

Assessment of auto-oxidation potential of some Indian coals by differential thermal analysis (DTA) technique

It is a well-known fact that coal when exposed to air, undergoes oxidation, even at ambient temperature conditions. The process is exothermic in nature. If the amount of heat liberated during the process is not dissipated, it leads to an increase in temperature, which in turn enhances the rate of reaction, ultimately culminating in open fire. These fires are the major causes of accidents resulting in loss of lives and property across the globe. Apart from loss of valuable coal reserves, blockage of resources and capital, reduction of heating values and coking properties; it also causes serious environmental pollution. However, all coals are not susceptible to auto-oxidation to the same extent. Therefore, accurate assessment of the oxidative tendency is essential to plan the production and storage capabilities in mines. There are different innovations and technologies available in various parts of world to assess and control spontaneous heating tendency, till date there is no unanimity to adopt single method for its assessment. In this study, an attempt has been made to study the auto-oxidative tendency of Indian coal seams by using different thermal technique (DTA). For this purpose 48 coal samples were collected from all major coalfields of the country. The intrinsic properties of these samples were determined using proximate analysis and bomb calorimeter. Correlation study between the intrinsic parameters and susceptibility indices indicate that DTA can be a better method for assessment of the auto-oxidation potential of coal.

Keywords: Auto-oxidation, spontaneous heating, differential thermal analysis, intrinsic properties, coal.

1. Introduction

Spontaneous heating caused mine fire is the prime concern in all coal producing countries as they involved in both the production and economic losses to the organization. Development of spontaneous heating not only creates the working difficulties but also accounts for scarcity of the resource. A careful analysis into occurrence of these fires tells that most of the cases these fires could have

been averted if suitable preventive measures have been taken. Therefore it is always necessary to assess the degree of proneness for advance precautionary measure against the occurrence of the coal mine fire.

All types of coal generally absorb oxygen and oxidise to evolve CO, CO₂, and H₂O along with heat. This heat slowly dissipates into air. If the rate of oxidation is slower and the amount of heat dissipation is higher than the amount of heat accumulation then coal does not reach ignition temperature (critical temperature for oxidation) and the process is referred to as weathering. But in case the oxidation rate is faster and the amount of heat accumulation is higher than the amount of heat dissipation, it makes the coal reach the critical temperature for oxidation resulting in coal ignition which is referred to as spontaneous heating or auto-oxidation of coal. Several incidents of mine fires followed by spontaneous combustion of coal can be found since the beginning of coal mining history. In India, spontaneous combustion is seen in all major coalfields like Raniganj, Jharia, Karanpura, Bokaro, Ib-valley, Talcher etc. (Pattanaik et.al, 2011). An example of most spontaneous combustion susceptible coalfield is Jharia coalfield in BCCL India which has many mine fires burning since 1925 (Ramlu, 2007). From the last 140 years, the Indian coalfields have been recorded a large number of extensive open and concealed fires (Raniganj, 1865; Jharia, 1916). 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 10 decades (Mohalik et.al, 2016).

Cases of fire have also been reported during transportation of coal. Another problem associated with spontaneous heating is the deterioration of coal quality due to oxidation on coming in contact with air. This reduces the heating value of coal and affects its coking properties. Storage and transport of run-of-mine coal is an integral part of coal mining industry. Coal handling systems are required not only at mines but also at power plants and metallurgical coking plants. Investigators have found that these incidents could have been prevented if proper planning and design for

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coal production and handling was adopted. This has led to number of researches to investigate and try to understand the process of spontaneous heating and find methods to prevent it. It is well known that spontaneous heating of coal depends mainly on two types of factors, such as intrinsic and extrinsic. The intrinsic parameters are mainly associated with the nature of the coal, i.e. its physico-chemical characteristics, petrographic distribution and mineral make up. On the other hand, the extrinsic parameters are related to atmospheric, geological and mining conditions prevailing during extraction of coal seams and these are mainly site specific. The intrinsic coal properties that control propensity for self-heating have been the subject of many investigations (Singh and Demirbilek, 1987; Smith and Lazzara, 1987; Beamish, 2008a; Panigrahi and Saxena, 2001. Kucuk et al., 2003).

