D. Chandra et al., 1991; D. Chandra & Prasad, 1990; Crelling, Schader, & Benedict, 1979; Gray, Rhoades, & King, 1976; Lowenhaupt & Gray, 1980; Marchioni, 1983; Misra & Singh, 1994; Ndaji & Thomas, 1995; Pearson & Creaney, 1981; Stach et al., 1982). These tests are required by consumer of coal users as well as mine operators for quality coal production. Thermal analytical studies are widely used all over world, and have developed into a mature technique to determine liability of a coal to spontaneously heat. In thermal studies, researchers have employed a range of different methods, including: heat based measurement -crossing point temperature (CPT) and ignition point temperature (IPT), modified CPT, puff temperature (PT), Olpinski index (Bagchi, 1965; S. C. Banerjee, Banerjee, & Chakravorty, 1970; Chamberlain & Hall., 1973; Chen & Chong, 1997; Feng, Chakravorty, & Cochrane, 1973; Ganguli & Banerjee, 1953; M. Gouws & L. Wade, 1989a, 1989b; Kreulen, 1948; D. S. Nimaje, Tripathy, & Nanda, 2013; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000; S. W. Parr & Coons, 1925; Sahu, Padhee, & Mahapatra, 2011; Tideswell & Wheeler, 1920); Calorimetric studies - adiabatic calorimetric (SHT - USA, R70 - Australia), isothermal calorimetric (B. Basil Beamish, Barakat, & George, 2000; B. Basil Beamish, Barakat, & St. George, 2001; Chen & Stott, 1993; Clemens, Matheson, & Rogers, 1991; Cliff, Rowlands, & Sleeman, 1996; Cudmore, 1988; Davis & Reynolds, 1928; M. Guney & Hodges, 1969; Humphreys et al., 1981 ; Ren, Edwards, & Clarke, 1999; Rosin, 1929; A. C. Smith & Lazzara, 1987; J. B . Stott, 1956); thermal analysis - differential thermal analysis (DTA), differential scanning calorimetric (DSC) and thermogravimetric analysis (TGA) (S. C. Banerjee, 2000; S.C. Banerjee & Chakraborty, 1967; Filho & Milioli, 2008; Gil, Casal, Pevida, Pis, & Rubiera, 2010; Idris, Rahman, & Ismail, 2012; Idris et al., 2010; Khare, Baruah, & Rao, 2011; M. V. Kok, 2012; C. Lu, Zheng, & Yu, 2009; Mahajan & Walker Jr, 1971; Marinov (a), 1977; Marinov (b), 1977; Marinov (c), 1977; Marinov, 1977; Mohalik, 2013; Mohalik, Panigrahi, & Singh, 2009; Mohalik, Panigrahi, & Singh, 2010; D. C. Panigrahi & Saxena, 2001; Slovak, 2001; Whitehead & Breger, 1950), to determine the susceptibility of coal towards spontaneous combustion. The principle of CPT, IPT, modified CPT and the basket heating test method

are similar. Mine regulators as well as mine operators requires these tests to predict spontaneous combustion/ fire risk to minimise hazards for ensuring safety of miners and machine for sustainable growth. The oxygen avidity studies mainly include the performance of a peroxy complex analysis (S. C. Banerjee, 2000; Sen, Srivastava, & Singh, 2009), rate of reaction studies (Filho & Milioli, 2008; Gil et al., 2010; Idris et al., 2012; Idris et al., 2010; Khare et al., 2011; M. V. Kok, 2012; C. Lu et al., 2009; Slovak, 2001), the determination of the Russian U-index (S. C. Banerjee, 2000). The wet oxidation potential and other oxidation methods are not now so widely used (S. C. Banerjee, 2000; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi, Udaybhanu, & Ojha, 1996; S. Ray & Panigrahi, 2015; S. K. Ray, Panigrahi, & Varma, 2014; Tarafdar & Guha, 1989). A few research workers have employed these methods, but the results are not significant when compared to the other methods. Similarly, the IR/FTIR (Iglesias, de la Puente, Fuente, & Pis, 1998; Low & Tascon, 1985; Materazzi & Vecchio, 2010; D. Wang, Zhong, Gu, & Qi, 2010), XRD (1975; 1998), NMR (Kalema & Gavalas, 1987; Sen et al., 2009; Yokono, Iyama, Sanada, Shimokawa, & Yamada, 1986), GC and GC-MS methods (Jo et al., 2013; Kalema & Gavalas, 1987; P. Lu, Liao, Sun, & Li, 2004; Pisupati & Scaroni, 1993; Sen et al., 2009) are not now widely used due to the associate costs and the availability of the equipment. Advanced techniques are requiring for the structural behaviour of coal towards innovation of science and mine closure planners for long term environmental risk as well as sustainability.

Amongst these assessment techniques: the CPT method is widely used in India, South Africa, Poland, China and Turkey; the DTA method is used in India and South Africa; the isothermal and adiabatic calorimetric methods are used in UK, USA and Australia. The puff temperature and Olpinski index methods are widely used in Russia. The development and applicability of these methods are critically appraised in the following sections.

### **3.1 Chemical Compositional Study**

#### **3.1.1 Proximate and Ultimate Analysis**

The basic chemical compositions of a coal may be determined by results of a proximate and elemental analysis. These are the universally accepted analytical methods to characterize coals for utilization (D. Chandra, Singh, & Singh, 2000; Krevelen, 1981; Sarkar, 1994). Some researchers have cross correlated the results of these analyses to the propensity of the coals to spontaneous combustion (D. Chandra et al., 1991; D. Chandra & Prasad, 1990; Miron et al., 1992; Mukherjee & Sarkar, 1997; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi & Saxena, 2001; D. C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000; Sahu et al., 2009). Panigrahi and Saxena (2001) studied the



intrinsic properties of coal from Jharia coalfields (JCF) of India by carrying out a proximate, elemental and petrographic analysis. They concluded that the susceptibility index of a coal sample to spontaneous combustion could be predicted from an assessment of the porosity and the moisture, volatile matter, ash content of the samples. The percentage of fixed carbon (FC) and volatile matter (VM) was concluded to increase with depth and reverse in nature for ash and moisture (Mohalik, 2013). The total of mass fraction of a sample attributable to carbon, hydrogen, nitrogen and sulphur are observed to stay increase or remain constant as depth increases, whereas the mass fraction of oxygen is observed to decrease with depth(Mohalik, 2013).

### **(a) Moisture Content**

The effects of moisture content (MC) within coal and in the surrounding environment on the susceptibility of a coal to spontaneously combust have been investigated by several researchers with no firm conclusions being drawn to date (Akgun & Arisoy, 1994; Avila, 2012; Bhat & Agarwal, 1996; Bhattacharyya, 1971; Didari, 1988; Mahajan & Walker Jr, 1971; Ren et al., 1999; Dick Schmal, Duyzer, & van Heuven, 1985; J. B. Stott, 1960). The moisture content of a coal may be found in different phases i.e. free moisture (chemically bound moisture is not included) which may influence the susceptibility of a coal to spontaneously combust(Chen, 1994). Some researchers have concluded that a low moisture content may assist rather than retard the combustion whilst a high moisture retards the combustion(B. Basil Beamish & Hamilton, 2005; McPherson, 1993 ). There have been many experimental studies of the effect of moisture on the spontaneous combustion process under extreme conditions for the interaction of coal and gas (air, O<sub>2</sub>, N<sub>2</sub> etc.) However, it is not yet well understood how the moisture affects the tendency of a coal to self-combust and what is the mechanism of adsorption and desorption. Ren et al. (1999) concluded that moisture had a major influence on the early stages of combustion. Nandy et al.(1967)studied the influence of moisture on the self-heating characteristics of coal and observed that there was a critical moisture value around 5% at which the coal was most prone to self-oxidation (Sondreal & Ellman, 1974). Raju (1988 ) concluded that the measured CPT decreases with an increase in moisture content up to 4 to 6%, and that the self-heating characteristics of coals are generally influenced by both the source of moisture, i.e. the moisture in the coal and humidity in the air. However, the inherent moisture in coal plays a more significant role in spontaneous combustion. Typically, it has been found that a coal reacts with oxygen more rapidly when the coal is wet rather than when it is dry (Bhattacharyya, Hodges, & Hinsley, 1968 ; D. J. Hodges & F. B. Hinsley, 1964). In surface stockpiles, the alternative drying and wetting of the coal accelerates the combustion process due to the desorption or absorption reactions (Clemens & Matheson, 1996; M. Guney, 1968; D. J. Hodges & F.

B. Hinsley, 1964; McPherson, 1993 ). The ability of coal to absorb the moisture present in stockpiles depends upon the porosity, permeability and internal surface area. Guney et al. (1968) have also established that temperature-time relationship would be the most practicable for characterising the tendency of coal to spontaneous combustion. The inherent moisture content of Barapukuria coal is 2.73% and the total moisture content value varies from 5.82% to 12.75% suggesting that, these coals are moderately liable to spontaneous combustion(Ahamed1, Monir1, Biswas, & Khan, 2016).

#### **(b) Volatile Matter Content**

A number of researchers use volatile matter (VM) content in combination with other parameters to assess the susceptibility of a coal to spontaneous combustion(S. C. Banerjee, 2000; D. C. Panigrahi & Saxena, 2001). The non-aromatic coal structure (functional groups or side chains) are major contributors to the volatile matter content of coal and plays an important role in the spontaneous combustion of coal. Some authors report that the VM of oxidised/ weathered coal is greater than that of fresh coal. In general a high VM content of coal is directly proportional to spontaneous combustion risk. Raju(1988)observed that the CPT decreases with an increase in the VM content up to a value of 35% after which there is no significant variation in the crossing point temperature observed (Raju, 1988 ). Chandra et al. (1991) also concluded that the VM content and crossing point temperatures decreased with an increase in coalification, which suggests a progressive increase of spontaneous combustion tendency with a corresponding decrease in the age of the coal(D. S. Pattanaik, P. Behera, & B. Singh, 2011).

#### **(c) Ash Content**

The ash content (AC) of coal is determined from proximate analysis. A number of researchers have concluded that the liability of a coal to spontaneously combust ash content may have on the liability of coals to spontaneously combust decreases as the AC increases (D. C. Panigrahi & Sahu, 2004). Beamish and Blazak (2005) and Beamish et al. (2005)showed a strong negative correlation exists between ash content and  $R_{70}$  self-heating rate for both low and high rank coals.

#### **(d) Fixed Carbon and Carbon Content**

The fixed carbon (FC) content of coal is determined from a proximate analysis by difference, by subtracting the % mass of moisture, volatile matter and ash content from 100 whereas, the carbon content is determined from performing an elemental analysis of

the sample. Given that the carbon content of a coal is always high when compared to fixed carbon, as some of the volatile matters will inevitably be included in the determination of total carbon content. Mukherjee and Sarkar (1997) proposed an empirical relationship between the results of an elemental analysis of bituminous coals and their crossing point temperature values. They articulated the relationship as the form:  $CPT (^{\circ}C) = 2(C-(H+O))$ . They found that this derived relationship is highly sensitive to any variation in the petrographic composition, air-dried moisture and ash level of the coal samples. They compared their results with other published data and concluded that the predicted values were within  $\pm 5\%$  of these previously published experimental values.

### **(e) Oxygen Content**

The oxygen (O) content of coal plays an important role in influencing the potential onset of spontaneous combustion. The oxidation of coal may be due to oxygen absorption from atmosphere or may be from oxygen inherent in the coal matrix. The oxygen inherent in coal may exist in two forms: inorganic oxygen (including moisture) and organic oxygen. Normally organic oxygen, determined from an elemental analysis by difference from 100 the values of C, H, N, S on a dry basis, dry ash free basis or dry mineral free basis. In addition, several methods have been used to determine the organic oxygen content in a coal matrix. However, difference method continues to be the standard method recommended by the various international standards organisations like the British Standard Institute (BSI) and the American Society for Testing and Materials (ASTM). The evolution of more modern analytical techniques like fast neutron activation analysis (FNAA) may be used to determine the total oxygen content of coal. Some researchers have employed the oxygen content of coal with other compositional parameters (i.e. H/C vs O/C or (O+N)/C in Seylor classification or Krevelen diagram) to determine the propensity of coal to spontaneously heat (van Krevelen, 1993). Jing et al carried out different experiments varying oxygen concentration of 5%、10%、15%、17%、21% and 30% and observed that coal ignition decreased as oxygen concentration increased (SHEN Jing, ZHU Hong-qing, & Zhen, 2015).

### **(f) Sulphur Content**

The sulphur (S) often present in coals in the form of pyrites has long been recognised as a potential contributory factor to the susceptibility of coal to spontaneous combustion (S. Parr & Hilgard, 1925). It is well known that low sulphur content coals are prone to initial heating. However, it is altogether probable that sulphur is not always the source of problematic. Under certain conditions sulphur in the pyritic form may set up oxidation processes which may account for this initial heating (S. H. Li & Parr, 1926). It has been

observed that coal even in the absence of sulphides would absorb oxygen. However, further research work modified this view and led to the present theory that pyrites plays a subsidiary role in promoting deterioration and spontaneous combustion (S. C. Banerjee, 2000; D. Chandra & Prasad, 1990; Didari, 1988; R. N. Singh & Demirbilek, 1987). Chandra and Prasad(1990) also observed that the pyrite in the coals from the Raniganj coalfield had no influence on the propensity of the of the coal seams to spontaneously heat. Miron et al.(1992)found that pyrite oxidation was the prime cause of the heating in underground mines. Coal samples with different pyrite contents (0 %, 3 %, 5 %, 7 % and 9 %) were artificially mixed with coal to know the spontaneous combustion propensity in laboratory. The study reveals that pyrite contents of 5 %-7 % in coal have the most significant effects on spontaneous combustion within the range of this study(Deng, Ma, Zhang, Li, & Zhu, 2015).

### **3.12 Mineral Matter**

The effect of mineral matter (MM) content on the spontaneous combustion of coal was not investigated intensely except the effect of the pyrite content of coal as described in above section. The presence of many other chemicals may also affect the rate of oxidation to some extent, either accelerating or retarding it. The results of a number of recent studies have demonstrated that the addition of a range of different minerals and inorganic constituents to a coal may inhibit the onset of self-heating(B. Basil Beamish & Arisoy, 2008; B. Basil Beamish & Blazak, 2005; B.B. Beamish et al., 2005; Humphreys et al., 1981 ; A.C. Smith et al., 1988). In contrast other studies concluded that the addition of some alkalis can act as accelerants, whereas others (i.e. borates and calcium chloride) can act as retardants to spontaneous combustion. Beamish and Arisoy (2008) assessed the theoretical heat sink effect of the mineral matter content of a coal towards susceptibility of spontaneous combustion. However, it was concluded that there was an additional effect that the presence of the mineral matter produced that could not be explained by a heat sink alone. Consequently, it was concluded that the disseminated mineral matter within the coal may therefore inhibit the oxidation reaction due to physicochemical effects.

## **3.2 Microscopy Studies**

### **3.21 Petrographic Studies**

#### **(a) Maceral Compositions**

Petrographic microscopy studies are a tool with which to identify the mineral content and the textural relationships within coal samples. The term maceral was first introduced by Stopes in the year 1935 and later Spackman (1958) who proposed a shorter definition of the term maceral i.e.: "...maceral are organic substance, or optically homogenous

aggregates of organic substances, possessing distinctive physical and chemical properties, and occurring naturally in the sedimentary, metamorphic, and igneous materials of the earth" (Scott, 2002; Spackman, 1958; Stopes, 1935). Early microscopic examination of coal macerals (vitrinite, liptinite, inertinite – semifusinite and fusinite) was pioneered by the use of transmitted, reflected light microscopy. This was subsequently improved by advances in microscope technology and the development of oil immersion techniques with image analysis system led to advancements in coal petrology. Geologists revealed that the number of total counts of maceral with vitrinite rank are useful to determine the susceptibility of coals to spontaneous combustion (Benfell, Beamish, & Rodgers, 1997; D. Chandra et al., 1991; D. Chandra & Prasad, 1990; Morris & Atkinson, 1988). The fusains were determined to be the least reactive, and in general, durainis more reactive than vitrain (Morris & Atkinson, 1988). Some other maceral observations like exinite has a much greater oxidation rate than the other two constituents i.e. vitrinite and inertinite. However, the macerals present in structured inertinite (semi-fusinite, fusinite, resin-inertinite bands of inertinite) have higher microscopic and sub-microscopic porosity which may provide channels for air passage within the coal matrix in comparison to vitrinite (Didari, 1988). It is concluded that the amount of vitrinite and liptinite (exinite) contents influence the spontaneous combustion susceptibility of coal seams, and with an increase in coalification, the liability to spontaneous combustion gradually decreases (Avila, 2012; D. Chandra et al., 1991; D. Chandra & Prasad, 1990; Misra & Singh, 1994; D. S. Pattanaik et al., 2011). If coal seams possess a high content of porous maceral other than vitrinite, e.g. structured inertinite, inertodetrinite, resinoinertinite and fragmented gymnospermous pollen, it becomes susceptible to auto ignition, especially in a sub humid climate. The vitrinite group of macerals are derived from the original cell tissues of the plants from which the coal was formed and form a major component of most coals. A change in the vitrinite reflectance is found to depend upon the environment and temperature and time of exposure. The higher the vitrinite content, the higher is the rate of oxidation, and the higher the rank of the coal, the lower is the rate of oxidation (Kruszewska & du Cann, 1996; Kruszewska, Labuschagne, & du Cann, 1996; D. Pattanaik, P. Behera, & B. Singh, 2011). The liptinite groups are derived from the waxy and resinous parts of plants such as spores, cuticles, plant cell secretion, resins and other membranes of spores including pollen grains and algal bodies. This group of macerals is very sensitive to advanced coalification and in particular the number of liptinite maceral observed on samples begins to reduce in coals of medium volatile rank to become absent in coals of low-volatile rank. The inertinite group of macerals are derived from plant material that has been strongly altered and degraded during the carbonisation process. These are generally less reactive in their behaviour and can be easily distinguished by their relative

reflectance and structures than the vitrinite and liptinite groups. The most dominant sub groups of macerals within the inertinite classification are fusinite and semifusinite. An attempt has been made to predict the concentration of macerals of Indian coals using artificial neural network (ANN) by incorporating the proximate and ultimate analysis of coal. It was found that coefficient of determination between measured and predicted macerals by ANN was quite higher as well as mean absolute percentage error was very marginal as compared to MVRA prediction(Khandelwal & Singh, 2010).

### **(b) Rank or Vitrinite Reflectance**

The rank of a coal depends on the character of the original plant debris from which it was formed and the amount of change that its organic matter has undergone during the period of formation(Prakash K. Singh et al., 2012). An increasing carbon content accompanied by a decreasing oxygen content are the most commonly accepted criteria of defining increasing coal rank. The higher the rank e.g. anthracite, the slower the oxidation process, whilst lignite of low rank, oxidises so rapidly that it is often stated that it cannot be stored after mining without ignition(D. Chandra & Prasad, 1990; A.G. Kim, 1977; Ogunsola & Mikula, 1990; Raju, 1988 ; Ren et al., 1999; A. C. Smith & Lazzara, 1987). Ogunsola and Mukula (1990) studied the spontaneous combustion characteristics of Nigerian coals and concluded that the spontaneous combustion is not simply rank dependent; rather it was more or less coal specific. There are however numerous anomalies to a straight rank order. One part of a seam may be particularly liable to spontaneous combustion a seam of higher rank may prove more troublesome than one of a lower rank or even the same seams in different mines may react differently(P. K. Singh, Singh, Singh, & Naik, 2013). The low rank coals (younger seams in the stratigraphic sequence) were more prone to spontaneous combustion whereas the higher rank coals (bottom of stratigraphic sequence) were less prone to spontaneous combustion(D. S. Pattanaik et al., 2011).

### **3.22 Petrographic Study of Oxidised Coal**

All major maceral groups (vitrinite, liptinite and inertinite) are susceptible to weathering and oxidation with respect to time, temperature and environment settings. Among all the maceral vitrinite is the most vulnerable, whereas the inertinite and liptinite groups are more resistant to oxidation (D. Chandra, 1958, 1962; Ingram & Rimstidt, 1984; Ivanova & Zaitseva, 2006; Marchioni, 1983). Both artificial oxidation and natural weathering cause changes in the petrographic texture of coal. The compositional and structural changes of the vitrinite in coals as a consequence of weathering or aerial oxidation were investigated, both qualitatively and semi quantitatively (i.e. the formation of rims along grain boundaries and micro-fractures, the formation of micro-pores and

micro-fissures, increased relief, coke formation and variation in reflectance) using different petrographic technique (Calemma et al., 1995; Crelling et al., 1979; Gray et al., 1976; Lowenhaupt & Gray, 1980; Marchioni, 1983). The changes observed in the petrographic texture of oxidised coal may be due to the reaction temperature and the chemical changes of coal, which can be determined by studying the change in reflectance and the presence of an oxidation rim (Avila, 2012; Benedict & Berry, 1964; Calemma et al., 1995; Gray et al., 1976; Marchioni, 1983; Nandi, Ciavaglia, & Montgomery, 1977 ; Pearson & Creaney, 1981; Ribeiro, Suárez-Ruiz, Ward, & Flores, 2016). Artificial oxidation due to heating develops brightness of vitrinite particle and at the boundaries of particles (oxidation rim) whereas natural weathering processes result in dull rims (Avila, 2012; Calemma et al., 1995; D. Chandra, 1958, 1965, 1975; Crelling et al., 1979; Gray et al., 1976; Lowenhaupt & Gray, 1980; Marchioni, 1983; Ndaji & Thomas, 1995; Pearson & Creaney, 1981; Stach et al., 1982). Alpern and Maume (1969) suggested that low-rank coals were unlikely to produce rims (Marchioni, 1983). Benedict and Berry (1964) reported on an extensive study of low temperature, laboratory-induced oxidation and concluded that vitrinite reflectance indicated an initial decrease followed by irregular overall increase, which also confirms the findings of Kojima and Ogoshi in 1973 and by the investigations performed in coal stockpiles by Alpern and Maume in 1969 (Marchioni, 1983). Chandra (1958, 1962) measured the change in reflectance of coals heated in air and oxygen and on a series of paired samples of fresh and outcrop coals. He found that naturally weathered outcrop coals had a lower reflectance than did the fresh equivalents sampled at depth and concluded that the variations observed were insignificant in terms of the accuracy of the method. There are a number of studies who have observed irregular cracks, micro fractures and pores that re proposed to be due to oxidation (Calemma et al., 1995; Ingram & Rimstidt, 1984; Ndaji & Thomas, 1995; Stach et al., 1982). The pseudovitrinite was earlier suggested to be formed by mild oxidation early in the coalification process, but it was latter concluded that it is an intermediate process between fresh and weathered vitrinite may be the cause (Benedict & Berry, 1964; Teichmuller & Teichmuller, 1982). Ferrari (1938) reported the formation of high-reflectance rims in coals affected by a mine fire (Marchioni, 1983). Benedict and Berry (1964) studied low temperature laboratory-induced oxidation by petrographic method and concluded that vitrinite reflectance variation during the course of oxidation was significant, showing an initial decrease, followed by irregular overall increase, which is verified by the findings of other researchers (Marchioni, 1983). Theoretically, a combination of short-chain and long-chain aliphatic would provide a variety of bond strengths leading to a gradual breakdown of any kerogen present over a range of temperatures, which would thus extend the plastic range of the coal. The dull/darkened rims observed around the outer edges of vitrinite particles of low rank coal is in contrast

to brighter rims which explain the higher observed reflectance (Bend & Kosloski, 1993). This literature survey reveals the mechanism of oxidation of coal either by weathering or artificial oxidation. There is no standard experimental technique to study oxidation and the researcher did not classify the coals as to their propensity towards spontaneous combustion. Avila (2012) studied thermally altered coals in laboratory by employing both manual and image analysis method to identify a potential link with the susceptibility of a coal to spontaneous combustion. He observed six different thermal morphotype (1-homogenous change of reflectance, 2-oxidation rim, 3-cracks and micro fractures, 4-strong reflectance change and cracks, 5-simultaneous reflectance change and oxidation rim and 6-internal oxidation lines) and classified these observations as either primary (single reaction) and secondary alternation (different reaction regime) in morphological change. He formed the opinion that reflectance change and ratio between homogenous reflectance changes to oxidation rim are directly proportional to the liability of the coal to spontaneous combustion(Avila, 2012). A new method has been developed (i.e. heat the sample up to 300 °C in muffle furnace method) to classify the morphology of oxidised coal. The morphology of oxidised coal is proposed to be divided into three different subgroups i.e. unaltered vitrinite (Vu), vitrinite having oxidation rim (Vor) and vitrinite having homogenous change (Vhc) for study of spontaneous combustion of coals. This proposed morphology classification gives a better indicator for study of spontaneous combustion of coal(Mohalik, 2013). The vitrinite reflectance study was carried out for unheated and oxidised coal for study of spontaneous combustion of coal. Avila (2012) proposed that a the observed change in reflectance (i.e. difference in unheated and oxidised coal) may be used as an indicator to determine the susceptibility of coal to spontaneous combustion. Further studies are required to confirm that this method is suitable to confirm the susceptibility of Indian coals to spontaneous combustion(Mohalik, 2013). Microscopy techniques are very simple experimental methods and need operator's expertise and often time consuming process. The repeatability and reproducibility are good for their reliability to laboratory as well as field condition.