On this context it is extremely important to determine and assess the spontaneous heating proneness for improved planning of the mine workings. In the present work, an extensive study has been carried out by collecting a large number of coal samples (48 coal samples) from different Indian coalfields and by determining their spontaneous heating susceptibility using differential thermal analysis technique. The intrinsic properties of the coal samples have been determined by using proximate analysis and bomb calorimeter. The indices that were obtained from DTA analysis are transition temperature (T_c), slopes of IIA, IIB and overall of stage II obtained from DTA thermogram. The correlation study was carried out between intrinsic parameters and susceptibility indices in order to assess their acceptability.

2. Details of the coal sample for the study

The samples were collected by using the channel sampling method (IS 436 Part I/Section I, 1964). The method consisted of cutting channels across the face of exposed ore and collecting the resulting chips, fragments and dust from each channel to make up a sample. First, a coal seam was chosen and the entire length up to a width of 10 cm of it demarcated by chalk was cut to a depth of 2.5cm. In selecting the place for sampling dirt bands exceeding have to be avoided.

The sample from different coalfields were brought to the laboratory. The relatively big pieces were crushed to small pieces. Coning and quartering procedure was done to get a representative sample of the entire coal sample. This was then ground and screened (sieving) to a size of -212 (micron). Finally, the samples were kept in air-tight plastic bags for further analysis and to isolate them from external factors which might result in some change of its properties. Table 1 presents the details of samples collected, i.e. name of the sample and the subsidiary company.

3. Determination of intrinsic properties of coal

The intrinsic properties of these coal samples were determined by proximate analysis and bomb calorimeter.

TABLE 1: LIST OF SAMPLES COLLECTED

Sample no.	Sample name	Sample no.	Sample name
1.	SCCL-1	25.	MCL-2
2.	SCCL-2	26.	MCL-3
3.	SCCL-3	27.	MCL-4
4.	SCCL-4	28.	MCL-5
5.	SCCL-5	29.	MCL-6
6.	SCCL-6	30.	MCL-7
7.	SCCL-7	31.	MCL-8
8.	SECL-1	32.	MCL-9
9.	SECL-2	33.	MCL-10
10.	SECL-3	34.	MCL-11
11.	SECL-4	35.	MCL-12
12.	SECL-5	36.	MCL-13
13.	SECL-6	37.	MCL-14
14.	SECL-7	38.	MCL-15
15.	CCL-1	39.	MCL-16
16.	CCL-2	40.	MCL-17
17.	ECL-1	41.	MCL-18
18.	ECL-2	42.	MCL-19
19.	ECL-3	43.	BCCL-1
20.	ECL-4	44.	BCCL-2
21.	ECL-5	45.	BCCL-3
22.	ECL-6	46.	WCL-1
23.	NEC-1	47.	NLC-1
24.	MCL-1	48	NCL-1

SCCL – Singareni Collieries Company SECL – South Eastern Coalfields Ltd. CCL – Central Coalfields Ltd. ECL – Eastern Coalfields Limited NEC – North Eastern Coalfields MCL – Mahanadi Coalfields Ltd. BCCL – Bharat Coaking Coal Ltd. WCL – Western Coalfields Ltd. NLC – Neyveli Lignite Corporation NCL – Northern Coalfields Ltd

Proximate analysis represents the percentage by weight of the fixed carbon, volatiles, ash, and moisture content in coal. Whereas the amounts of fixed carbon and volatile matter present in the coal directly contribute to the heating value of coal. Fixed carbon present in the coal acts as a main heat generator during heating process leads to burning. Content of high volatile matter indicates easy ignition of fuel. The content of ash is important in the design of the furnace grate, combustion volume, pollution control equipment and ash handling system of a furnace. These variables are presented in an air dried basis. This basis neglects the presence of moisture other than inherent moisture.