### **3.3 Thermal Studies**

#### **3.3.1 Heat Based Measurements**

##### **(a) Crossing Point Temperature (CPT) method**

A number of previous research studies attempt to establish spontaneous combustion susceptibility index for coal samples based upon their relative ignition temperature, which is related to crossing point temperature of coal. This method entails heating coal samples in an oxidising atmosphere to increase the oxidation process of coal either at a constant temperature or at a programmed heating rate from ambient to the ignition



point temperature of the coal. Most of these previous studies proposed the use of an increasing temperature bath system to surround the test sample, to replicate the in-situ conditions associated with a spontaneous combustion, where the exothermic heat liberated by the seat of the heating is accumulated by the surrounding coal mass which thus gradually increases in temperature. As the coal oxidation process is an exothermic reaction, heat will be evolved thereby increasing the temperature of coal bed, thus reducing the difference between coal and bath temperature. As the temperature of the water bath increases the oxidation rate is enhanced until a point will be reached when the coal bed temperature equals the water bath temperature and then exceed it. This equalisation temperature is referred as the crossing point temperature (CPT) or critical oxidation temperature. The higher the determined CPT value, the less will be the susceptibility of coal to spontaneous combustion (Fig.2) and is used as a risk assessment index in India, South Africa, Poland and Turkey, to classify the propensity of coal to spontaneous combustion.

At the beginning of twentieth century Par and Wheeler (1908) investigated the oxidation of coal due to weathering and quantified the effect of volatile matter content. Following this study, a number of improved experimental techniques and designs for the CPT method apparatus have been developed. The first method developed by Nubling and Wanner(1915) heats powdered coal samples at a steady rate in an oil bath, whilst allowing oxygen to pass through the coal bed (S. C. Banerjee, 2000). Subsequently a number of further modifications to the above experimental parameters and the apparatus design have been proposed (Bagchi, 1965; S. C. Banerjee et al., 1970; Chamberlain & Hall., 1973; Chen & Chong, 1997; Feng, Chakravorty, et al., 1973; Ganguli & Banerjee, 1953; M. Gouws & L. Wade, 1989a, 1989b; Kreulen, 1948; D. S. Nimaje et al., 2013; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000; S. W. Parr & Coons, 1925; Sahu, Padhee, et al., 2011; Tideswell & Wheeler, 1920). The determination of the CPT of a coal depends upon the experimental conditions. Bagchi (1965) optimised the experimental parameters and concluded that a heating rate of  $0.5\text{ }^{\circ}\text{Cmin}^{-1}$  in a glycerine bath and with a flow rate of  $80\text{ mlmin}^{-1}$  of humid air for a bed of 20 g of coal (Bagchi, 1965, 1973). It is noted that this method may on occasions overestimate the CPT values because the heating ramp does not always provide a stationary temperature profile across the sample holder which may create an off centre hotspot in the sample. The determined crossing point temperature is merely a 'snapshot' of the whole process, and it does not reflect the rate at which the sample temperature increases before and after the crossing point(D.K. Nandy, Banerjee, & Chakravorty, 1972).Other research studies have proposed modified techniques to interpret the data generated from the above experiment termed: the

modified crossing point temperature, the liability index, the Feng Chakraborty (FC) modified index, the Mahadev and Ramlu (MR) index, the WITS-EHAC index (M. Gouws & L. Wade, 1989b; M. J. Gouws & L. Wade, 1989; Mahadevan & Ramlu, 1985; D. K. Nandy, Banerjee, & Chakravorty, 1965). Nandi et al. (1965) suggested that both the CPT value and the slope of the time temperature curve (which signifies rate of temperature rise) should both be considered to categorise a coal (S. C. Banerjee, 1985). The CPT is directly proportional to the propensity of a coal to spontaneous combustion. The official Indian government coal classification code enshrined in the Coal Mine Regulations, CMR 1957, state that a coal seam may be classified according to the determined CPT and moisture content (M) of the coal. The classification system is as follows: CPT > 160 °C and M < 2% is poorly susceptible to spontaneous combustion; CPT – 140 °C to 160 °C and M – 2% to 5% is moderately susceptible; CPT – 120 °C to 140 °C and M > 5% is highly susceptible to spontaneous combustion. Nugroho et al. (1996) suggest that both the determined CPT and activation energy can be used to define the propensity of coals and coal blends to self-heat (Y.S. Nugroho, McIntosh, & Gibbs, 1996; Y. S. Nugroho, McIntosh, & Gibbs, 2000). Chen (1999) used several thermocouples to measure the temperature gradient close to the centre of the sample during an experiment and consequently proposed a new method to determine the self-heating risk of coal. Recently Avila (2012) adapted the method of Chen using a spontaneous combustion risk assessment rig within the laboratories at University of Nottingham (UoN). The sponcomb rig sample holder developed has thirteen thermocouples along the inner surface at varying radial and vertical distances and three on the outer surface to determine the CPT of coal samples (Avila, 2012). The spatial thermal profile recorded by these thermocouples may be subsequently analysed. Avila observed that a hot spot was created at the centre of the sample, whose temperature could be used to estimate the risk of spontaneous combustion. This sponcomb rig has recently been used to determine the spontaneous combustion risk of Indian coals by studying both thermal and gas profiles. The experimental methods proposed by Avila (2012) for different coals (mainly from UK, USA and Germany) was slightly modified for this study i.e. heating rate of samples from 0.5 to 1 °Cmin<sup>-1</sup> to reduce time. The new CPT methods both thermal (CPTCT and CPTHR) were proposed to determine the spontaneous combustion of Indian coals. An attempt was carried out to assess the gas profiles liberated during the heating tests (i.e. COCT50, COCT100, H2CT50 and H2CT100). It is proposed that the profile of the gases liberated may be an additional index that may be used to determine the spontaneous combustion risk of a coal. This proposed method needs further study to assess its wider applicability to other types of coals all over world (Mohalik, 2013). The measures of uncertainty and repeatability associated with the CPT methods adopted by previous researchers may be mainly classified in terms of the experimental parameters

that are difficult to standardise e.g. the packing density of the coal samples, the air flow rate and direction passing through the sample and the design of the sample holder. However, despite the reservations documented above, due to its simplicity, low cost it continues to be the most common and widely used technique to assess the propensity of coal to spontaneous combustion.

#### **(b) Basket Heating Test Method**

The operating principles governing this method are very similar to CPT method i.e. a maximum sample temperature is pre-set and a constant rising temperature is applied to the sample until the ignition point is determined. The prepared coal sample is placed within a basket of particular size and shape to behave like solid pack and the oven is allowed to gradually rise up to an upper pre-set temperature level. If the basket of coal fails to ignite then the process is repeated by incrementally increasing the level of the upper pre-set temperature level. These techniques possess a number of limitations e.g. it is difficult to maintain the same sample shape, the trial and error method to determine the ignition temperature is tedious, and the results obtained are not repeatable. However, the experimental data generated during this process may be used to design and operate the safe storage of coal in stock piles. A few research workers have applied this technique to determine the spontaneous combustion of coal (Bowes & Cameron, 1971; Cameron & Macdowall, 1972; Frank-Kamenetskii, 1969; Jones, 1996, 1996 ).

#### **(c) Olpinski Index Method**

The concept behind this method is similar to the basket heating test method other than the size of the pellet sample used (0.3 or 0.4 g) is smaller. The pellet of powdered coal is heated at a constant rate in a quinoline vapour bath with a current of air flowing past the coal pellet, which is similar to CPT method. The time versus temperature curve is continuously recorded until a temperature of 235 °C is reached (boiling point of quinoline) and the rate of temperature rise of the sample at this point will determine the propensity of the coal to spontaneously combust. This method is popular in Poland to categorise the propensity of a coal to self-heat, and is called the  $S_{za}$  index. If the method is modified for ash correction, the resultant measure is called the  $S_{zb}$  index. A value of  $S_{zb}$  above 120 indicates that a coal is highly susceptible to spontaneous combustion. However, it has some limitation with respect to repeatability; pellet size; hard coal ignition point more than 235 °C and doubts of coal swelling in quinoline vapour (S. C Banerjee, 1985). Nimaje and Tripathi (2013) carried out statistical analysis between Intrinsic properties viz. proximate and ultimate analysis; and susceptibility indices like crossing point temperature, flammability temperature, Olpinski index and wet oxidation

potential method of Indian coals to ascertain the liability of coal to spontaneous combustion. Best correlated parameters (ultimate analysis) were used as inputs to the radial basis function network model and observed that Szb provides better fire risk prediction of Indian coals to spontaneous combustion (D. Nimaje & D. P. Tripathy, 2016; Devidas S. Nimaje, 2015; Devidas S. Nimaje & Tripathy, 2015; D. S. Nimaje & D. P. Tripathy, 2016; D. S. Nimaje et al., 2013).

#### **(d) Chen's Method**

The governing principles behind this method are based on the transient solution to the heat and mass transfer of the tested sample and produces similar results to the CPT method. In the application of the CPT method, the temperature of the heated coal sample is compared to the bath temperature (outside sample holder). For this method the central core temperature of the sample is compared to the different radial temperature measured within the sample. Consequently, a number of thermocouples are placed within the sample holder to determine the radial temperature profile of the sample from the central core. At a particular point of time the heat flux disappears at a point near the centre of the sample. This method of assessment of spontaneous combustion of coal, particularly used to study the apparent chemical kinetics of coal oxidation is of relatively recent origin (Avila, 2012; Chen & Chong, 1997). The rate of heat release and kinetics can be directly estimated and compared to the propensity of the coal to spontaneously heat. The challenge of this method is to maintain the sample holder at the centre of the heating medium. In addition, the experimental parameters are difficult to standardize, including: the sample holder position, the packing density of the sample in the sample holder, the sample particle size distribution, the reactor design and air gap between reactor and sample holder (all of which affect the exposure of the sample to the reacting air/oxygen in the presence of the heating rate of the source). The spontaneous combustion assessment rig at the University of Nottingham was adapted by Avila (2012) to determine the temperature profile within the heated coal sample and thus compared the relative propensity of different coals towards spontaneous combustion.

#### **(e) Critical Air Blast Method**

When heated dissimilar coal samples will react differently in the presence of various reactive gases (air/ oxygen/ carbon dioxide) and in particular reactive coals will oxidise faster. The coal oxygen interaction defined as oxy-reactivity can be measured by employing the critical air blast technique. The critical air blast is defined to be the minimum rate of air blast to produce the reactivity of coal to air, and is found by trial and error methods. The more reactive the coal towards air, the lower is its determined

CAB value. The test rig consists of a combustion chamber, a metal gauge disk, a heating element, an air flow control system, a gas flow meter, a dome and a drying tower. The method is similar to the heat basket method and a metallic wire gauge supports the coal bed in the combustion chamber. The technique is not popular as they have number of practical limitations similar to the heat basket methods (D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000).

#### **(f) Puff Temperature Method**

This method was pioneered in Russia during the mid-20<sup>th</sup> century. The coal test sample is mixed with some additives ( $\text{NaNO}_2$ ) in defined proportions (usually 2:1) and placed on a temperature controlled reactor bed. The temperature measurement of the sample was measured with respect to time to determine when the sample bed undergoes ignition known as the puff temperature (ignition point temperature). This method may be modified by changing the chemicals (replacing  $\text{NaNO}_2$  by  $\text{H}_2\text{O}_2$ ) which produces a lower puff temperature of the sample. The method is not popular due to the lack of a consistent repeatability of results, but is often used to give a supportive index to compare the propensity of sample towards spontaneous combustion(S. C. Banerjee, 2000).

Heat based measurement techniques are very simple experimental methods and often time consuming process. The repeatability and reproducibility are good for their reliability to laboratory as well as field condition. Among these methods CPT method is widely used by regulators of all over world for ensuring safety of mines and miners. The basket heating test method are widely used for coal consumers specifically safety of coal stocks and other industries like food processing and pharmaceutical industries.

### **3.32 Calorimetric Studies**

#### **(a) Adiabatic Calorimetric Method**

This method is more widely used in the UK, USA, Australia and New Zealand. The coal samples are placed inside a reaction vessel (i.e. adiabatic oven or oil bath) such that the heat losses are not dissipated from the reactor and the reactor temperature is adjusted periodically to follow the temperature rise of the sample. The reacting gas (air/oxygen) is passed through reactor. The oven temperature and sample temperature are maintained using a controlled heating programme. This method is generally accepted to replicate the in-situ condition for determination of spontaneous combustion of coal (Cliff et al., 1996; Davis & Reynolds, 1928). The ignition temperature, rate of rise temperature and kinetic constant of coal are used to predict the propensity of coal to spontaneous combustion(Ren et al., 1999). Rosin (1929) argues that the ignition point

temperature determined from the adiabatic test is dependent upon the experimental conditions and cannot be used to compare the liability of coals to spontaneous combustion. The low ignition temperature is not the primary cause of spontaneous combustion. A number of investigators have highlighted the important role that moisture and humidity of air/oxygen flow may play under adiabatic conditions, although it is not clear to what extent it affects the liability of coals to self-heat (Cudmore, 1988; M. Guney & Hodges, 1969; Rosin, 1929). Several authors have also combined these methods to re-examine historical spontaneous combustion records, chemical and petrographic properties to classify spontaneous combustion of coal (Moxon & Richardson, 1985; Ren et al., 1999). In Australia and New Zealand this test is called the  $R_{70}$  test (B. Basil Beamish et al., 2000; B. Basil Beamish et al., 2001; Humphreys et al., 1981). Whereas in the USA they call this the self-heating temperature which is used to determine spontaneous combustion risk of a coal (A. C. Smith & Lazzara, 1987). In some countries the basic design of the adiabatic oven used and the test procedures employed are modified (Chen & Stott, 1993; Cliff et al., 1996; Ren et al., 1999; A. C. Smith & Lazzara, 1987). It has some limitations to their standardisation and experimental procedures i.e. several hours to run one experiment, design of reacting vessel and amounts of sample, diverse particle size and air/oxygen flow rate.

#### **(b) Isothermal Calorimetric Method**

This test method is similar to the heat basket method where the coal sample is placed in an oven at a pre-set temperature and both time and temperature are measured to get change in temperature of sample. If the samples do not change during an experiment, then the experiment is repeated by increasing the pre-set temperature of the oven. This measured rate of production of thermal energy at constant temperature has been used as a measure of spontaneous combustion propensity of coal. Few researchers have used this method where the propensity towards spontaneous combustion is measured in terms of the rate of oxygen consumption during test (Clemens et al., 1991; J. B. Stott, 1956). It has similar limitations as documented for the heat basket method above. The repeatability and reproducibility of laboratory results are often intricate to field condition. Experimental techniques are often time consuming process.

### **3.33 Thermal Analysis Method**

#### **(a) Differential Thermal Analysis (DTA)**

The differential thermal analysis technique, a method originally used by Le-Chatelier, involves heating a small test specimen, at a constant rate and continuously recording the instantaneous temperature difference ( $\Delta T$ ) between it and an identically heated inert reference material as a function of the temperature ( $T$ ) prevailing in the inert medium.

The resultant thermograph, a record of temperature difference ( $\Delta T$ ) against temperature ( $T$ ) with its characteristic heat changes and intensities, reflects the physical and/or chemical changes of the material at the particular temperature and is characteristic of the material used (Mohalik, Panigrahi, & Singh, 2009; Whitehead & Breger, 1950).

The earliest recorded work in the literature with respect to the application of this technique to the assessment of the spontaneous combustion of coal was carried out by Whitehead & Berger (1950). This technique has subsequently been used by a number of researchers to assess the susceptibility of coals to self-heat (Whitehead & Breger, 1950). A critical literature review by Mohalik et al (2009) concludes that there is no consensus on the experimental parameters chosen in the application of DTA techniques. If the experimental parameters chosen are different, then the results of two samples analysed under two different experimental conditions will not be comparable to determine the proneness of the samples to spontaneous combustion (Mohalik, Panigrahi, & Singh, 2009). Banerjee and Chakravorty (1967) recommended the use of some experimental parameters, but a few other parameters like flow rate of reacting gas, packing density (the ratio of sample weight to sample holder capacity) were not defined. They proposed that the derived DTA thermograph may be divided into three stages. They proposed that the slopes of stage II and the temperature initiation of stage III can be used to categorise the propensity of the coals to spontaneous combustion (S. C. Banerjee, 2000; S.C. Banerjee & Chakraborty, 1967; D. C. Panigrahi & Saxena, 2001). This method is used in India and South Africa to categorise coals, although it is not included as one of the current recommended Indian coal mine regulation standards. A few research studies have suggested that this technique is more useful to understand spontaneous combustion mechanisms rather than for coal categorisation. The technique provides both qualitative and quantitative measures, but is limited by its lack of repeatability and the ability to reproduce the same DTA curve under the same conditions. The nature of the curve i.e. peak width and depth varies with small change in sample holder and furnace replaced (S. C. Banerjee, 2000). Sometimes the value of these indices contradicts the assessment produced by other methods but these contradictions may often be resolved by standardising the experimental parameters using rigorous testing of samples from all over world. The DTA studies shows that the lower first endothermic peak temperature range are highly prone to spontaneous combustion (D. S. Pattanaik et al., 2011).

#### **(b) Differential Scanning Calorimetry (DSC)**

Differential scanning calorimetry is a technique in which the difference in energy inputs delivered to a substance and a reference material is measured as a function of temperature whilst both materials are maintained at a controlled temperature. In this

technique any difference in the energy measured in sample as compared to reference pan is directly proportional to physical and/or chemical transformation undergone by the sample. The earliest research was carried out by Mahajan et al. (1971) to study the spontaneous combustion of coal using the DSC technique (Mahajan & Walker Jr, 1971; Mohalik, Panigrahi, Singh, & Singh, 2009). Mohalik et al (2009) reveals that there is no unanimity on the experimental parameters used by different researchers in the execution of the DSC method (Mohalik, Panigrahi, & Singh, 2009). Mohalik et al propose a standardisation of the optimised experimental parameters employed (Mohalik et al., 2010). The experimental studies of Mahajan et al. (1971) were limited to the assessment of only four Indian coal samples. The data analysis and interpretation is important for comparison of different samples towards spontaneous combustion. There is no clear guidance to compare the propensity towards spontaneous combustion between two samples, but a standardisation of the experimental parameters used across the testing of a wide spectrum of coal would be a profitable future direction of research effort.

### **(c) Thermogravimetric Analysis (TGA)**

This technique is based on loss of sample mass at different temperatures as a result of heating. In this technique a given mass of sample is heated via a programmed heating process and plotted against temperature/ time are termed thermo-gravimetric (TG) curves. The derivative of the TG curve is known as differential thermo-gravimetric (DTG) curve. When this technique is applied under oxidizing conditions, the curve obtained is called the "combustion or burn profile" of the sample (Fig. 3) and under inert atmosphere (nitrogen/ argon/helium) termed pyrolysis.

A number of research investigations have applied the TG technique to study the characteristics of a coal sample and in particular its propensity towards spontaneous combustion. The earliest studies were performed by Ciuryla and Welmar (1979). In both coal pyrolysis and combustion studies the particle size distribution of the samples, the weight of the coal sample, the reaction gas, the flow rate of reaction gas and the heating rate have been shown to be important during characterisation (Mohalik, Panigrahi, & Singh, 2009; Slovak & Taraba, 2010). These studies have clearly revealed that there is no general agreement and unanimity on the choice of the experimental parameters to be used to conduct TGA experiments. If the experimental parameters used in the TGA experiments are different, then the results will be different and therefore will not be comparable. Consequently, there is a need to develop a standard experimental method in the use of TGA methods to determine the proneness of coals to spontaneous combustion of coal. In the analysis of the trends exhibited by a typical TGA curve,



following the desorption of moisture e.g. at the first stage of endothermic reaction there is an increase in weight followed by a sudden inflexion in the mass curve (Marinov (a), 1977; Marinov (b), 1977; Marinov (c), 1977; Marinov, 1977). The smaller the gain lower the ignition temperature and more susceptible to spontaneous ignition (Avila, 2012). However, there are several different interactions taking place at the same time including water vaporisation, oxygen adsorption, volatile release, oxygen desorption and volatile combustion, which affects both weight loss/gain and heat flow. An increase in weight may be interpreted as the absorption of oxygen to form coal-oxy-complexes over the coal surface at different temperatures dependant on the coal type. An increase in weight at the measured temperature may be used to classify a coal as reactive and non-reactive (Avila, 2012). The chemical reactivity of a coal depends upon an endothermic reaction (i.e. 80 to 110 °C) due to the loss of water vapour and numbers of major exothermic reactions i.e. primary and/or secondary and/or tertiary de-vitalisation, which is dependent upon the coal type. The rate of de-volatilisation of different coal depends upon the TGA burning profile and few studies have identified different temperature zone for primary i.e. 110-350°C, secondary i.e. 350 - 650°C and tertiary i.e. 650 - 1000°C. The observed endothermic peak in the stage I for all the DTG curves is indicative of the release of moisture from the samples. The two or three exothermic peaks observed in the stage II are due to a stepwise release of the volatile matter and burning of the heterogeneous organic matter in the coal samples. The exothermic peak recorded in stage III is mainly due to the decomposition of the mineral matter within the samples. The maximum rate of de-volatilisation increases with heating rate for all coals and their mean activation energy. Kinetic parameters (i.e. activation energy and pre exponential factor) of the samples for both the pyrolysis and combustion processes were determined under isothermal and non-isothermal conditions (Sima-Ella, Yuan, & Mays, 2005). Different kinetic models are applied to characterize both raw coal and cleaned coal samples as per their reactivity. These factors are subsequently compared with other parameters, including: the heating rate, peak temperature, different temperature zone, reaction interval, particle size and weight loss (Filho & Milioli, 2008; Gil et al., 2010; Idris et al., 2012; Idris et al., 2010; Khare et al., 2011; M. V. Kok, 2012; C. Lu et al., 2009; Slovak, 2001; W. Zhang et al., 2015). The measured reactivity of the tested coal samples were also studied and cross correlated with other coal parameters including: the mean vitrinite reflectance, structure, fuel ratio and hydrogen/carbon (H/C) ratio. Some research studies have investigated the oxygen chemisorption potential of coals by employing a TGA instrument to study the relationship between the measured amount of heat release and the weight of oxygen chemisorbed as a function of the reaction time. The rate of heat release and weight increase generally decreased with an increasing rank of the original coal. The coal was also investigated in a fixed bed reactor to determine

the influence that temperature and heating rate of the pyrolysis may have on the yield of the products and composition of the gases evolved. The main emission gases produced were identified as  $H_2$ ,  $CH_4$ ,  $C_2H_2$ ,  $C_2H_4$ ,  $C_2H_6$ ,  $C_3H_6$  and  $C_3H_8$  and also minor concentrations of other gases (P. Lu et al., 2004; Seo, Park, Hwang, & Yu, 2010; Seo, Park, Kim, Hwang, & Yu, 2011). The TGA has been used to study coal characteristics for last several decades and Avila (2012) extensively used this method to propose a thermogravimetric index but failed to fully define all of the experimental parameters. The TGA experimental parameters were proposed (i.e. sample size – 212 micron (-72 mesh), mass of sample - 10 mg and flow rate – 40 mlmin<sup>-1</sup> sample gas (reaction gas) and 60 mlmin<sup>-1</sup> balance gas) to determine spontaneous combustion study of coal (Mohalik, 2013). The spontaneous combustion mechanism is clear in slow heating rate (1 °Cmin<sup>-1</sup>) but time consuming whereas higher heating rate (15, 30 °Cmin<sup>-1</sup>) reveals the reverse results. A heating rate of 5 °Cmin<sup>-1</sup> has been proposed to determine the spontaneous combustion risk of a coal by considering both the quality of the results and the time required to complete each experiments. The effect of the application of four heating rate (i.e. 1, 5, 15 and 30 °Cmin<sup>-1</sup>) on the combustion experiments reveals there is increase in the weight recorded in the low temperature zone (200 - 350 °C) before the ignition point of the coal is observed due to oxygen adsorption on surface (Avila, 2012, Marinov (a), 1977). The effect of the application of the four heating rates (i.e. 1, 5, 15 and 30 °Cmin<sup>-1</sup>) during the pyrolysis experiment reveals there is no increase of weight in low temperature zone (200 - 350 °C) before the ignition point of the coal. The increase in weight gain, spontaneous combustion susceptibility indices (SHSI) and the activation energy determined from the TGA experiments may be used to estimate spontaneous combustion of the coals (Mohalik, 2013). Thermal studies are very simple experimental methods, need operator's expertise and often time consuming process. The repeatability and reproducibility are excellent for their reliability in laboratory condition. The laboratory results are yet to be established in field condition in large scale.