3.1 PROXIMATE ANALYSIS (IS 1350 PART I -1984)

The coal samples were collected from different seams were brought to the laboratory in sealed condition for analysis. The moisture (M), volatile matter (VM) and ash content (A) of coal samples were determined by proximate analysis following the method specified by IS (Indian Standard) 1350, Part - I (1969). The calorific value of these samples were determined using a Parr 6100 bomb calorimeter. The results of the intrinsic properties are presented in Table 2.

TABLE 2: RESULTS OF PROXIMATE ANALYSIS AND GROSS CALORIFIC VALUE

Sample no.	Sample name	Moisture (%)	Volatile matter (V.M.%)	Ash (%)	Fixed carbon (F.C.%)	Gross calorific value (Kcal/kg)
1	SCCL-1	5.50	26.00	18.00	50.50	5708.50
2	SCCL-2	4.50	32.00	12.00	51.50	6290.20
3	SCCL-3	5.97	35.49	26.49	32.05	5015.32
4	SCCL-4	2.31	26.34	29.72	41.63	5425.22
5	SCCL-5	4.29	11.99	35.16	48.56	4624.71
6	SCCL-6	2.78	16.93	33.21	47.08	5028.17
7	SCCL-7	4.98	18.78	20.01	56.23	5950.77
8	SECL-1	5.00	26.50	17.00	51.50	5931.04
9	SECL-2	5.00	32.33	34.00	28.67	4316.27
10	SECL-3	7.67	29.83	18.88	43.62	7160.23
11	SECL-4	4.85	16.9	21.85	56.40	5796.44
12	SECL-5	9.96	12.60	30.39	47.05	4248.30
13	SECL-6	2.93	12.04	30.88	54.15	5225.73
14	SECL-7	7.67	10.80	30.92	50.61	4531.82
15	CCL-1	5.05	22.49	48.51	23.95	3156.57
16	CCL-2	10.00	32.27	18.00	39.73	5119.78
17	ECL-1	1.00	21.89	34.33	57.22	5337.35
18	ECL-2	1.00	24.62	26.50	47.88	6295.61
19	ECL-3	1.00	22.54	34.16	42.30	5426.97
20	ECL-4	1.00	22.11	34.33	42.56	5121.95
21	ECL-5	8.43	24.43	9.60	57.54	7315.23
22	ECL-6	1.80	36.13	14.27	47.80	3481.05
23	NEC-1	1.50	43.03	5.37	50.10	8075.08
24	MCL-1	6.50	32.00	23.50	38.00	4782.11
25	MCL-2	2.98	34.17	12.00	50.85	6691.48
26	MCL-3	11.13	25.19	38.46	25.22	3463.14
27	MCL-4	14.29	31.25	16.27	38.19	5156.32
28	MCL-5	7.25	23.90	40.50	28.35	3512.81
29	MCL-6	8.97	29.49	22.88	38.66	4987.39
30	MCL-7	10.02	26.06	31.57	32.35	3999.07
31	MCL-8	11.32	29.81	21.80	37.07	4896.30
32	MCL-9	9.12	33.51	8.37	48.91	6377.20
33	MCL-10	8.72	25.14	13.75	52.39	6147.50
34	MCL-11	6.74	26.14	34.89	32.23	3802.35
35	MCL-12	7.64	22.13	37.02	33.21	4112.82
36	MCL-13	5.00	24.01	32.40	38.59	4027.36
37	MCL-14	4.88	24.17	38.87	32.08	3800.69
38	MCL-15	6.31	28.39	32.42	32.88	2366.66
39	MCL 16	14.5	31.97	12.35	41.18	5285.92
40	MCL 17	6.65	30.31	22.53	40.51	5470.33
41	MCL 18	6.94	28.48	28.22	36.36	4892.34
42	MCL-19	6.7	11.5	40.9	40.90	3733.33
43	BCCL-1	1.90	33.08	16.00	49.02	6858.61
44	BCCL-2	0.60	22.32	10.73	66.35	6697.94
45	BCCL-3	1.00	17.36	15.73	65.91	7177.43
46	WCL-1	14.39	29.31	12.76	43.54	5216.94
47	NLC-1	37.30	32.32	3.40	26.98	5017.34
48	NCL-1	9.68	29.80	18.12	42.40	5684.60

4. Determination of susceptibility indices

4.1 DIFFERENTIAL THERMAL ANALYSIS

Thermal analysis is the technique by which physical and chemical changes of a substance are measured as a function of temperature as the substance is subjected to a controlled heating rate. Differential thermal analysis is a technique in which the temperature of the substance under investigation is compared to the temperature of a thermally inert material such as alpha-alumina and is recorded with furnace temperature as the substance is heated or cooled at a predetermined uniform rate. If a sample undergoes no thermal change and the temperature difference is zero, then a base line parallel to recording instrument is given by the recording instrument. If the sample undergoes a thermal decomposition reaction with loss or gain of heat then the temperature gradient with respect to inert material will change producing a positive or negative ΔT depending on whether the transformation taking place in the sample is endothermic or exothermic. DTA is a quick method which does not require complicated instruments. With modern DTAs, the measurements can be programmed so that the analysis is not very time-consuming. The curve recorded with ΔT plotted on the ordinate and temperature or time on the abscissa is called the DTA curve or thermogram.

The schematic diagram of a DTA apparatus has been presented in Fig.1. The sample holder assembly consists of a thermocouple each for the sample and reference, surrounded by a block to ensure an even heat distribution. The sample is contained in a small crucible designed with an indentation on the base to ensure a snug fit over the thermocouple bead.

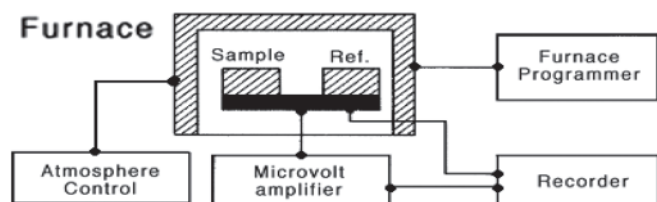


Fig.1 Schematic diagram of a typical DTA apparatus

Experimental procedure

In the present study a differential thermal analyser DTA 60/60H (Schimadzu) was used (Fig.2). Standardized parameters suggested by Banerjee and Chakravorty (1967) were followed while performing the experiments. DTA thermograms were obtained by maintaining a heating rate of 5°C per min and alumina powder was taken as the reference material. Thermograms for all the samples were obtained by following this procedure.

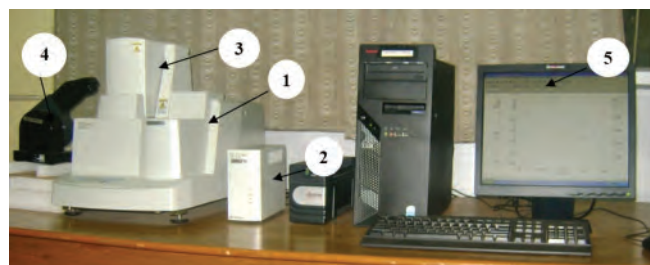


Fig.2: Experimental set up of differential thermal analysis apparatus (1) Differential thermal analyser (DTA- 60H) (2) TA - 60 WS collection monitor (3) Tubular furnace (4) Blower (5) Computer for data processing

It has been shown by Banerjee and Chakravorty (1967) that a thermogram of coal can be divided into three segments or stages. In the initial stage of heating (stage I), the endothermic reaction predominates, probably due to the release of inherent moisture in coal. In the second stage (stage II), the exothermic reaction becomes significant, but the rate of heat release is not steady all through, as it changes with temperature. A steep rise in heat evolution is observed in the third stage (stage III). The same observation has been made in this study.