### **3.4 Oxygen Avidity Studies**

#### **3.41 Peroxy Complex Analysis**

The most commonly accepted theory of the spontaneous combustion of coal is the physico-chemisorption and chemical reaction process. In this process an unstable coal oxygen complex (peroxy complex) is generated dependent upon the temperatures and the duration. This peroxy complex will eventually decompose with the subsequent liberation of a number of gases including  $CO$ ,  $CO_2$ ,  $H_2O$ . A quantification of these produced gases in terms of oxygen content gives a measure of the mechanism of the spontaneous combustion of coal. A number of research studies have used this concept to develop an analytical method with which to measure the peroxy formation and hence

assess the liability of coals to spontaneous combustion (S. C. Banerjee, 2000). The method is simple and once standardised may be used to broadly classify the susceptibility of coals. In recent years, the use of advanced instrumental techniques (including IR, FTIR, XRD and NMR) have been used to qualitatively and quantitatively measure the formation of the peroxy complexes (Sen et al., 2009).

### **3.42 Wet Oxidation Potential Method**

The wet oxidation potential (WOP) of coal is a three stage process that tracks the stepwise oxidation of a coal leading to: the immediate formation of surface oxides of easily oxidizable species, followed by the formation of colloidal humic acids and finally to small aromatic and aliphatic acids. A chemical wet oxidation of coal may be achieved by a variety of reagents including nitric acid ( $\text{HNO}_3$ ), alkaline and acidic permanganates. It has been reported that an alkaline permanganate oxidation of different coals can produce carbonic, acetic, oxalic and many other benzene carboxylic acids (S. C. Banerjee, 2000; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi et al., 1996; Tarafdar & Guha, 1989). The most vulnerable structures in coal liable to oxidation are the alicyclics or hydro aromatics. The chemical structure of a coal may consist of two parts: the aliphatic or hydro aromatic structure (which are more prone to oxidation) and the condensed aromatic structure (which are resistant to oxidation). The presence of hydroxyl groups in the aromatic structure produces a very high degree of reactivity such that they get oxidised faster. Consequently, low rank coals are more readily oxidised; although this may also be due to a smaller degree of condensation of the aromatic structures within them. On oxidation lower rank coals produce large amounts of aliphatic acids as compared to higher rank coals, this is because low rank coals contain more branched aliphatic hydro-carbons. As high rank coals have structures close to that of graphite, they are less liable to oxidation and the resultant products will contain more aromatics than aliphatic. A typical measurement instrument consists of a beaker (100 ml capacity), one saturated calomel electrode ( $\text{Hg}/\text{Hg}_2\text{Cl}_2/\text{Cl}^-$ ) and one carbon electrode. Both the calomel and carbon electrodes are connected to a mili-voltmeter to measure the potential difference generated during the oxidation process. The beaker containing the electrodes is placed over a magnetic stirrer to ensure that a homogeneous mixture of the coal and the alkali solution is maintained. The teflon coated fish of the magnetic stirrer is placed inside the beaker (D. C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000; D. C. Panigrahi et al., 1996; Tarafdar & Guha, 1989). This technique has been employed by a number of investigators in India to assess the spontaneous combustion potential of coal. However, although it is very useful due to its simplicity and low cost, it requires to be standardised using a wide range of coals from around the world. Altogether 78 coal samples collected from thirteen mining companies spreading over most of the Indian

Coalfields have been used for experimental investigation by varying different experimental conditions to standardize wet oxidation potential method. The experimental investigation clearly indicates that WOP method with 0.2N KMnO<sub>4</sub> in 1N KOH solution at 45 °C (susceptibility index PD12) should be used for determination of susceptibility of coal to spontaneous combustion and the results of these studies are consistent with those obtained for the same coal samples using the CPT method (Banka, Ray, & Panigrahi, 2016; D.C. Panigrahi & Ray, 2014; S. Ray & Panigrahi, 2015; S. K. Ray et al., 2014).

### **3.43 Russian U Index**

The principle of this method is to measure the amount of oxygen absorbed by a given coal sample over a period of one day. This may be indirectly calculated by the measurement of gas generated over a day by a compositional analysis of the gas. Fresh crushed coal samples (100 g) are kept in a glass vessel for one day to enable the absorption of oxygen to form a coal-oxygen complex. After one day a compositional analysis of the air remaining within the gas vessel is measured and the oxygen absorbed by the coal is determined. The oxygen absorption has been shown to be directly proportional to the propensity of the coal to spontaneously heat (S. C. Banerjee, 2000). The limitation to the application of this method is that the oxygen sorption capacity of coal sample over a 24hr period is not the same as propensity of coal oxidation. This method does not provide an accurate prediction for high moisture content coal.

### **3.44 Rate Study or Kinetic Study**

It is proposed that the results of kinetic studies of coal oxidation may also provide a good indicator for the liability of coals to spontaneously heat. A range of different experimental methods have been used by scientists to study the spontaneous combustion of coal but the results of this test can be extrapolated to study coal kinetics. Banerjee et al. (2000) used a reaction rate constant and frequency factor to determine the propensity of coal to spontaneous combustion. The earlier isothermal kinetic studies of coal were more popular, but recently the use of non-isothermal kinetic parameters has been proposed by number researchers as suitable to the study of the spontaneous combustion of coal. It has been shown that the non-isothermal kinetic method is less time consuming than the isothermal kinetic method. It has been proposed that the non-isothermal thermal kinetic method is simpler to use and can provide a good indicator to characterise the propensity of coal to spontaneous combustion (Filho & Milioli, 2008; Gil et al., 2010; Idris et al., 2012; Idris et al., 2010; Khare et al., 2011; M. V. Kok, 2012; C. Lu et al., 2009; Slovak, 2001). The activation energy from TGA experiments may be used for the determination of spontaneous combustion of coal (Mohalik, 2013). The

activation energy increased along with oxygen concentration increasing (SHEN Jing et al., 2015). Oxygen avidity studies are very simple, user friendly, cheap experimental methods and often time consuming process. The repeatability and reproducibility are sometime uncertain for their reliability to laboratory as well as field condition.

### **3.5 Advanced Miscellaneous Techniques**

#### **3.5.1 IR/FTIR Spectroscopy**

Infra-red (IR) and Fourier transform infra-red (FTIR) spectroscopy are the most widely used technique to assess coal oxidation and weathering, and can provide additional coal structural change information (Balachandran, 2014; Di Nola, de Jong, & Spliethoff, 2010; Fang et al., 2006; Huffman, Huggins, Dunmyre, Pignocco, & Lin, 1985; Kister, Guiliano, Mille, & Dou, 1988; M. Kok, 2008; Ledesma, Kalish, Nelson, Wornat, & Mackie, 2000; Liotta, Brons, & Isaacs, 1983; MacPhee, Giroux, Charland, Gransden, & Price, 2004; Mahidin, Usui, Ishikawa, & Hamdani, 2002; Ogloza & Malhotra, 1989; Painter, Snyder, Pearson, & Kwong, 1980; Rhoads, Senftle, Coleman, Davis, & Painter, 1983; Wu, Robbins, Winschel, & Burke, 1988). The changes of IR/FTIR spectra observed for a coal i.e. various bands (increase, decrease, generation of new bands), spectral subtraction and curve fitting gives information about the occurrence of oxidation, which may then be related to the propensity of the coal to spontaneous combustion. The band characteristics of aliphatic C–H stretching with the ratio of aliphatic oxygen-containing compounds decreases with increasing rank (W. Li & Zhu, 2014). Previous researchers have generally accepted that the formation of intermediate peroxide and hydroxides are due to the decomposition of oxygenated hydroxyl groups under low temperature oxidation (Iglesias et al., 1998; Materazzi & Vecchio, 2010; Tang & Xue, 2015; D. Wang et al., 2010). A number of researchers have postulated that the oxidation of coal below 100 °C, involves at least three simultaneous interdependent reactions involving (i) addition of oxygen to form carbonyl species via hydro-peroxide (ii) decomposition of hydro-peroxides to carbon oxides and water at 100 °C and (iii) direct decomposition of carboxyl and carbonyl groups and resultant formations (Gethner, 1985, 1987). Similarly, Photo acoustic – FTIR techniques (non-destructive techniques), TGA-FTIR and FTIR–Photo thermal beam deflection spectroscopy (PBDS) have been used by a few researchers to study low temperature oxidations of coals. Raman spectra parameters (i.e. the band area ratios) can be used to characterize structure changes in the different rank and correlate with the oxidation activity of coal. Higher ranked coal was found to have higher values of ratios (W. Zhang et al., 2015). However, these techniques may have explained the reaction mechanisms associated with the spontaneous combustion of coal, but they did not suggest the classification system to be employed to categorise the propensity of different coals (Low & Tascon, 1985). Infrared studies prove that stronger

absorbance of coals are relatively more prone to spontaneous combustion as compared to other coal seams(D. S. Pattanaik et al., 2011). Peak area values of different functional groups at same temperature from Fourier transform infrared spectroscopy are different for different coal samples. This is due to the different rank of coal samples and the order in which the functional groups are activated during coal oxidation at low temperature. Variations of peak area, band ratios values of each functional group along with temperature in coal are identified to compare propensity of coal towards spontaneous combustion(Hongqing Zhu, Jing Shen, & Chang, 2015; W. Zhang et al., 2015).

### **3.52 XRD Technique**

A number of researchers have used x-ray diffraction (XRD) techniques with other FTIR, scanning electron microscope energy dispersive x-ray (SEM EDX) , Mossbauer spectroscopy and IR spectroscopy to examine the coal oxidation by studying mineral transformation and catalytic action of minerals (Gong, Pigram, & Lamb, 1998; Kelemen, George, & Gorbaty, 1990; M. Kok, 2008). The results obtained from these studies were compared against the results of the coal maceral and other thermals methods to verify the efficacy of these methods. Cole et al. (1975) suggested that the transformation of iron minerals during a low temperature oxidation of coal may be due to the catalytic action of the iron minerals and their oxidation products. It was observed that the iron content mineral pyrite was the most readily altered mineral matter during low temperature oxidation and that the oxidative transformation of pyrite varies with the difference in the sulphur content and its mineralogy(Ribeiro et al., 2016). Iglesias et al. (1998) proposed that thermal oxidation is different from weathering as far as the alternation of minerals is concerned and makes no significant contribution towards oxidation. Similar to the IR/FTIR methods, this technique did not readily provide a classification system with which to categorise the propensity of different coals to self-heating.

### **3.53 Nuclear Magnetic Resonance (NMR) Spectroscopy**

Solid state Carbon 13NMR has been employed to investigate coal oxidation and latterly combined with magic angle spinning (<sup>13</sup>C MAS), cross polarisation and dipolar dephasing techniques to study coal oxidation in low temperature (Mao, Schimmelmann, Mastalerz, Hatcher, & Li, 2010). However, although the conclusions of these studies confirm the reaction mechanisms driving the spontaneous combustion of coal obtained from IR/FTIR, XRD analyses described above, they did not propose a classification system with which to categorise the propensity of different coals to spontaneous combustion(Kalema & Gavalas, 1987; Sen et al., 2009; D.-m. Wang et al., 2016; Yokono et al., 1986). The

<sup>13</sup>C NMR spectra reveal that the aliphatic carbon content decreases progressively with increasing thermal maturity for the replacement of aromatic hydrogens by condensation(W. Li & Zhu, 2014).

### **3.54 Gas Chromatography and GC-MS Method**

This method is similar to the performance of a peroxy complex analysis in that the gaseous products of combustion gases are measured by gas chromatography (GC) or gas chromatography mass spectrophotometer (GC-MS). In the middle of 19<sup>th</sup> century Chinese researchers used this technique to study spontaneous combustion. The physical adsorption of capacity of 1 g of dry coal at 30 °C is determined by gas chromatography which has been adopted as a Chinese national measurement standard to assess the propensity of coal to spontaneous combustion (GB/T 20104-2006) (Sen et al., 2009). Similarly, a number of investigators have studied the pyrolysis of both fresh and oxidised (natural and laboratory induced) coal by GC-MS (Kalema & Gavalas, 1987; Pisupati & Scaroni, 1993). The above Chinese method is not considered comprehensive as the test is only carried out at one fixed temperature, and the reaction kinetics of a coal sample are accepted to be dependent upon both the duration and temperature. The oxygen absorption promoted at low temperature principally depends upon the physical pore structure and the surface area rather than physicochemical reaction. Organic geochemical characteristics of three Chinese spontaneous combustion coals has been observed that they have more compounds with low to medium carbon number (Shu, Xu, Xu, Ge, & Chen, 1996). GC and GC-MS analyses have been used to determine the index gases liberated from a coal bed heated under temperature controlled conditions to determine susceptibility of coal to spontaneous combustion (Jo et al., 2013; P. Lu et al., 2004). Consequently, the results of this method may thus be applied to characterise the liability of coals to spontaneous combustion. The standardisation of this technique is required to achieve its wider acceptability. Advanced miscellaneous techniques are very complex, need operator's expertise, costly experimental methods as well as very high investment and often time consuming process. The repeatability and reproducibility are sometime uncertain for their reliability to laboratory as well as field conditions.