The rate of temperature rise in stage II is cited by different researchers (Banerjee and Chakravorty, 1967; Gouws and Wade, 1989, Sahu et al, 2005) as being less for coals with less susceptibility to spontaneous heating. The exothermicity in stage III is not regarded as a reliable indicator of the self-heating risk, because it may be equally high for low rank coals. However, the temperature of transition or characteristic temperature or onset temperature is considered to be significant. It is considered that in lower temperatures, the coal is more susceptible towards spontaneous heating.

Therefore, all the thermograms were analyzed for the following details:

Onset temperature or characteristic temperature

The onset temperature or characteristic temperature was determined by the following procedure:

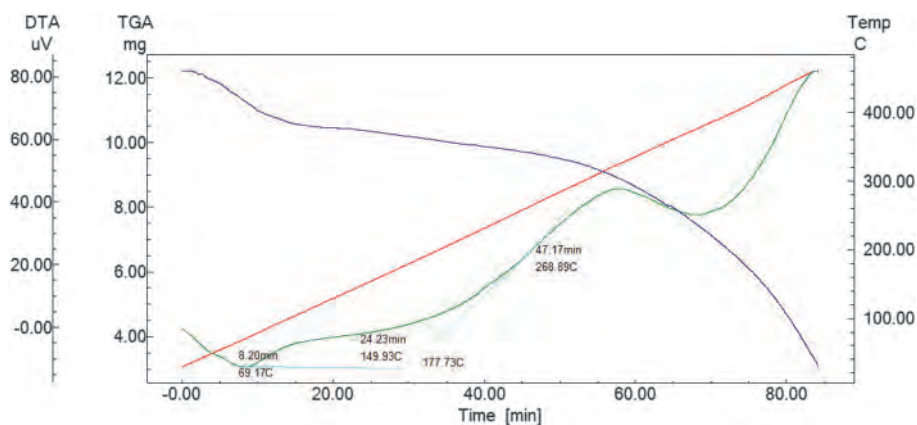


Fig.3 Determination of onset temperature from DTA thermogram for SCCL-1 coal sample

- ♦ A tangent was drawn at the inflexion point of the endothermic region and another tangent was drawn at the rising portion of the curve of stage III.
- ♦ The intersection between the two tangents gives the characteristic temperature.
- ♦ The depiction of characteristic or onset temperature (T_c) from DTA thermogram has also been presented in Fig.3.

Slopes of stage II of the thermogram

In the thermograms obtained from the experiments, linear stage II exothermicity is not observed. In view of the disjointed nature of stage II slopes it was further divided into two different regions, viz. stage IIA and stage IIB. The following three parameters of stage II were determined for further analysis.

- ♦ Average slope of stage IIA.
- ♦ Average slope of stage IIB.
- ♦ Overall slope of stage II.

The characteristic or onset temperature (T_c) and the slopes of stage IIA, IIB and overall slope of stage II of DTA thermograms were obtained by using the TA-60 software provided with the equipment and the results have been presented in Table 3.

5. Correlation study

Correlation study was carried out to determine the relationship between the intrinsic properties of coal with the susceptibility indices. The intrinsic parameters determined by proximate analysis and bomb calorimetry were taken as independent variables; whereas T_c , slopes of stage IIA, IIB and overall slope of stage II obtained from DTA thermograms, as depended variables. The results of the correlation study are presented in Table 4. The correlation plots for some of the cases has been presented in Fig.4:

TABLE 3: TRANSITION TEMPERATURE AND SLOPES OBTAINED FROM DTA THERMOGRAMS

Sample no.	Sample name	Transition temperature (°C)	Slope		
			IIA	IIB	II
1.	SCCL-1	177.73	0.1329	0.2360	0.1611
2.	SCCL-2	197.82	0.1429	0.3149	0.2063
3.	SCCL-3	182.20	0.1222	0.3516	0.1723
4.	SCCL-4	168.02	0.0720	0.1098	0.0772
5.	SCCL-5	158.95	0.1367	0.2419	0.1581
6.	SCCL-6	151.70	0.1230	0.1843	0.1319
7.	SCCL-7	115.39	0.1844	0.1473	0.1433
8.	SECL-1	175.15	0.1274	0.3345	0.1747
9.	SECL-2	188.42	0.0974	0.3183	0.1567
10.	SECL-3	172.55	0.1051	0.1646	0.1047
11.	SECL-4	159.83	0.0786	0.0938	0.1647
12.	SECL-5	168.15	0.0799	0.1517	0.0900
13.	SECL-6	167.53	0.0870	0.1416	0.0947
14.	SECL-7	167.18	0.0635	0.1329	0.0784
15.	CCL-1	192.00	0.0812	0.2904	0.1274
16.	CCL-2	180.03	0.1129	0.2277	0.1291
17.	ECL-1	234.54	0.0103	0.2556	0.0522
18.	ECL-2	230.77	0.0213	0.3321	0.0744
19.	ECL-3	233.33	0.0053	0.1784	0.0529
20.	ECL-4	235.41	0.0122	0.2837	0.0546
21.	ECL-5	238.44	0.0045	0.1275	0.0332
22.	ECL-6	178.16	0.0901	0.1466	0.0922
23.	NEC-1	178.44	0.0472	0.2831	0.1132
24.	MCL-1	190.20	0.1837	0.4113	0.2298
25.	MCL-2	174.36	0.0275	0.1849	0.0527
26.	MCL-3	198.00	0.0712	0.2481	0.1103
27.	MCL-4	152.74	0.1380	0.1551	0.1194
28.	MCL-5	176.67	0.0755	0.1361	0.0800
29.	MCL-6	198.49	0.1053	0.2281	0.1331
30.	MCL-7	182.90	0.0905	0.1825	0.1083
31.	MCL-8	168.25	0.0981	0.1614	0.1004
32.	MCL-9	167.98	0.0906	0.1574	0.0963
33.	MCL-10	164.58	0.0927	0.1740	0.1038
34.	MCL-11	193.29	0.1027	0.2349	0.1327
35.	MCL-12	189.46	0.0984	0.2469	0.1300
36.	MCL-13	195.15	0.0681	0.1354	0.0065
37.	MCL-14	185.08	0.0806	0.1878	0.0999
38.	MCL-15	187.56	0.0574	0.1321	0.0676
39.	MCL-16	178.32	0.0773	0.2192	0.1088
40.	MCL-17	158.47	0.0506	0.2083	0.0782
41.	MCL-18	190.02	0.0690	0.1795	0.0957
42.	MCL-19	178.77	0.0657	0.1770	0.0950
43.	BCCL-1	206.48	0.0327	0.2430	0.1044
44.	BCCL-2	213.41	0.0163	0.1354	0.0540
45.	BCCL-3	235.12	0.0021	0.1339	0.0301
46.	WCL-1	172.55	0.1651	0.2272	0.1575
47.	NLC-1	192.47	0.0573	0.2000	0.1001
48.	NCL-1	193.54	0.0763	0.1569	0.0819