The statistical regression analysis was carried out between the parameters of intrinsic properties and the susceptibility indices. The best-correlated parameters were used as inputs to the soft computing models(Khandelwal & Singh, 2010). The intrinsic properties with the susceptibility indices were used as input to the different Artificial Neural Network (ANN) techniques viz. Multilayer Perceptron (MLP), Functional Link Artificial Neural Network (FLANN), and Radial Basis Function (RBF) to predict in advance the spontaneous combustion/ fire risk of Indian coals(Banka et al., 2016; Devidas S.

Nimaje, 2015; D. S. Nimaje et al., 2013; S. K. Ray et al., 2014). To investigate the appropriateness of this approach, the predictions by ANN are also compared with conventional multi-variate regression analysis (MVRA)(Banka et al., 2016; D.C. Panigrahi & Ray, 2014; D. S. Nimaje et al., 2013; Sahu, Mahapatra, Sirikasemsuk, & Panigrahi, 2011). This can help the mine management to adopt appropriate strategies and effective action plans to prevent occurrence and spread of fire.

#### **4.0 CONCLUSION**

The following conclusions may be drawn from the summary critical literature review presented in Table 4. The results of proximate, ultimate and petrographic constituents of a coal may be used to assess the susceptibility of coal to spontaneous combustion. However, the following gaps in knowledge have been identified. The literature survey reveals that in the Indian context, a limited number of research studies have been attempted to establish a link between all of these intrinsic characteristics and the petrographic constituents of these coals to their susceptibility to spontaneous combustion. Amongst all of the parameters identified, the moisture content of a coal appears to be principal in confirming the susceptibility of a coal to spontaneous combustion. A number of investigations conducted across world have attempted to study oxidation of coals to spontaneous combustion. In an Indian context, there are only few studies conducted by Chandra (1958, 1962) in which he inferred the fundamental mechanisms present. Mine planners and operators need the chemical composition data before mining and during mining. These methods are simple, quickest and cheap as compared to other methods. The results are quite useful for their end use locations i.e. in thermal power plant, coke oven plant, coal stocks etc. Similarly, petrographic analysis (maceral, rank and morphology of oxidised coal) data will be helpful for coal reactivity study for their pre and post combustion behaviour. Mine operators, consumers and mine closure planner need these information for their purpose. The crossing point temperature assessment method is a widely accepted technique to determine the propensity of coal to spontaneous combustion. In particular, in India the CPT is the standard recommended technique by the Directorate General Mine Safety (DGMS) and is prescribed within the statutory Indian Coal Mine Regulations – CMR 1957 to determine spontaneous combustion risk of Indian coals (CMR, 1957). The CPT and moisture content of coal data is required by mine planner to design the mine, mine operators and regulators for ensuring safety of miners and machines. These two techniques are very simple, basic practices, user friendly and often time consuming process. The repeatability and reproducibility are sometime uncertain for their reliability to laboratory as well as field condition. The results of the use of TGA techniques have been widely used to



characterise the spontaneous combustion behaviour of coals but no experimental standard have been established to confirm the robustness and repeatability of tests and to provide a consistent interpretation of the results obtained. The rapid experimental methods are cost effective and reliable with respect to repeatability and reproducibility. The test results with small sample provide sufficient information to know about spontaneous combustion mechanism. Application of soft computing techniques to these data will help to identify and control measure of its risk before or after mining operation.

Consequently, it is proposed to conduct a comprehensive study on a group of Indian coals to identify any link between the results of a TGA analysis and the susceptibility of these coals to spontaneous combustion. However, different countries using various methods to assess spontaneous combustion of coal in laboratory, but still there is a knowledge gap of one single method for testing spontaneous combustion of coal. Some of the techniques viz. chemical constituents of coal, CPT, TGA have been increasingly used to assess the proneness of coal to spontaneous combustion all over world. However, it is conceded that the results of many of these laboratorie analytical techniques may often be contradicted by actual mine conditions due to the influence of extraneous parameters such as mining, geological and environmental parameters.

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Table 1 :Disasters in Indian coal mine due to fire and explosion from 1901 to 2014 (DGMS, 2011; Jitendra et al., 2016).

<b>Sl. No.</b>	<b>Date of Accident</b>	<b>Name of Mine</b>	<b>Persons Killed</b>
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1.	February 16, 1908	Nadir Khan, ECL	20
2.	February 07, 1910	Dishergarh, ECL	11
3.	November 26, 1910	Namdaang, ECL	14
4.	November 09, 1911	Kendwadih, BCCL	14
5.	December 22, 1913	Chowrasi, ECL	27
6.	July 20, 1916	Disergarh, ECL	14
7.	November 18, 1918	Disergarh, ECL	10
8.	February 28, 1921	Amalabad, BCCL	11
9.	March 09, 1922	Khoast, Baluchistan	13
10.	January 04, 1923	Parbelia, ECL	74
11.	June 29, 1935	Bagdigi, BCCL	19
12.	July 24, 1935	Kurhurbaree, CCL	62
13.	January 30 1936	Loyabad, BCCL	35
14.	December 18, 1936	Poidih, ECL	209
15.	March 19, 1946	Begunia, BCCL	13
16.	March 14, 1954	Damra, ECL	10
17.	February 05, 1955	Amalabad, BCCL	52
18.	February 19, 1958	Chinakuri, ECL	175
19.	May 28, 1965	Dhori, CCL	268
20.	March 18, 1973	Jitpur, IISCO	48
21.	October 04, 1976	Sudamdih, BCCL	43
22.	October 22, 1979	Baragolai, ECL	16
23.	June 24, 1981	Jaganatha, MCL	10
24.	January 25, 1994	New kenda, ECL	55
25.	March 03, 1997	New Mogla, J&KMDC	10
26.	September 06, 2006	Bhatdih, Nagda, BCCL	54
27.	May 6, 2010	Anjana Hill, Chirimiri, SECL	15

**Note: ECL-Eastern Coal Limited, BCCL- Bharat Coking coal Limited, CCL- Central Coking Coal Limited. MCL- Mahanadi Coalfield Limited, SECL- South Eastern Coalfield Limited, JKMD- Jammu-Kashmir Mineral Development Corporation, IISCO- Indian Iron and Steel Co. Limited.**

Table 2 :Different test methods to identify the propensity of coals to spontaneous combustion (S. C. Banerjee, 2000).

<b>Chemical constituents</b>	<b>Oxygen avidity studies</b>	<b>Thermal studies</b>
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<ul style="list-style-type: none"> <li>• Proximate analysis</li> <li>• Ultimate analysis</li> <li>• Petrography study</li> <li>• Mineral matter study</li> <li>• Nuclear magnetic resonance spectroscopy (NMR)</li> <li>• Infrared /Fourier transform infrared spectroscopy(IR/FTIR)</li> <li>• Gas chromatography (GC),</li> <li>• Gas chromatography-mass spectrometry (GCMS)</li> </ul>	<ul style="list-style-type: none"> <li>• Peroxy complex analysis</li> <li>• Rate studies</li> <li>• Russian u index</li> <li>• Wet oxidation potential</li> </ul>	<ul style="list-style-type: none"> <li>• Initial temperature</li> <li>• Crossing point temperature</li> <li>• Crossing point and ignition point studies</li> <li>• Modified CPT</li> <li>• Adiabatic calorimetric</li> <li>• Isothermal Calorimetric</li> <li>• Critical Air Blast</li> <li>• Basket Heating Testing Methods</li> <li>• Olpinski index</li> <li>• Russian test of ignition temperature (Puff temperature)</li> <li>• Differential Thermal Analysis(DTA)</li> <li>• Differential Scanning CalorimetryDSC</li> <li>• Thermo-gravimetric Analysis (TGA)</li> </ul>
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Table 3: Different test methods to identify the propensity of coals to spontaneous combustion

<b>Chemical Composition study</b>	<b>Optical Microscopy studies</b>	<b>Thermal studies</b>	<b>Oxygen avidity studies</b>	<b>Advanced Miscellaneous Techniques</b>
<ul style="list-style-type: none"> <li>• Proximate and Ultimate analysis (D. Chandra et al., 1991; D. Chandra &amp; Prasad, 1990; Miron et al., 1992; Mukherjee &amp; Sarkar, 1997; D. C. Panigrahi &amp; Sahu, 2004; D. C. Panigrahi &amp; Saxena, 2001; D. C. Panigrahi, Saxena, V. K, Udaybhanu, G., 2000; Sahu et al., 2009)</li> <li>• Mineral</li> </ul>	<ul style="list-style-type: none"> <li>• Petrographic study of fresh coal (Avila, 2012; D. Chandra et al., 1991; D. Chandra &amp; Prasad, 1990; Misra &amp; Singh, 1994)</li> <li>• Petrographic study of thermally altered coal (Avila, 2012; Calemma et al., 1995; D. Chandra, 1958, 1965, 1975; Crelling et al., 1979; Gray et al., 1976; Lowenhaupt &amp; Gray, 1980; Marchioni, 1983; Ndaji &amp; Thomas,</li> </ul>	<ul style="list-style-type: none"> <li>• Heat based measurement (Bagchi, 1965; S. C. Banerjee et al., 1970; Chamberlain &amp; Hall., 1973; Chen &amp; Chong, 1997; Feng, Chakravorty, et al., 1973; Ganguli &amp; Banerjee, 1953; M. Gouws &amp; L. Wade, 1989a, 1989b; Kreulen, 1948; D. S. Nimaje et al., 2013; D. C. Panigrahi &amp; Sahu, 2004; D.</li> </ul>	<ul style="list-style-type: none"> <li>• Peroxy complex analysis (S. C. Banerjee, 2000; Sen et al., 2009)</li> <li>• Wet oxidation potential (S. C. Banerjee, 2000; D. C. Panigrahi &amp; Sahu, 2004; D. C. Panigrahi et al., 1996; S. Ray &amp; Panigrahi, 2015; S. K. Ray et al., 2014; Tarafdar &amp; Guha, 1989)</li> <li>• Russian U index (S. C. Banerjee, 2000)</li> <li>• Rate studies / kinetic study (Filho &amp; Milioli, 2008; Gil et al., 2010; Idris et al., 2012;</li> </ul>	<ul style="list-style-type: none"> <li>• Infra Red (IR)/Fourier Transform Infra Red (FTIR) Spectroscopy (Iglesias et al., 1998; Low &amp; Tascon, 1985; Materazzi &amp; Vecchio, 2010; D. Wang et al., 2010)</li> <li>• X-ray diffraction (XRD) (1975; 1998)</li> <li>• Nuclear Magnetic Resonance (NMR) study (Kalema &amp; Gavalas, 1987; Sen et al., 2009; Yokono et al., 1986)</li> <li>• Gas Chromatography (GC) or GC-Mass Spectrometry (MS) study (Jo et al., 2013; Kalema &amp; Gavalas, 1987; P. Lu et al., 2004; Pisupati &amp;</li> </ul>



<p>matter study (B. Basil Beamish &amp; Arisoy, 2008; B. Basil Beamish &amp; Blazak, 2005; B.B. Beamish et al., 2005; Humphreys et al., 1981 ; A.C. Smith et al., 1988)</p>	<p>1995; Pearson &amp; Creaney, 1981; Stach et al., 1982)</p>	<p>C. Panigrahi, Saxena, V. K, Udaybhanu , G, , 2000; S. W. Parr &amp; Coons, 1925; Sahu, Padhee, et al., 2011; Tideswell &amp; Wheeler, 1920) Calorimetric study (B. Basil Beamish et al., 2000; B. Basil Beamish et al., 2001; Chen &amp; Stott, 1993; Clemens et al., 1991; Cliff et al., 1996; Cudmore, 1988; Davis &amp; Reynolds, 1928; M. Guney &amp; Hodges, 1969; Humphreys et al., 1981 ; Ren et al., 1999; Rosin, 1929; A. C. Smith &amp; Lazzara, 1987; J. B . Stott, 1956) Thermal analysis combustion (S. C. Banerjee, 2000; S.C.</p>	<p>Idris et al., 2010; Khare et al., 2011; M. V. Kok, 2012; C. Lu et al., 2009; Slovak, 2001)</p>	<p>Scaroni, 1993; Sen et al., 2009)</p>
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		<p>Banerjee &amp; Chakraborty, 1967; Filho &amp; Milioli, 2008; Gil et al., 2010; Idris et al., 2012; Idris et al., 2010; Khare et al., 2011; M. V. Kok, 2012; C. Lu et al., 2009; Mahajan &amp; Walker Jr, 1971; Marinov (a), 1977; Marinov (b), 1977; Marinov (c), 1977; Marinov, 1977; Mohalik, 2013; Mohalik, Panigrahi, &amp; Singh, 2009; Mohalik et al., 2010; D. C. Panigrahi &amp; Saxena, 2001; Slovak, 2001; Whitehead &amp; Breger, 1950)</p>		
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Table 4: Overview of the experimental methods for studying the spontaneous combustion susceptibility of coal