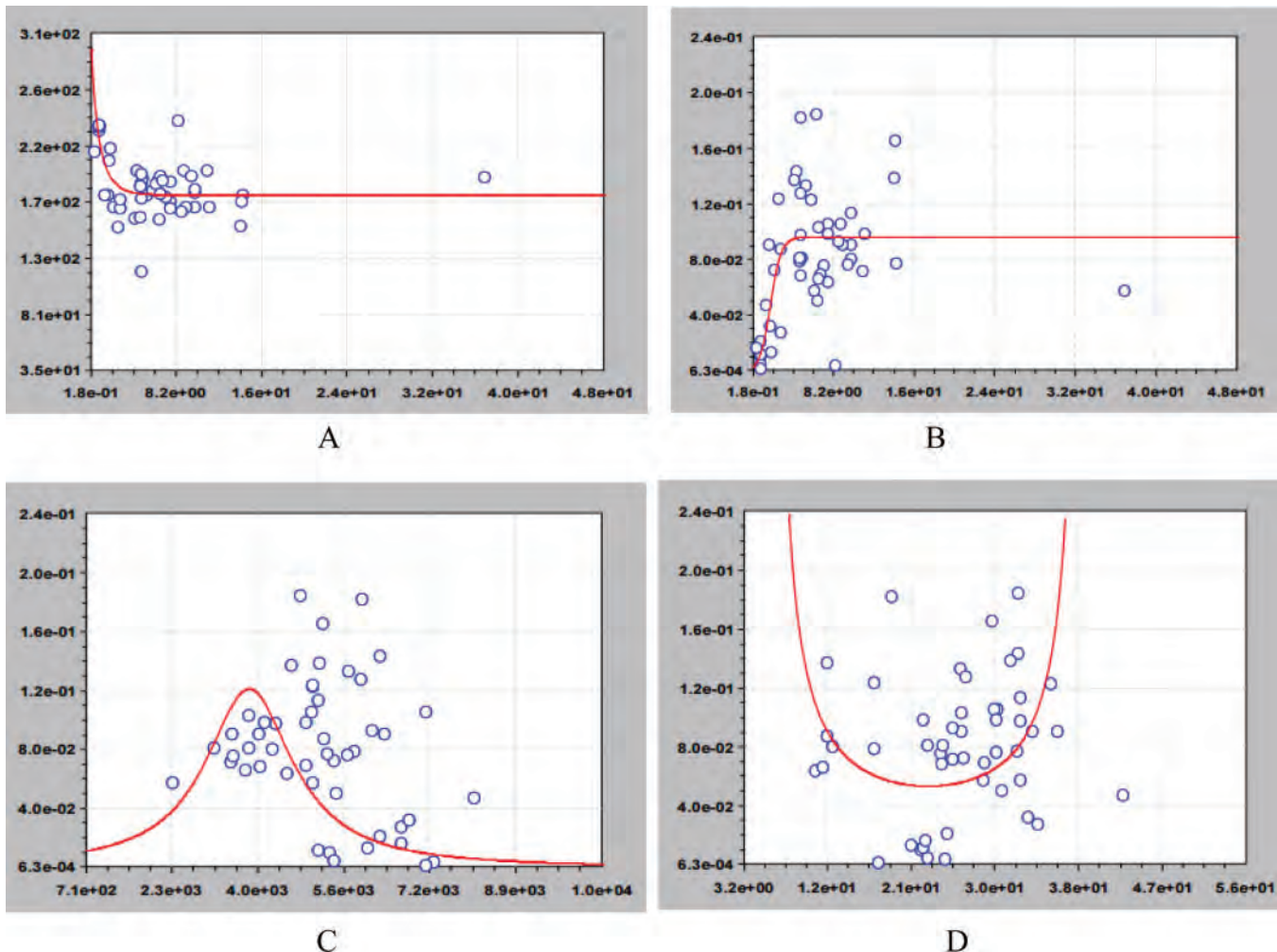


Fig.4 Correlation plots: A – Correlation plot between Tc and moisture content B – Correlation plot between IIA slope and moisture content C – Correlation plot between GCV and IIA slope D – Correlation plot between II slope and volatile matter content

TABLE 4: CORRELATION BETWEEN INTRINSIC PROPERTIES AND SUSCEPTIBILITY INDICES

Independent variable		DTA			
		Tc	IIA	IIB	II
1.	M	0.62	0.64	0.08	0.46
2.	VM	0.34	0.58	0.36	0.58
3.	A	0.19	0.15	0.13	0.23
4.	FC	0.39	0.61	0.38	0.74
5.	GCV	0.22	0.88	0.24	0.32

6. Discussion and conclusion

5.1 DISCUSSION

In order to assess the spontaneous heating tendency of Indian coals, 48 coal samples belonging to all major coalfields were collected. Their intrinsic properties were determined by proximate analysis and bomb calorimetry and spontaneous heating tendency by differential thermal analysis method.