Experimental Method	Brief Description	Parameters Measured	Major Features	Advantages	Limitation	Experimental Parameters	Literature Reported
<b>Chemical compositional study</b>	<b>Basic chemical properties</b>	Proximate, ultimate and mineral matter analysis	Weight loss/remainder of sample	Moisture, volatile matter, ash, carbon, oxygen sulphur and mineral matter study	Standard worldwide acceptable method; Easy to conduct; Cost effective	Time consuming	Size of the sample (-212 micron); mass - 1g; (Akgun & Arisoy, 1994; Avila, 2012; B. Basil Beamish & Arisoy, 2008; 2005; B.B. Beamish et al., 2005; Bhat & Agarwal, 1996; Bhattacharyya, 1971; D. Chandra et al., 2000; Clemens & Matheson, 1996; Didari,

								1988; M. Guney, 1968; D. J. Hodges & F. B. Hinsley, 1964; Humphreys et al., 1981 ; Mahajan & Walker Jr, 1971; McPherson, 1993 ; Miron et al., 1992; Mukherjee & Sarkar, 1997; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi & Saxena, 2001; D. C. Panigrahi, Saxena, V. K,
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								Udaybhanu, G., 2000; Ren et al., 1999; Sahu et al., 2009; Dick Schmal et al., 1985; A.C. Smith et al., 1988; Sondreal & Ellman, 1974; J. B. Stott, 1960; van Krevelen, 1993)
<b>Microscopy study</b>	<b>Petrographic properties</b>	Maceral and rank of fresh coal	Distribution of Maceral composition; Reflectance of vitrinite content	Distribution of Maceral composition Vitrinite, liptinite and inertinite content; Reflectance of vitrinite content	Easy to conduct; Available; Cost effective	Time consuming for preparation of blocks and its analysis	Size of the sample (-1mm to +425 micron) ; mass - 1g	(Avila, 2012; Benfell et al., 1997; D. Chandra et al., 1991; D. Chandra & Prasad, 1990; Didari,

								1988; A.G. Kim, 1977; Kruszews ka & du Cann, 1996; Kruszews ka et al., 1996; Misra & Singh, 1994; Morris & Atkinson, 1988; Ogunsola & Mikula, 1990; D. Pattanaik et al., 2011; Raju, 1988 ; Ren et al., 1999; A. C. Smith & Lazzara, 1987)
	<b>Petrographic proper</b>	Morphology study of macerals;	Artificial oxidation time and	Distribution of morphological changes of macerals,	Easy to conduct; Available; Cost effective	Manual error; Time consuming to	Size of the sample (-1mm to +425	(Avila, 2012; Benedict

	<b>ties of oxidised coal</b>	Reflectance of altered vitrinite	temperature	Reflectance of altered vitrinite		prepare sample blocks and to perform analysis	micron) ; mass - 1g; heating rate (0.5 - 3.5 °Cmin <sup>-1</sup> ); Temperature range (200 - 350 °C), Atmosphere - oxygen/ air	& Berry, 1964; Calemma et al., 1995; D. Chandra, 1958, 1962, 1965, 1975; Crelling et al., 1979; Gray et al., 1976; Ingram & Rimstidt, 1984; Ivanova & Zaitseva, 2006; Lowenhaupt & Gray, 1980; Marchioni, 1983; Mohalik, 2013; Nandi et al., 1977; Ndaji & Thomas, 1995;
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								Pearson & Creaney, 1981; Stach et al., 1982; Teichmuller & Teichmuller, 1982)
<b>Thermal Studies</b>	<b>Heat Base Measurement</b> CPT and IPT method	Heating samples in an oxidising atmosphere at a constant heating rate in a glycerine or air bath from ambient to the crossing point temperature	Single point thermal profile of sample and bed	Critical temperature of self-heating or auto-ignition of coal	Simple, easy to handle, cost effective;	Difficult to standardise the parameters e.g. packing density, air flow rate, design of sample holder; Time consuming, difficult to compare spot temperatures inside sample holder; High Moisture coal misleads results. The CPT is merely a 'snapshot' of the whole	Mass of sample ( $\leq 100$ g); Constant heating rate ( $0.5$ to $1$ $^{\circ}\text{Cmin}^{-1}$ ), Flow rate: $20 - 100$ $\text{mlmin}^{-1}$ , Atmosphere - oxygen/ air	(Avila, 2012; Bagchi, 1965, 1973; S. C. Banerjee, 1985, 2000; S. C. Banerjee et al., 1970; Chamberlain & Hall., 1973; 1999; Chen & Chong, 1997; Feng, Chakravor



						process		ty, et al., 1973; Ganguli & Banerjee, 1953; M. Gouws & L. Wade, 1989a, 1989b; M. J. Gouws & L. Wade, 1989; Kreulen, 1948; Mahadevan & Ramlu, 1985; Mohalik, 2013; D. K. Nandy et al., 1965; D. S. Nimaje et al., 2013; 1915; Y.S. Nugroho et al., 1996; Y.
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								S. Nugroho et al., 2000; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000; S. W. Parr & Coons, 1925; Sahu, Padhee, et al., 2011; Tideswell & Wheeler, 1920)
CPT at University of Nottingham	Heating coal samples in an oxidising atmosphere at a constant heating rate from	Multi point vertical and horizontal thermal profiles of sample	Determination of CPT of sample using thermal profile data as well as the POC gases released i.e. CO, CO <sub>2</sub> , H <sub>2</sub> , CH <sub>4</sub> and O <sub>2</sub>	Simple, easy to handle, cost effective, both horizontal and thermal profiles of sample	Difficult to maintain the sample holder position inside sponcomb rig, Difficult to	Mass of sample ( $\leq 100$ g); Constant heating rate (1 °Cmin <sup>-1</sup> ), Flow rate: 200 – 100	(Avila, 2012; Mohalik, 2013)	

		ambient to the crossing point temperature	determined using a water bath and a multi-gas analyser to identify major gaseous POC		determined, Study of POC gases emission	maintain air flow direction	mlmin <sup>-1</sup> , Atmosphere - air	
Chen's method	Transient solution to the heat and mass transfer of the sample;	Multi point Radial thermal profile of sample holder and bath	Central core temperature of the sample is compared to the different radial temperature	Simple, easy to handle, cost effective, radial thermal profile	Difficult to standardize the packing density of the sample in sample holder, the sample particle size distribution, the reactor design and air gap between reactor and sample holder; Limited research all over world	-	(Avila, 2012; Chen & Chong, 1997)	
Basket heating test method -(s)	Heating coal samples in an oxidising atmosphere at a constant	Temperature of sample as well as bath	Critical temperature of self-heating or auto-ignition of coal	May be used to design and operate the safe storage of coal in stock piles	Trial and error method, Tedious to determine CPT; poor	-	(Bowes & Cameron, 1971; Cameron &	

		temperature				repeatability and reproducibility ; the size of the reactor and the size of the sample basket used are arbitrary; Maintenance of same shape of sample difficult;		Maccowall , 1972; Frank-Kamenets kii, 1969; Jones, 1996, 1996 )
	Olpinski Index	Heating coal samples in an oxidising atmosphere at a constant heating rate in a quinoline vapour bath up to 235 °C	Temperature of sample as well as bath	Critical temperature of self-heating or auto-ignition of coal	Providing the information for ranking the propensity of coal to spontaneous combustion;	Repeatability poor; Difficult to maintain pellet size; Hard coal ignition point more than 235 °C and doubts of coal swelling in quinoline vapour: May not be theoretically sound	Mass : 0.3 to 0.4 g; Size of sample:-212 micron	(S. C Banerjee, 1985)
	Critical Air Blast method	Heating coal samples in an oxidising atmosphere	Temperature of sample as well as bath	The minimum rate of air blast to produce the reactivity of coal to air found by trial and error	Provides information to rank the propensity of a	Trial and error Method tedious to determine	Size of sample: -1.18 mm to +600 micron; Flow rate: 4.2	(D. C. Panigrahi & Sahu, 2004; D.

		at a constant temperature		methods	coal to spontaneous combustion;	CPT; Very poor repeatability and reproducibility; limited research all over world;	litre per minute;	C. Panigrahi, Saxena, V. K, Udaybhanu, G, , 2000)
	Puff temperature method	Sample is mixed with some additives (NaNO <sub>2</sub> ) in defined proportions and placed on a temperature controlled reactor bed.	Temperature of sample as well as bath	Critical temperature of self-heating or auto-ignition of coal	Provides the data with which to rank the propensity of a coal to spontaneous combustion;	Inconsistent repeatability of results; limited research studies reported.	-	(S. C. Banerjee, 2000)
	Calorimetric studies Adiabatic methods	Samples are placed inside a adiabatic oven or oil bath and the reactor temperature is adjusted periodically to match the change in sample	Both time and rate of temperature rise are measured to get change in temperature of sample	The ignition temperature, rate of rise temperature and kinetic constant of coal are used to predict the propensity of a coal to spontaneous combustion	Provides the data with which to rank the propensity of coal to spontaneous combustion;	Some limitations to standardisation of method; Experimental procedures consuming time; design of reacting vessel and amounts of	Mass of sample ≥ 200 g); Size of sample: - 115 to +74 micron; Temperature range: 35 - 130 °C; Atmosphere - air	(B. Basil Beamish et al., 2000; B. Basil Beamish et al., 2001; Chen & Stott, 1993; Cliff et al.,

		temperature				sample, diverse particle size and air/oxygen flow rate; Heat release is low		1996; Cudmore, 1988; Davis & Reynolds, 1928; M. Guney & Hodges, 1969; Humphreys et al., 1981; Moxon & Richardson, 1985; Ren et al., 1999; Rosin, 1929; A. C. Smith & Lazzara, 1987)
	Isothermal methods	Similar to heat basket method where the coal sample is placed in an oven at a preset temperature	Both time and temperature are measured to get change in temperature of sample	Heat released during the oxidation process, accounts for the overall effects of physical and chemical processes	Provides the data with which for ranking the propensity of <b>coal to spontaneous combustion</b> ;	The pre-set period of time to increase the oven temperature; the size of the reactor and the size of the sample basket used are	-	(Clemens et al., 1991; J. B. Stott, 1956)

						arbitrary; Results may contradict other methods		
	Thermal analysis : Differential thermal analysis (DTA)	Both sample and reference materials are heated from ambient to upper limit of oven temperature in a constant heating rate	Record of temperature difference against temperature with its characteristic heat changes and intensities,	Heat released during the oxidation process; Reflect the physical and/or chemical changes of the material at the particular temperature; slopes of stage II DTA curves; temperature initiation of stage III	Technique provides both qualitative and quantitative measurements; Providing the information for ranking the propensity of coal to spontaneous combustion; appropriate for examining the reaction steps	Lack of repeatability and reproducibility under same experimental conditions; Curve characteristics i.e. peak width and depth varies with small change in sample holder and furnace replaced; Contradicts the assessment produced by other methods; Small amount of sample	Mass of sample ( $\leq 30$ mg); Constant heating rate ( $0.5$ to $30$ $^{\circ}\text{Cmin}^{-1}$ ), Flow rate: $20 - 100$ $\text{mlmin}^{-1}$ , Atmosphere - oxygen/ air	(S. C. Banerjee, 2000; S.C. Banerjee & Chakraborty, 1967; Mohalik, Panigrahi, & Singh, 2009; D. C. Panigrahi & Saxena, 2001; Whitehead & Breger, 1950)
	Differential Scanni	Both sample and reference	Difference in energy inputs into a	Heat released during the oxidation process;	Technique provides both qualitative and	No unanimity on the experimental	Mass of sample ( $\leq 30$ mg); Constant	(Mahajan & Walker Jr, 1971;