It may be noted from Table 2 that the moisture content of

coal samples varied from 0.6 (BCCL-2) to 37.30% (NLC-1). The volatile matter content varied from 10.80 (SECL-7) to 43.03% (NEC-1). The NEC coal has highest volatile matter content followed by NCL, SCCL, MCL and WCL. The MCL coals have high to medium volatile matter content. Similarly the maximum ash content is in CCL-1 coal (48.51%)

and lowest ash 3.40% in NEC-1 sample. The NEC sample has the highest gross calorific value with 8075.08cal/gm, while the lowest calorific value is of MCL-15 with 2366.66 cal/gm. MCL coals have medium to high calorific value while SCCL, SECL, ECL and BCCL has high calorific value.

The transition temperature (Tc) value of the coal samples varied from 115.39°C (SCCL-7) to 238.44°C (ECL-5). Panigrahi and Sahu (2004) have found that the coal seams having onset temperature in the range of 122°C to 140°C are highly susceptible to spontaneous heating. As per the

classification suggested by Panigrahi and Sahu (2004) sample SCCL-7 is the highest susceptible to spontaneous heating. This is followed by SCCL-6 and MCL-4 with a Tc of 151.70°C and 152.74°C respectively. Similarly the samples collected from ECL has lower susceptible to spontaneous heating with high transition temperature value.

The IIA slope values vary from 0.0021 (BCCL-3) to 0.1884 (SCCL-7), IIB slope varies from 0.0938 (SECL-4) to 0.4113 (MCL-1) and overall slope of stage II varies from 0.0065 (MCL-13) to 0.2298 (MCL-1). It has been observed in the past that higher is the slope values, higher will be the spontaneous heating susceptibility of coal. SCCL-7 has the lowest onset temperature and the highest slope values among all the samples. So it may be considered to be very highly susceptible.

It may be noted from the correlation study (Table 5) that moisture, volatile matter and gross calorific value shows good correlation with the onset temperature and slopes obtained from DTA thermogram.

Conclusion

From the experimental investigation it was found that the coal samples from SCCL were very highly susceptible to spontaneous heating, whereas the samples from BCCL and ECL were found to less susceptible to spontaneous heating. In general the correlation of the intrinsic properties is better with the susceptibility indices obtained from DTA thermogram, particularly with the onset temperature (Tc) and IIA slope of the DTA parameters. Therefore the onset temperature (Tc) and the IIA slope obtained from DTA thermogram can be adopted as a better measure of spontaneous heating tendency of Indian coals. The correlation of intrinsic properties with the susceptibility indices showed that the properties like moisture, volatile matter and fixed carbon have good correlation with the onset temperature (Tc) and IIA slope. This is consistent with earlier works by other researchers.

In India, crossing point temperature (CPT) is taken as the measure of spontaneous heating susceptibility of coals in India. However, it has been observed in the past that it does not predict the susceptibility for all types of coals, particularly for high moisture coals. Moreover, it takes approximately 3 hours to determine the CPT which is time consuming. In the event of a power failure, the whole experiment has to be repeated. Reproducibility of experimental results of crossing point temperature also posed a major problem in the dissertation. Some of these demerits can be overcome by carrying out the differential thermal analysis, where it takes about 2 hours for the completion of an experiment. The results are highly reproducible and it is very easy to determine the transition temperature and slope values using the software available with the equipment.

Acknowledgements

Authors extend their sincere gratitude towards the staff and management of the Institute for rendering the admirable support and assistance during the experimental work. The authors thank the Institutional funding provided by National Institute of Technology Rourkela for the purpose of present study.

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