DSC	materials are heated from ambient to upper limit of oven temperature in a constant heating rate	substance and a reference material is measured	Reflect the physical and/or chemical changes of the material at the particular temperature; onset temperature, integral value	quantitative measurements; Providing the information for ranking the propensity of coal to spontaneous combustion; Appropriate for examining the reaction steps	parameters used by different researchers; No clear guideline for comparison of spontaneous combustion propensity between two samples	heating rate (0.5 to 30 °Cmin <sup>-1</sup> ), Flow rate: 20 – 100 mlmin <sup>-1</sup> , Atmosphere – oxygen/ air	Mohalik, Panigrahi, & Singh, (2009)
TGA	Sample is subjected to a controlled temperature programme from ambient to upper limit of oven temperature	Mass change with respect to time and temperature, derivatives of above data	Mass variation reflects the adsorption of O <sub>2</sub> and the production of the oxidation products in both the gas and solid phases; 0.1 dw/dt temperature; rate of heat release; weight changes; thermogravimetric spontaneous combustion index	Technique provides both qualitative and quantitative measurements; Provides data with which to rank the propensity of coal to spontaneous combustion; Determines spontaneous combustion mechanism; appropriate to examine reaction steps	Limited research on the experimental parameters used; Need to standardise experimental parameters by rigorous tests of samples from all over world	Mass of sample (≤ 30 mg); Constant heating rate (0.5 to 30 °Cmin <sup>-1</sup> ), Flow rate: ≤ 200 mlmin <sup>-1</sup> , Atmosphere – oxygen/ air	(Avila, 2012; 1979; Filho & Milioli, 2008; Gil et al., 2010; Idris et al., 2012; Idris et al., 2010; Khare et al., 2011; M. V. Kok, 2012; C. Lu et al., 2009; Marinov



								(a), 1977; Marinov (b), 1977; Marinov (c), 1977; Marinov, 1977; Mohalik, 2013; Slovak, 2001)
Oxygen Avidity studies	Peroxy complex analysis	The physico-chemisorption and chemical reaction process	Addition of different oxidizable agents i.e. $\text{TiCl}_3$ or $\text{C}_2\text{FeN}_2\text{S}_2$ or $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ to coal for oxygen consumption	Liberation of gases i.e. $\text{CO}$ , $\text{CO}_2$ , $\text{H}_2\text{O}$ and its quantification with respect to oxygen consumption	Simple ; Less time consuming; Evaluation of spontaneous combustion mechanism;	No standardisation; Sensitivity of results may produce poor repeatability; Difficult to maintain experimental conditions	-	(S. C. Banerjee, 2000; Sen et al., 2009)
	Wet oxidation potential methods	Reagents including nitric acid ( $\text{HNO}_3$ ), alkaline and acidic permanganates	Coals liable to oxidation are the alicyclics or hydroaromatics	Potential difference between saturated calomel and carbon electrode	Simple ; Provides data with which to rank the propensity of coal to spontaneous combustion; Evaluation of spontaneous combustion	No standardisation; Research limited to Indian coal samples; Sensitivity of results may be produce poor	Mass of sample (1 g); Constant temperature ( $45^\circ\text{C}$ ), 0.2 N $\text{KMnO}_4$ in 1 N $\text{KOH}$ solution	(S. C. Banerjee, 2000; D. C. Panigrahi & Sahu, 2004; D. C. Panigrahi, Saxena,

					mechanism	repeatability		V. K, Udaybhanu, G, , 2000; D. C. Panigrahi et al., 1996; S. Ray & Panigrahi, 2015; S. K. Ray et al., 2014; Tarafdar & Guha, 1989)
	Russian U index	Amount of oxygen absorbed by known coal sample for one day	Compositional analysis of the gas	Determined oxygen absorbed by the coal	Provides data with which to rank the propensity of coal to spontaneous combustion	Simple; No standardisation; Results may contradict other methods: Poor repeatability: Limited number of research studies	-	(S. C. Banerjee, 2000)
	Rate studies or kinetic	Heating samples either isothermal or	Reaction steps depends upon	Reaction rate constant; frequency factor; activation energy	Provides data with which to rank the propensity of	No standardisation; Time consuming	-	(S. C. Banerjee, 2000; Filho &

	studies	adiabatic methods	temperature and time		coal to spontaneous combustion; Evaluation of spontaneous combustion mechanism			Milioli, 2008; Gil et al., 2010; Idris et al., 2012; Idris et al., 2010; Khare et al., 2011; M. V. Kok, 2012; C. Lu et al., 2009; Mohalik, 2013; Slovak, 2001)
Advanced miscellaneous techniques	IR/FTIR	Changes to IR/FTIR spectra	Concentration of solid oxygenated species formed at coal surface	Changes to IR/FTIR spectra	Evaluation of spontaneous combustion mechanism	Results have not been related to a classification system	-	(Di Nola et al., 2010; Fang et al., 2006; Gethner, 1985, 1987; Huffman et al., 1985; Iglesias et al., 1998; Kister et

								al., 1988; M. Kok, 2008; Ledesma et al., 2000; Liotta et al., 1983; Low & Tascon, 1985; MacPhee et al., 2004; Mahidin et al., 2002; Materazzi & Vecchio, 2010; Ogloza & Malhotra, 1989; Painter et al., 1980; Rhoads et al., 1983; D. Wang et al., 2010; Wu et al., 1988)
XRD	Studies the	Concentratio	Studies the mineral	Evaluation of	did	not	-	(1975;

		mineral transformation and catalytic action of minerals	n of the solids oxygenated species formed at coal surface	transformation and catalytic action of minerals	spontaneous combustion mechanism; structural changes of basic composition	suggest the classification system Results have not been related to a classification system		Gong et al., 1998; 1998; Kelemen et al., 1990; M. Kok, 2008)
	NMR		Concentration of solid oxygenated species formed at coal surface	Reaction mechanism of spontaneous combustion of coal	Evaluates the spontaneous combustion mechanism; structural changes of basic composition	Results have not been related to a classification system	-	(Kalema & Gavalas, 1987; Mao et al., 2010; Sen et al., 2009; Yokono et al., 1986)
	GC and GC-MS	Product of combustion gases	The index gases liberated from a coal bed heated under a temperature controlled source	Identifies the solid oxygenated species formed at coal surface; Reliable data with which to identify the principal elementary mechanisms and to quantify the rate parameters	Provides the data with which to rank the propensity of a coal to spontaneous combustion;	One fixed temperature; No standardisation	-	(Jo et al., 2013; Kalema & Gavalas, 1987; P. Lu et al., 2004; Pisupati & Scaroni, 1993; Sen et al., 2009)

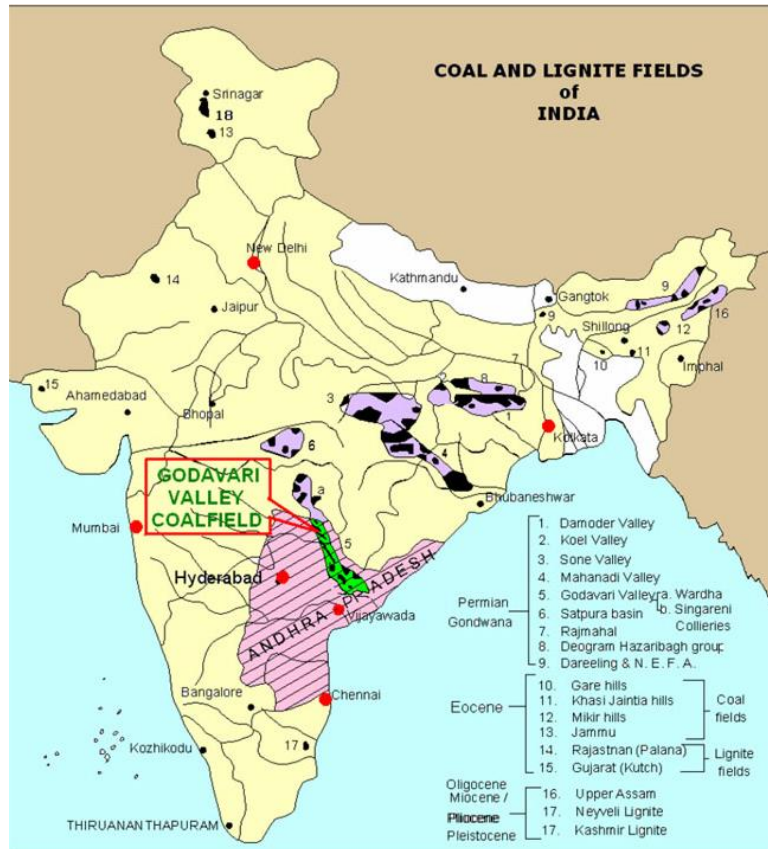


Fig.1. Major coalfields of India (<http://rajikorba.blogspot.in/2011/06/coal-lignite-fields-in-india.html>)(Joshua, 2011)

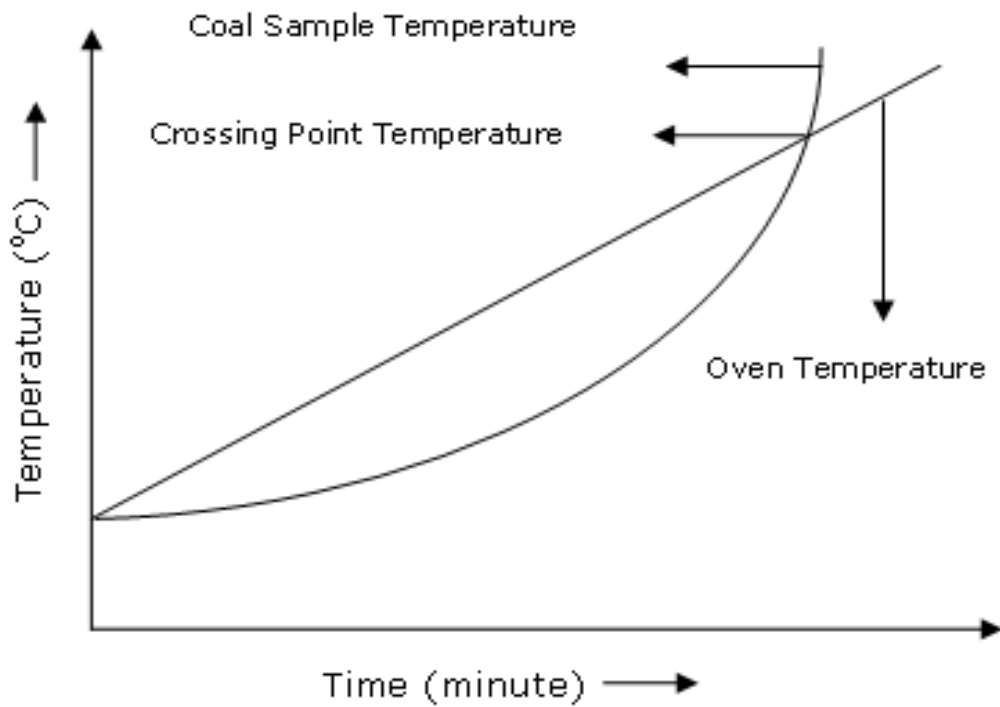


Fig. 2. Crossing point temperature determination of coal

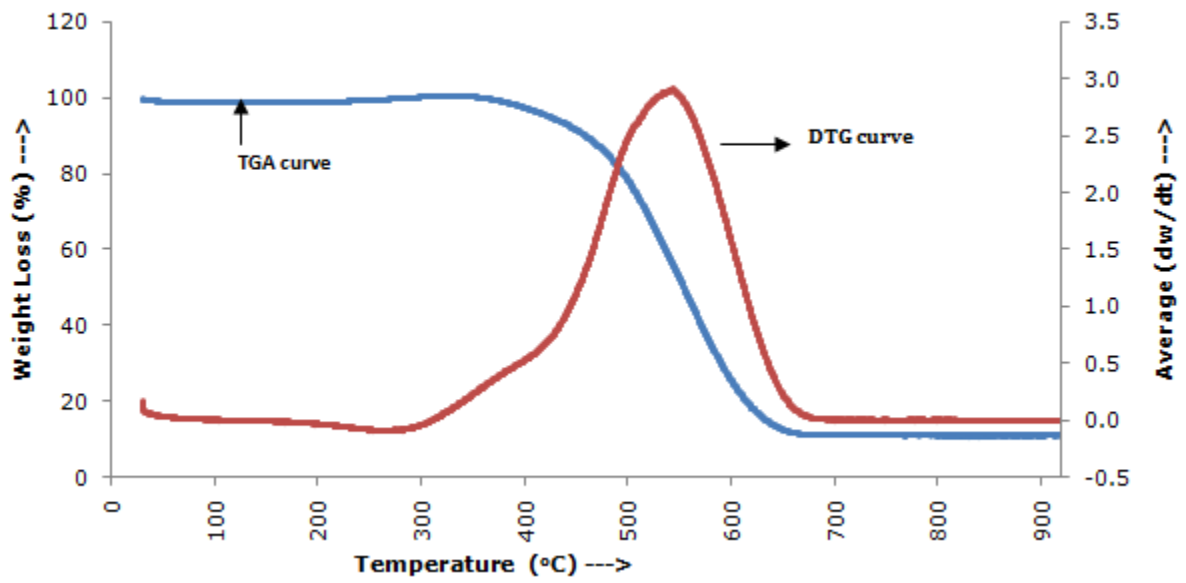


Fig. 3. TGA and DTG curve of coal sample under combustion